Review

Current Research on the Speciation and Determination of Liquid Metal: Mercury in Different Environmental Matrices Using Various Analytical Instruments

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Abstract: This paper reviews the current research on the speciation and determination of mercury by various analytical instruments such as, atomic absorption spectrometer (AAS), voltammeter, inductively coupled plasma optical emission spectrometer (ICP-OES), ICP-atomic emission spectrometer (AES), ICP-mass spectrometer (MS), atomic fluorescence spectrometer (AFS), spectrophotometer, spectrofluorometer, and high performance liquid chromatography (HPLC). Approximately 126 research papers on the speciation and determination of mercury by various analytical instruments published in international journals since 2015 are reviewed. All the analytical parameters, such as limit of detection, linearity range, quality assurance and quality control studies, applicability, and interfering ions studies evaluated in the reviewed articles are tabulated. This review find outs the lack of information on speciation studies of mercury in recent years. Another important prediction from this review is authors are not much studied about the concentrations of mercury in the atmosphere.

Keywords: speciation; mercury; analytical instruments; speciation; environmental samples

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1. Introduction

Mercury is only the metal exists in liquid state among the elements present in our modern periodic table. The determination and speciation studies of mercury attract the researchers due to its toxicity to the humans and other animals in food web. The difference between the toxicity of mercury and to other metals is, mercury can accumulate in biological materials easily. A few studies reported the bioaccumulation of mercury into different aquatic animals, such as fishes, pelagic feeding seabirds and earthworms [1-12].

This section describes the sources and fate of mercury into the environment and toxicity of mercury.

1.1 Sources and Fate of Mercury into the Environment:

Mercury can enter into the environment by natural and/or anthropogenic sources. Natural sources of mercury include volcanoes, forest fires, cinnabar (ore) and fossil fuels, such as coal and petroleum. Regarding anthropogenic sources, a large number of human activities are responsible for mercury in the environment. The anthropogenic sources of mercury are landfills, dental preparations, and combustion processes, such as coal-fired power generation, medicinal waste incinerators and municipal waste combustions. Manufacture of metals, alkali and cement also releases mercury into the environment [13]. Anthropogenic sources depend on human activities at contaminated places. This section describes the sources of mercury into the environment reported in various parts of the world. Zhuang and Gao [14] reported that the higher concentrations of mercury in riverine sediments than those in marine sediments and indicates that the river transportation was the main source of mercury into the southwestern Laizhou Bay of China. Xu et al. [15] reported that combustion and road traffic emissions were the major sources for atmospheric mercury in Xiamen, a coastal city of China. In Columbia, the higher concentrations of mercury were found in water streams, which were carrying loads of mercury from gold mining areas. In gold mining areas, elemental mercury amalgamation has employed to gold recovery [16]. Moreno et al. [17] found that the impact of mercury mines and its concentrations in surrounding atmosphere and water streams. In this study, they used mercury isotopic signatures in lichens and sediments as markers of mercury concentrations in atmosphere. They concluded that mercury mines in Almaden mining district of Spain has a significant impact on mercury concentrations in both the atmosphere and rivers. Kwon et al. [18] found that the watershed runoff was the primary mercury transfer pathway between lake and forest.

Xu et al. [19] revealed that the mercury concentrations in soil have increased by 3-10 times in recent days due to combustion of fossil fuels combined with long-range atmospheric transportation processes. Han et al. [20] found lower concentrations of wet deposited mercury in forest areas of South Korea during summer due to precipitation. The concentrations of mercury in the atmosphere influenced with different seasons. In the atmosphere, coal combustions are the major sources of gaseous elemental mercury but traffic emissions contributes for particulate mercury and domestic pollutants are major sources for reactive gaseous mercury [21].

1.2 Toxicity and Health Implications of Mercury and its Different Species:

Mercury attracts the researchers to determine its concentration in environmental segments due to its toxic nature. A numerous articles were published regarding the toxicity of mercury and its different forms. Yoshida et al. [22] reported the neurobehavioral toxicity in mice while exposed to low-level mercury vapor and methylmercury. Bucio et al. [23] studied the toxicity of mercury in a human hepatic cell line (WRL-68 cells). This study found that the higher doses of mercury show cytotoxic effects with the release of lactate dehydrogenase from the cells. Mercury exposure can causes neurodegeneration with oxidative stress in mitochondria [24]. Occupational exposure of mercury in workers of a fluorescent lamp factory in Egypt causes the symptoms, such as tremors, emotional ability, memory changes, neuromuscular changes and performance deficits in tests of cognitive function [25]. Mercury(II) and methylmercury toxicity can inhibit the human thioredoxin system. Mercury inhibition is selective towards the thioredoxin system; particularly they are bind with selenol-thiol in the active sites of thioredoxin reductase [26]. Methylmercury reacts with the sulfhydryl groups presents in throughout the human body and influences the function of any cellular or subcellular structures. Mercury toxicity in various forms can causes thyroid dysfunction due to associated with the inhibition of 5′deiodonases, spermatogenesis due to accumulation in the testicles and Atrophy and capillary damage in thigh muscles [27]. Tonazzi et al. found a correlation between mitochondrial carnitine/acylcarnitine transporter inactivation and mercury toxicity in animals [28]. Mercury toxicity in humans can causes a numerous neurological or psychiatric disorders not limited to autism spectrum disorders, Alzheimer’s disease, Parkinson’s disease, epilepsy, depression and tremor. In rats, mercury(II) toxicity effects on central neurons and leading to cytoskeleton instability [29]. Exposure to organic forms of mercury, such as ethylmercury and/or methylmercury can causes neurotoxic effects in developing mammals. Ethylmercury exposure in human will occurs due to immunization with thimerosal-containing vaccines [30]. The toxicity of mercury not only limited to neurological effects in humans but also causes vascular effects, such as increased oxidative stress and inflammation, thrombosis, endothelial dysfunction, dyslipidemia, immune and mitochondrial dysfunctions [31]. In overall, the toxicity of mercury in animals and/or humans are effects the cardiovascular, hematological, pulmonary, renal, immunological, neurological, endocrine, reproductive and embryonic systems [32].

Regarding plants, they are exposed to mercury compounds by the administration of anti-fungal agents. The toxicity of mercury affects the seed germination, growth and development in higher plants. It also causes the breakdown of photosynthesis by affecting the chlorophyll and magnesium molecules [33]. Mercury toxicity also induces oxidative stress in growing cucumber seedlings and resulting plant injury [34]. Mercury which is accumulated in different forms in plants can causes phytotoxicity and impairs numerous metabolic processes including nutrient uptake, water status, and photosynthesis [35].

In this present study we reviewed the speciation and determination studies of mercury in different environmental samples using various analytical instruments such as, atomic absorption spectrometer (AAS), voltammeter, inductively coupled plasma optical emission spectrometer (ICP-OES), ICP-atomic emission spectrometer (AES), ICP-mass spectrometer (MS), atomic fluorescence spectrometer (AFS), spectrophotometer, spectrofluorometer, and high performance liquid chromatography (HPLC). For this purpose over 126 research papers published since 2015 in reputed international journals are reviewed. This review clearly predicts the current research about the speciation and determination studies of mercury in all over the world.

2. Reviews regarding the Determination of Mercury:

The toxic nature of mercury and its different species attracts the researchers to find out their concentrations in different environmental samples. Recently, a number of reviews were published about the determination of mercury and describes various factors regarding the concentrations of mercury in environment. This section describes the reviews published regarding the determination of mercury in recent years.

Hanna et al. [36] reviewed the concentrations of mercury in freshwater fishes of Africa. In this study, they reviewed 30 identified studies in which the authors were assembled 407 total Hg concentrations from 166 fish species, 10 types of invertebrates, and various plankton, in 12 countries of Africa. This study concluded that the lack of data about Hg concentrations in African countries and based on the available data they found that the lower Hg concentrations than World Health Organization (WHO) recommendations in commercially available fishes in Africa. Ferreira et al. [37] reviewed about the analytical strategies of sample preparation for the determination of mercury in food samples by using cold vapor atomic absorption spectrometer (CV-AAS), cold vapor atomic fluorescence spectrometer (CV-AFS), inductively couple plasma mass spectrometer (ICP-MS), voltammeter, and neutron activation analysis. Based on the reviewed papers, they concluded that the determination of mercury and their species in food samples with CV-AFS or CV-AAS were very simple and inexpensive than other methods.

Colorimetric and visual assays determination of Hg(II) based on gold nanoparticles, fluorescent gold nanoparticles, gold nanorods, gold nanoflowers and gold nanostars was reviewed by Chansuvarn et al. [38]. This study found that the gold nanoparticles would be the promising luminescent nanomaterials for the detection of Hg(II) due to high selectivity and ultra-sensitivity. Regarding the analytical instruments, UV-visible spectrophotometer would be the cost-effective for limited budget laboratories for the real-time monitoring of Hg(II) in environmental samples. Ariya et al. [39] reviewed physiochemical and biogeochemical transformation of mercury in the atmosphere and atmospheric interfaces. This review describes the analytical methodology for speciation of mercury in the atmosphere, exchange of Hg between the atmosphere and aquatic interfaces, and exchange of Hg between the atmosphere and terrestrial environments. The determination of mercury content in hard coal and fly ash using X-ray diffraction and scanning electron microscopy coupled with chemical analysis was reviewed by Rompalski et al. [40]. This study found that the previously mentioned techniques were proved to be useful for the detection of Hg and identification of the form of its occurrence in coals and furnace wastes. Shrivastava et al. [41] reviewed the detoxification mechanisms of Hg toxicity in plants. This study found that Hg has harmful toxic effects on the molecular and physiobiochemical behavior of plants. Another important prediction of this study is most of the research work was done on seed germination, shoot, root and leaf morphology. Duarte et al. [42] reviewed about the disposable sensors for detection of lead(II), cadmium(II), and mercury(II) in the environment. It describes about the analytical performances, and role of certain factors, such as immobilization procedure and surface modification affecting the analytical characteristics of sensors. This study found that the disposable sensors used for one-shot measurements of lead(II), cadmium(II), and mercury (II) in environmental samples shows that adequate inter-sensor reproducibility, sensitivity and selectivity, and very low detection limit values. The determination of Hg(II) and As(III) using electrochemical methods, especially stripping voltammetry was reviewed by Zaib et al. [43]. This study concluded that the modified carbon paste electrode would be provides better determination of Hg(II) and As(III) due to better deposition with linear and improved response under studied set of conditions. It found the disadvantages of using macro-electrodes was they were expensive and suffered from fouling of surface problem even though they provided better sensitivity and selectivity for the determination of Hg(II) and As(III).

Jagtap and Maher [44] reviewed the measurement of mercury species in sediments and soils by high-performance liquid chromatography (HPLC) coupled with inductively coupled plasma mass spectrometry (ICP-MS). This study recommends the extraction of Hg species in their determination is distillation or using 2-mercaptoethanol. It also recommends usage of C8 stationary phase and 2-mercaptoethanol as mobile phase in HPLC to accurate quantification of methyl mercury in presence of large amounts of Hg(II). Gustin et al. [45] reviewed about the measuring and modelling of mercury in the atmosphere. This study reveals that mercury in the atmosphere can exists in three different forms, such as gaseous elemental mercury (GEM), gaseous oxidized mercury (GOM), and particulate bound mercury (PBM). Among these forms, there is relative confidence in GEM measurements only but about the remaining two forms are less well understood. This study concludes that only through the comparison of multiple calibrated measurements would make the results to be determined were accurate. McLagan et al. [46] reviewed the passive air sampling of GEM in the atmosphere. This study found that the passive air sampling method performance must be validated against to active air monitoring systems with satisfactory precision and accuracy. Jackson and Punshon [47] reviewed the recent advances in the measurements of arsenic, cadmium and mercury in rice and other food materials. It described about the challenges, state-of-art methods, and usage of spatially resolved techniques for arsenic and mercury within rice grains. But this review mainly focused on the determination and speciation studies of arsenic in rice and other food materials. Duan and Zhan [48] reviewed nanomaterials (noble metal nanoparticles, fluorescent metal nanoclusters, semiconductors quantum dots and carbon nanodots) -based optical sensor for Hg(II) detection reported in recent days. It predicts the advantages of using nanomaterials for Hg(II) detection and/or removal over conventional methods, such as high sensitivity, selectivity, simple, rapid and inexpensive. Sun et al. [49] reviewed about the recent progress in detection of Hg using surface enhanced Raman spectroscopy (SERS). This study states that substantial enhancement in detectable Raman signal coupled with unique nanoparticle-based approach makes SERS a powerful tool for the detection of Hg(II). Suvarapu and Baek [50] reviewed the speciation and determination of mercury using various analytical techniques. This study discussed about the research papers published during 2013-2014 about determination and speciation studies of mercury.

3. Discussion

In recent years, a large numbers of research articles are published regarding the determination and speciation of mercury using various analytical techniques. We divided this section into four parts based on the analytical instruments used in the determination of mercury. They are i) Spectrometric instruments (AAS, AFS, ICP-OES, AES, MS) ii) Spectrophotometer and Spectrofluorometer iii) Electrochemical instruments (Voltammeter, Potentiometer), iv) Miscellaneous instruments.

The determination and speciation of mercury using spectrometric instruments, such as AAS, AFS, ICP-OES, AES, MS are presented in Table 1. In Table 2, the studies reported using spectrophotometer and spectrofluorometer are presented. Table 3, represents the determination of mercury using electrochemical instruments and Table 4, the determination of mercury using miscellaneous instruments. In these tables we are incorporated all the analytical figures of merits, such as limit of detection, linearity range, quality control and quality assurance, applicability (analyzed samples) and interference studies reported in the determination of mercury.

Regarding the usage of analytical instruments in the determination studies of mercury as presented in Figure 1, 40% researchers were used spectrometric instruments, such as AAS, AFS, and ICP-OES, AES, and MS, 24.60% researchers were used spectrophotometer, spectrofluorometer, 23.80% of researchers were used electrochemical instruments, such as voltammeter and potentiometer and 12% of researchers were used miscellaneous instruments.

Table 1. Analytical parameters of reviewed research papers about the speciation and determination of mercury by Spectrometric instruments (AAS, ICP-OES, AES, MS and AFS)

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Fluorescence	Dithioacetal-sbubstituted triphenylimidazole	Fluorescence spectrometer	4.3 nM	0-10 µM	Sensitivity and selectivity of the method evaluated	-	Cations, such as Ag^{+}, CO^{2+}, K^{+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Mn^{2+}, Na^{+}, Ca^{2+}, Mg^{2+}, Pb^{2+}, Fe^{3+} and Cd^{2+} were not interfered in determination of Hg^{2+}.	51
Hg(II)	Fluorescence	2-aminoethyl pipera zine and 4-chloro-7-nitro benz-2-oxa-1,3-diazole	Fluorescence spectrometer	0.57 µM	0.00-16.6 µM	The metod is selective over 18 metal ions. Recoveries of Hg(II) in water samples in the range of 95-98.2%.	Water samples	Either no or a small fluorescence signal observed for Na^{2+}, K^{2+}, Mg^{2+}, Ca^{2+}, Al^{3+}, Ga^{3+}, In^{3+}, Cr^{3+}, Mn^{2+}, Fe^{2+},Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+} Cd^{2+} and Pb^{2+} ions.	52
Hg(II)	LLME	-	ET-AAS	5.7 ng L^{-1}	19-450 ng L^{-1}	SRM was analyzed	SRM and spiked water samples	Cd^{2+}, Co^{2+},Fe^{2+}, Mn^{2+}, Ni^{2+}, Pb^{2+}, and Zn^{2+} were not interfere up to 2.00 mg L^{-1}	53
Hg speciation	Green chemical vapor genera - tion	-	AFS	0.005 µg L^{-1}		CRMs (GBW 10029, DORM-4 and TORT-3) were analyzed. Sensitivity and RSD studies were reported	Water and biological samples	Interference of Fe^{3+}, Co^{2+}, Ni^{2+}, and Cu^{2+} were studied. This method was highly sensitive for Hg(II) and ultrasensitive for total Hg and MeHg.	54
GEM	Acid digestion	Teflon filters	ICP-MS	3 ppt	-	SRM 1633c was analyzed. Recoveries were in the range of 84-112%	Particulate matter	-	55
Hg(II)	CVG	$LiAlH_4$, $SnCl_2$/THB	HG-AFS	0.08 µg L^{-1}		The RSD values were less than 7.0% for 11 measurements. CRMs were analyzed.	Soil, water and human hair samples	10 mg L^{-1} for Fe^{3+}, 20 mg L^{-1} for Co^{2+}, 10 mg L^{-1} for Ni^{2+} and 20 mg L^{-1} for Cu2+. For arsenic and antimony, no interference from 25 mg L^{-1} Cu^{2+}, Co^{2+} or Ni^{2+} was observed,	56
Hg(II)	Photochemical vapor generation	-	ICP-OES	1.2 µg L^{-1}	Upto 50 µg L^{-1}	Recoveries of Hg(II) in reals samples were in the range of 79-112%	Petroleum production water	K^{+}, Ba^{2+}, Sr^{2+} , Mg^{2+}, Cu^{2+}, Pb^{2+} and Zn^{2+} over the concentration ranges studied, whereas the presence of Ca^{2+},Ni^{2+}, Sb^{3+},	57

								As^{3+}, Se^{4+}, Fe^{3+} and Cr^{3+} was significant	
Hg(II)	Cold vapor generation	1,5-bis(di-2-pyridil) methylene thiocarbo hydrazide	ET-AAS	7.8 ng L^{-1}	0.099-10 µg L^{-1}	SRM 2976 and CRMs were analyzed. The recoveries were in the range of 97-107%	Biological and sea water samples	No intereference of transitional metals included in SRM in the determination of Hg(II)	58
Hg(II)	Acid digestion	-	ICP-MS	-	0.1-100 µg L^{-1}	Precision of the method for Hg2+ is 5.6% with six replicates	Fish samples	Hg(II) was analyzed along with Cd^{2+} and Pb^{2+}	59
Total Hg	Wet digestion	-	AAS	-	-	-	Fish samples	Hg(II) was analyzed along with Cd^{2+} and Pb^{2+}	60

LLME: Liquid liquid micro extraction; CRM: Certified reference material; CVG: Chemical Vapor Generation; THB: Tetra Hydro Borate; RSD: Relative Standard Deviation;

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg speciation	Acid digestion	-	ICP-MS	40 ng/kg	-	NIST 612 was analyzed	Petroleum hydrocarbons	-	61
Hg speciation	Magnetic solid phase extraction	Magnetic nano particles	ICP-MS	1.6 (MeHg), 1.9 (THg) ng L^{-1}	5-10,000 (MeHg), 10-10,000 (THg) ng L^{-1}	The RSD values for 7 replicates was less than 3.4%	Water and human hair samples	Tolerance of Al^{3+}, Zn^{2+} (500 mg L^{-1}) Fe^{3+} (100 mg L^{-1}) for Hg determination	62
Total Hg	Chemical vapor generation	Non-ionic surfactants	AFS	4.5 ng L^{-1}	-	CRMs (GBW08603, GBW(E) 080401 and GBW(E)080402) were analyzed	Natural water samples	Severe interference of cations was observed at 10 mg L^{-1} concentration	63
Total Hg	Sequential extraction	-	CV-AAS	-	-	Comparison of the results with other methods was performed	Thar coal	-	64
Hg(II)	Aptasensor	Core-shell Ag@SiO2 nanoparticles	AFS	0.33nM	0-1.2, 1.2-14 nM	Recoveries were over 94% for the determination of Hg(II) in real samples. The RSD values for Hg(II) determinations were lower than 5.1%.	Real water samples	Selective in presence of Cd^{2+}, Pb^{2+}, Zn^{2+}, Mn^{2+}, Co^{2+}, Fe^{2+}, Cu^{2+} and Ag^{+}	65

Hg(II)	Biosensor	Lanthanide coordination polymers	Luminescence spectrometer	0.16nM	-	Recoveries were in the range of 96.2-99.8% in real samples	Drinking water and human urine	No interference of Cd^{2+}, Fe^{3+}, Fe^{2+}, Cr^{3+}, Ni^{2+}, Mn^{2+}, Co^{2+}, and Cu^{2+} was observed	66
Hg(II)	Cehmosensor	Porphyrin-thymine conjugates	Luminescence spectrometer	6.7 nM	-	Method reversibility was reported	-	Interference of Zn^{2}+, Cu^{2+}, Ni^{2+}, Mn^{2+}, Mg^{2+}, Pb^{2+}, and Cd^{2+} was inhibited	67
Hg speciation	Solid phase extraction	Polymer	AFS	500 ng L^{-1}	-	CRM (BCR-464) was analyzed	Fish and sea water samples	Mercury retention was uneffected by the presence of Cu^{2+}, Cd^{2+}, Pb^{2+}, As^{5+} and Se^{6+} ions	68
Hg	PVG and CV	NaBH$_4$ / SnCl$_2$	PVG-AAS, CV-AAS (NaBH$_4$) and CV-AAS (SnCl$_2$)	0.12, 0.11 and 0.04 μg L^{-1}	2.5-10.0 μg L^{-1}	The accuracy was evaluated by assessing recoveries in spiked samples which were in the range of 84-108.3%	Glycerin samples	-	69
Total Hg	Solid sampling analysis	-	GF-AAS	1.4 ng g^{-1}	-	SRM (NIST-695) was analyzed. The RSD values were better than 8.2% for five replicates	Phosphate fertilezers	-	70
DGM, TGM	-	-	CV-AFS	2.7 pg L^{-1} (DGM), 0.03 ng m^{-3}(TGM)	-		Air-sea interface of Minamata	-	71
Methyl Hg	Distillation and solvent extraction	KBr/CuSO$_4$	GC-ICP-MS	0.02 μg Kg^{-1}	-	CRM of ERM-CC580 was analyzed	Peat soil	-	72

PVG: Photochemical vapor generation; CVG: Cold vapor generation; DGM: Dissolved gaseous mercury; TGM: Total gaseous mercury;

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Total Hg	Ultra-sound extraction	NaBH$_4$/isoamyl alcohol, thiourea	CV-AAS	14 ng L^{-1}	-	The RSD values of Hg determinations in vinegar was less than 8.11%	Vinegar	Vanadium also determined in the same samples	73
Total Hg	Slurry sampling		CV-AAS	30 ng L^{-1}	-	The RSD values in the mercury determinations was less than	Iron suplement	-	74

						10.89%. The recovieries were in the range of 85-106%			
Total Hg		$NaBH_4$	AES	0.04 ppb	-	-	Sea food	-	75
Hg speciation	Diffusive gradient technique	2-mercaptoethanol	CV-AFS	38 (MeHg), 13 (Hg^{2+}), 34 (EtHg), 30 (PhHg) ng L^{-1}	-	The extraction efficiency was higher than 95% and RSD value was 3.5%	Ambersep and Duolite resin gels	-	76
Hg(II)	Preconcentration	Metal-organic frame work	CV-AAS	0.01 ng mL^{-1}	-	SRMs (DOLT-4 and DORM -2) were analyzed. The RSD values in the determination of Hg(II) was less than 10%	Sea food samples	Majority of cations were not interfere in the determination of Hg(II) at pH 6.25 experimental condition	77
MeHg	ISO-17025	2-mercaptoethanol, sodium thiosulphate	GC-py-AFS	0.45 pg	1-800 pg	CRM (IAEA-405) was analyzed and recoveries were in the range of 94-96%.	Marine sediment samples	Selectivity was compared with GC-py-AFS with HPLC-ICPMS and concluded that no interference of other metal ions	78
Total Hg	Solid sampling	-	HR-AAS	0.025 ng (sediment), 0.096 ng (marine biota)	0.025-4 ng (sediment), 0.06-25 ng (Marine biota)	CRMs (PACS-2, IAEA-405, SRM 2703, BCR-464, IAEA-436, DORM-2, MA-ROPME-2/TM) were analyzed	Sediment and marine biota samples	-	79
Total Hg	Thermal desorption	-	AAS	0.6 ng g^{-1}	-	CRMs (CRM-1515, MESS-3 and TORT-2) were analyzed with recoveries 96.0-104.8%	Fish and sea food samples	-	80
Hg speciation	Solid phase extraction	2-mercaptoethane-sulphonate	ET-AAS	0.01 µg L^{-1} (total Hg)	-	CRMs (SRM 1515, SRM 1572, NRC-CNRC DOLT-2, NRC-CNRC DORM -2) were analyzed	Fish edible oil	-	81
Total Hg	ISO guide 34	-	CV-ICP-MS	0.8 ng/kg	1.9-50 ng/kg	CRM (BCR-579) was analyzed	Sea water	By using cold vapor generation spectral interferences were avoided	82
Total Hg	Acid digestion	Nitric and perchloric acid	AAS	0.4749 ng/g	0.05-2.0 µg L^{-1}	The mean recovery of Hg was 78.65%. RSD values for interday precision of Hg was	Cream cosmetics	Along with Hg, Zinc was also determined	83

						7.17%			
Total Hg	Extraction	$NaBH_4$	CV-AAS	0.6 µg L^{-1}	10-100 µg L^{-1}	The recoveries of Hg in oil samples were in the range of 80-103%	Oil samples	-	84

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Total Hg	Preconcentration	-	ICP-MS	0.37 pg	-	Precision was less than 7% RSD. CRMs (NIST-2709, NIST-1547 and DORM-3) were analyzed	Sediments, leaves and fish muscle samples	Amalgamation step isolates only Hg and there was no interferences of other ions	85
Total Hg	Thermal desorption	-	TDA-AAS	25.0 µg/kg (LOQ)	-	Mean recovery of Hg in real samples was 94.2% and SD was 3.5%	Sea food	-	86
Total Hg	Method EPA 7473	Teflon	CV-AFS	0.6 ng g^{-1}	2-80 ng g^{-1}	CRM (BCR-279) was analyzed and RSD in the determination of Hg in seaweeds is less than 10%	Seaweeds	-	87
Total Hg	Ultrasound-assisted treatment	-	CV-AAS MIP-OES	MIP-OES has lower detection limit		Recoveries by both methods were greater than 92% and RSD values were less than 10%	Inorganic fertilizers	Even 100 folds of Cu, Zn, Fe, Mn, Pb, Cd, Cr, and As were not interfere in the determination of Hg	88
Hg(II)	Immobilization	Dithizone	FAAS	2.0×10^{-9} M	1.1×10^{-8} -2.0×10^{-6}M	The coefficients of variation for Hg(II) was found to be 2.7%	Industrial wastewaters Spiked tap water and natural waters	Except Cu^{2+}, the other ions (Mn^{2+}, Ni^{2+}, Pb^{2+}, Co^{2+}, Cd^{2+}, Fe^{2+}, Fe^{3+}, and Al^{3+}) were not interfere upt0 50 fold excess	89
Total Hg and isotopic composition	Purge and trap Preconcentration	-	ICP-MS	-	-	NIST SRM 3133 was analyzed	Dissolved Hg in seawater	Purge and trap method was free from matrix interferences	90
MeHg	Online preconcentration	-	HPLC-CV AFS	8 pg L^{-1}	1-500 ng L^{-1}	CRMs (NIES CRM no. 13 and IAEA-085) were analyzed. Recoveries of MeHg from real	Sewage, river and seawater samples	-	91

						samples were in the range of 91.4-101.8%			
Hg(II)	Solid phase extraction	Ion imprinted polymeric nanomaterials	CV-AAS	0.036 ng mL^{-1}	-	The RSD values for eight replicates was 4.2%	Water and Human hair samples	In presence of large amounts of Cu^{2+}, Ni^{2+}, Cd^{2+}, Zn^{2+}, Mn^{2+}, Pb^{2+}, Fe^{3+}, and Cr^{3+} ions Hg(II) was effectively determined	92
Hg(II), MeHg	Rapid extraction	-	HPLC-ICPMS	0.2 (Hg2+), 0.1 (MeHg) ng g^{-1}	-	CRMs(TORT-2 and DORM-2) were analyzed	Fish samples	-	93
Hg(II)	Solid phase extraction	Silver nano particles	ET-AAS	0.005 ng mL^{-1}	Up to 20 ng mL^{-1}	CRMs (TMRAIN-04, ERM-CA011b) were analyzed. The RSD values for real water samples was 6-11%	Spring, tap and groundwaters	-	94
Speciation	Liquid-liquid microextraction	Ionic liquid vortex-assisted	HPLC- CV-AFS	0.037-0.061 ng/g	0.1-70 ng/g	The RSD values were less than 6.4%	Sediment samples	-	95

LOQ: limit of quantification; MIP-OES: microwave-induced plasma optical emission spectrometer

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg speciation	Magnetic solid phase micro extraction	Fabricated chip	HPLC-ICPMS	18.8 (Hg^{2+}), 12.8 (MeHg), 17.4 (EtHg), 41.8 (PhHg) ng L^{-1}	0.05-20 ng mL^{-1}	The RSD values for the determination of Hg species were within 6.7%. The recoveries were in the range of 85-115%.	Incubated HepG2 cells	The interference of K^+, Na^+, Ca^{2+}, Mg^{2+}, Fe^{3+}, Al^{3+}, Cl^-, NO_3^-, SO_4^{2-}, HCO_3^-, and PO_4^{3-} were negligible in the determination of Hg species	96
Hg speciation	Liquid-liquid micro extraction	Hollow fiber	HPLC-ICPMS	2.95.6 ng L^{-1}	-	CRM (DORM-2) was analyzed and the RSD values were in the range of 5.6-10.6%	Lake water, finsh, plant and sediment	-	97
Hg speciation	Adsorption	Enrichment nanosorbent	CV-ICPMS	0.002-0.004 µg L^{-1}	0-70 µg L^{-1}	CRMs (LGC 6016, SRM 2976) were analyzed	Seawater samples	No interferences of Na^+, K^+, Ca^{2+} and Mg^{2+} was observed	98
Total Hg	Solid sampling	-	TDA-AAS	1.0 µg kg^{-1}	25.0-200.0 µg kg^{-1}	Recoveries of Hg from real samples were in the range of 89-99%	Fish and shrimp samples	-	99

Total Hg	Solid phase adsorption	Silica monoliths	CV-AFS	1.31 ng L^{-1}	1.31-10,000 ng L^{-1}	The RSD values were less than 5.37%	Natural waters	Gold nanoparticles deposition served as selective solid-phase adsorbent for Hg	100

Analytical Instruments: ; CV-AAS: Cloud Vapor Atomic Absorption Spectrometer; GF-AAS: Graphite Furnace AAS; ICP-OES: Inductively Coupled Plasma Optical Emission Spectrometer; ICP-MS: ICP-Mass Spectrometer; ICP-AES: ICP-Atomic Emission Spectrometer; HPLC: High Performance Liquid Chromatography; AFS: Atomic Fluorescence Spectrometer;

Table 2. Analytical parameters of reviewed research papers about the speciation and determination of mercury by Spectrophotometer and Spectrofluorometer

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Chemiluminescence	Carbon nitride quantum dots	Spectrofluorometer	0.08 ng mL^{-1}	0.25-10 ng mL^{-1}	Analyzed SRM 1566b. The recoveries of analyzed samples were with 95.7-102.8%	Water, food spiked samples	Interference of Fe^{3+},Co^{2+}, Ni^{2+}, Cu^{2+},Cr^{3+}, Zn^{2+}, Pb^{2+}, Cd^{2+}, Mn^{2+} and Al^{3+}were reported and Cu^{2+} having low tolerance limit as 100 folds than other	101
Hg(II)	Fluorescence	Gold nano particles	Spectrofluorometer	31 nM	0.16-1.60 μM	Recoveries of Hg(II) in water samples were in the range of 95.7-103.6% and RSD were less than 5.0%.	Drinking water	Co^{2+}, Ba^{2+},Pb^{2+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+}, Mg^{2+}, Ca^{2+}, Zn^{2+}, Ag^{+}, Na^{+}and K^{+} were not interfered up to 12.8 μM concentrations	102
Hg speciation	Cloud point extraction	Polyethylene glycol	Spectrophotometer	9 μg L^{-1}	10-100 μg L^{-1}	The RSD values of the method were below 2.6%	River water and river sediment	No interference of Cd2+, Bi3+, and Pb2+ were observed in the determination of Hg(II)	103
Hg(II)	Fluorescence	Poly(arylene ether nitrile)	Spectrofluorometer	10 nM	-	The RSD values of the method were below 7.05%	Spiked tap water and lake water	The method was selective towards the presence of Ag^{+}, Ba^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Mg^{2+}, Pb^{2+}, Zn^{2+},Al^{3+}, and Fe^{3+} ions.	104
Hg(II)	Chemiluminescence	Rhodamine B	Spectrophotometer	0.013-4.67 μg L^{-1}	5.42 ng L^{-1}	The RSD values were about 0.7-2.1% for different concentrat- ions of Hg	Tap, rain, river water samples	The tolerance limits for Cr^{3+}, Co^{2+}, Zn^{2+}, Ni^{2+} and Mn^{2+} was 400 folds than Hg^{2+} and for Cd^{2+}, and Pb^{2+}, it was 100 folds.	105
Hg(II)	Colorimetric	1,5-diphenyl thiocarbazone	Flow injection spectrophotometer	0.03 μg mL^{-1}	0.05-1.50 μg mL^{-1}	The results were compared with the data obtained with ICP-MS	Cosmetics and Thai traditional medicines	20 folds of Fe^{2+}, Zn^{2+}, and Cu^{2+}, 40 folds of Pb^{2+}, 50 folds of Al^{3+}, Fe^{3+} and Mn^{2+} were not	106

								interfere in the determination of Hg(II)	
Hg(II)	Colorimetric, fluorescence	Calixpyrrole hydrazide	Spectrofluorometer	1 nM/L	1nM/L – 1μM/L	-	Ground water and industrial effluent water	No interference of Pb(II), Cd(II), Mn(II), Fe(III), Ni(II), Zn(II), Hg(II), Co(II), Cu(II) was observed	107
Hg(II)	Fluoroscence	Gold nanoclusters	Spectrofluorometer	58nM	4.95× 10^{-7} -8.36×10^{-6}M	Recoveries were in the range of 97-102% and RSD values were lower than 3.1%.	Tap and river water	The method was selective towards Li+, Na^+, K^+, Ba^{2+}, Ca^{2+}, Cd^{2+},Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Mg^{2+}, Pb^{2+}, $Al^{3+}$and Fe^{3+}.	108
Hg(II)	Plasmon sensors	Ag-Au alloy nanoparticles	UV-Vis spectrometer	0.01 ppb	0.02-100 ppb	Recoveries of Hg in real samples were in the range of 98-111%	Tap water, river water, milk, facial cleanser	Cations such as, Fe^{3+}, Co^{2+}, Pb^{2+}, Al^{3+}, Cu^{2+}, Ni^{2+}, and Zn^{2+} were not interfere even up to 10 ppm levels	109
Hg(II)	Fluoresence probe	Chitosan hydrogel	Fluoroscence spectrophotometer	0.9 nM	5.0-50nM	-	Water samples	Cations such as, Fe^{3+}, Co^{2+}, Pb^{2+}, Cu^{2+}, Cd^{2+}, Ni^{2+}, and Zn^{2+} were not interfere in the determination of Hg(II)	110
Hg(II)	Fluoresence sensor	CdTe quantum dots	Fluoroscence spectrophotometer	4.0 nM	6.0-450nM	RSD values were less than 4.15%	Lake water samples	Interference of 10-fold Pb^{2+}, Cu^{2+}, and Ag^+ was less than 7%	111

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Fluorescent chemosensor	DA	Fluoroscence spectrophotometer	2.8 ppb	-	Theoretical and experimental results were in good agreement with each other	-	Simultaneous determination of Ag+, Cu2+ was reported	112
Hg(II)	Time-gated fluorescent sensing	Thymine	Spectrofluorometer	0.11 nM	0.20-10 nM	Recoveries of Hg(II) in environmental water samples were in the range of 93.75-102.5%	Drinking water samples	No interference of Ag+, Co^{2+}, Ni^{2+}, Ca^{2+}, Cd^{2+}, Al^{3+}, Fe^{3+}, Au^{3+}, Cr^{2+}, Mn^{2+}, Pb^{2+}, Cu^{2+}, Mg^{2+}, Zn^{2+}, and $Ba^{2+}$was reported	113
Hg(II)	Fluorescent sensor	Calixarene	Fluoroscence spectrophotometer	5.56× 10^{-7} M	-	The method results were compared with confocal fluorescence microscope	Living cells (humans)	Li^+, Na^+, Cs^+, Ag^+, Ca^{2+}, Mg^{2+}, Sr^{2+}, Ba^{2+}, Zn^{2+}, Ni^{2+}, Cu^{2+},Cd^{2+}, Co^{2+}, Mn^{2+}, Pb^{2+},	114

								Fe^{2+}, Fe^{3+}and Al^{3+} were not interfere in the determination of Hg^{2+}	
Hg(II)	Colorimetric	Gold nano particles	Spectrophotometer	0.5 nM	0.5-300 nM	CRM (GBW (E) 080392) was analyzed and the recoveries were found in the range of 88.9-106%	Tap water and lake water	Cr^{3+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Fe^{3+}, Zn^{2+}, Cd^{2+}, and Pb^{2+} ions were not interfere in the determination of Hg^{2+}	115
Hg(II)	fluorescence	Schiff base	Fluoreswcence spectrophotometer	2.82× 10^{-6} M	-	Effectiveness of the method was proved by confocal fluorescence microscope	Living cells	-	116
Hg(II)	Colorimeteric	Silver nanoparticles	Spectrophotometer	1.18× 10^{-9} M	10-50 nM	A good linear correlation (R^2= 0.9799) was obtained for differernt concentrations of Hg(II) and absorbance ratio	Lake, seawater and groundwaters	Fe^{2+}, Fe^{3+}, Cr^{6+}, Pb^{2+},Mn^{2+}, Al^{3+}, Ni^{2+}, Cr^{3+}, Cd^{2+}, Mg^{2+}, and Zn^{2+} were not interfered upto 1000 times of detection limit of Hg(II)	117
Hg(II)	Chemosensor	Dimerci binol	Spectrofluorimeter	4.4× 10^{-7} M	-	-	Wastewater samples	100 equivalents of Na^{+}, K^{+}, Mg^{2+}, Ce^{3+}, Ca^{2+}, Ba^{2+}, Cd^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Cr^{3+}, Zn^{2+}, Pb^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+} and Ag^{+}	118
Hg(II)	Colorimetric	Bovine serum albumin	Spectrophotometer	7.2 nM	0-120 nM	Results were compared with ICP-MS	Drinking water samples	No interference of Na^{+}, Mg^{2+}, Ca^{2+}, Cd^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Fe^{3+}, and Au^{3+} was observed	119
Hg(II)	Flouroscence	Gold nano cluster	Spectrofluorometer	30 nM	-	Recoveries of Hg(II) in spiked samples were in the range of 97.7-99.3%	Lake water samples	No interference of Na^{+}, Mg^{2+}, Ca^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Ce^{3+}, Pt^{4+} and Al^{3+} was observed	120
Hg(II)	Colorimetric	Rhodamine B	Spectrofluorometer	1.71× 10^{-6}M	-	-	Spiked tap water samples	Interference of several ions was negligible in the determination of Hg(II)	121
Hg(II)	Visual optode sensor	Rhodamine 6G	Spectrophotometer	1.3 ng mL^{-1}	10-5000 ng mL^{-1}	The recoveries of Hg(II) in water samples was in the range of 96-100%	Groundwater samples	Except Cu2+, the other ions, such as Cd^{2+}, Cr^{3+}, Fe^{2+}, Fe^{3+},	122

		derivative						Zn^{2+}, Al^{3+}, Pb^{2+}, and Ni^{2+} were not interfere	

DA: Dimethylaminocinnamaldehyde-aminothiourea

Analyte	Method	Supporting media	Analytical Instrument	LOD	Linearity range	QA/QC studies	Analyzed Samples	Interference study	Ref
Hg(II)	Colorimetric	Rhodamine-thiosemicarbazide	Spectrofluorometer	10^{-8} M	2.5× 10^{-7} -2.0×10^{-5}M	-	Aqueous media	No interference of Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Ag^{+}, Cd^{2+}, Pb^{2+}, Al^{3+}, and Fe^{3+} was observed	123
Hg(II)	fluorescence	-	Fluorescence spetrometer	9.56× 10^{-9} M	-	-	-	Hg(II) can be detectable in presence of Fe^{3+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, and Cr^{3+}.	124
Hg(II)	Colorimetric	PPT	Fluorescence spetrophotometer	1.86 nM	-	Recoveries of Hg(II) in real samples were inthe range of 97.43-97.92%	Fish samples	No interference of As^{3+}, Pb^{2+}, Cd^{2+}, Cu^{2+} and Ni^{2+} was observed	125
Hg(II)	Adsorbance	Rhodamine	Fluorescence spectrophotometer	3.42× 10^{-6} M	0-6.0 μM	-	Drinking and lake waters	No interference of Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, Pb^{2+}, and Zn^{2+} at 581 nm	126
Hg(II)	Fluorimetric	Coumarinyl dithiolane	Fluorescence spectrophotometer	-	0.06-1.5 μM	-	Aqueous solutions	No influence of Al^{3+}, Zn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Cd^{2+}, Cr^{3+}, and Pb^{2+} on thedetermi-nation of Hg(II) in presence of probe	127
Hg(II)	Colorimetric	LSPR	Spectrophotometer	0.2 nM	5.0-10.0 μM	The RSD value for triplicate measurements was 4.81%	Wastewater samples	The cations, such as Al^{3+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{3+}, Ni^{2+}, Pb^{2+}, and Sr^{2+} were not interfere in the determination of Hg(II)	128
Hg(II)	Fluorescence sensors	Peanut shell	Fluorescence spectrometer	8.5× 10^{-9} M	0-19× 10^{-8} M	-	Lake waters	The method was selective for Hg(II)	129
Hg(II)	colorimetric	L-Arginine	Spectrophotometer	5 nM	1-20 and 20-600 μM	-	Food samples	No interferences from Cd^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, and Pb^{2+} was observed	130

PPT; poly (1,4-bis-(8-(4-phenylthiazole-2-thiol)-octyloxy)-benzene); LSPR: localized surface plasmon resonance

Table 3. Analytical parameters of reviewed research papers about the speciation and determination of mercury by electrochemical instruments

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Biosensor	Y-shaped DNA	Square wave voltammeter	0.094nM	1nM-5µM	Selectivity, sensitivity and repeatability were studied	River water samples	Interferences of Cu^{2+}, Al^{3+}, Co^{2+},Fe3^{+}, Zn^{2+}, Ni^{2+}, Cd^{2+}, Ba^{2+}, Cr^{3+}, Mg^{2+}, and Pb^{2+} were reported	131
Hg(II)	Preconcentration	N-octylpyridinium	Stripping Voltammeter	0.3 µg L^{-1}	0-100 µg L^{-1}	The RSD of the method was 10%	Tap, Pond and wastewaters	No significant interference of 100 µg L^{-1} of Cu^{2+}, Pb^{2+}, Cd^{2+} and Zn^{2+} was observed in the determination of Hg^{2+}	132
Hg(II)	Electrochemical	Screen printed carbon electrode	Anodic stripping voltammeter	1.02 µg L^{-1}	1-100 µg L^{-1}	Accuracy of the method was evaluated with ICP/MS	Ground water	Interference of Cu^{2+}, Co^{2+},Fe^{2+}, Zn^{2+}, Ni^{2+}, Cd^{2+}, Mn^{2+}, Mg^{2+}, and Pb^{2+} were negligible in the determination of Hg^{2+}	133
Hg(II)	Electrochemical sensor	1-(2, 4-dinitro phenyl)-dodecanoyl thiourea	Cyclic, square wave and differential pulse voltammeter	0.64 µg L^{-1}	Upto 2 µg L^{-1}	The RSD of the method was 3.5%	Drinking and tap water samples	5 folds of Cu^{2+}, Cd^{2+}, Pb^{2+},and Zn^{2+} were not interfered in the determination of Hg(II)	134
Hg(II)	Electrochemical	N-PC-Au	Anodic stripping voltammeter	0.35 nM	0.001-1 µM	-	Drinking water	The electrode was not affected by the presence of Zn^{2+}, Pb^{2+}, Cu^{2+} and Cd^{2+} ions in the determination of Hg(II)	135
Hg(II)	Electrochemical sensor	Modified gold nano particles	Cyclic voltammeter	1.5 ng L^{-1}	1.0-10 ng L^{-1}	-	Spiked water samples	The method is selective towards the presence of Zn^{2+}, Cd^{2+}, Pb^{2+}, Cu^{2+}, Ni^{2+}, and Co^{2+} ions.	136
Hg(II)	Electrochemical	N-doped graphene electrode	Differential pulse voltammeter	0.05 µM	0.2-9 µM	The RSD of Hg determination with six repetetions was 2.1%		Simultaneosly Cd^{2+}, Cu^{2+}, Pb^{2+} were determined along with Hg^{2+}	137
Hg(II)	Electrochemical sensor	Screen printed carbon electrode	Differential pulse Anodic stripping voltammeter	0.021 µg L^{-1}	0.05-2.0 µg L^{-1}	Recovery of Hg(II) was found as 106%	Real water samples	High tolerance limits were observed for Fe^{3+}, Zn^{2+} and Cd^{2+} but lower tolerance limits for Pb^{2+} and Cu^{2+} were found.	138

Hg(II)	Electrochemical sensor	DNA probe	Cyclic and square wave voltammeter	5.6 nM	10-100 nM	-	-	10 folds of Pb^{2+}, Mn^{2+}, Zn^{2+}, Ni^{2+}, Cu^{2+}, Fe^{2+}, Ba^{2+}and Cd^{2+}were not interfere in the determination of Hg(II)	139
Hg(II)	Electrochemical	Carbon ionic liquid paste electrode	Anodic stripping voltammeter	0.1nM	0.5-10nM and 0.08-2 µM	-	Wastewater samples	Over 30 folds of Zn2+, Cr3+, Pb2+, and over 45 folds of Cd2+, Cu2+, Ni2+ and Mn2+ were interfere in the determination of Hg(II).	140
Hg(II)	Electrochemical	Carbon paste sensor	Potentiometer	1.95× 10^{-9} M	4.00× 10^{-9} -1.30×10^{-3}M	Reproducibility of the method was reported	Water samples	Selective coefficients of various cations for Hg(II) selective sensors was reported	141

N-PC-Au: Nitrogen-doped porous carbon-gold nano composite.

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Biosensor	Thymine	Differential pulse and cyclic voltammeter	0.08 nM	0.5-5000 nM	Recoveries of Hg(II) in real samples were in the range of 96.4-103%	Water and human serum	Selective in presence of Al^{3+}, Ba^{2+}, Cd^{2+}, Co^{2+}, Cr^{3+}, Fe^{3+}, Mn^{2+}, Pb^{2+} and Zn^{2+}	142
Hg(II)	Biosensor	Cyclic dithiothreitol	Cyclic voltammeter	28 pM	0.1 nM- 5 µM	Recoveries of Hg(II) in water samples were in the range of 98.8-104%	River water samples	Excellent selectivity for Hg(II) detection was observed in presence of Cd^{2+}, Pd^{2+} and Co^{2+}	143
Hg(II)	Biosensor	Methylene blue	Cyclic voltammeter	8.7× 10^{-11} M	1.0× 10^{-10} - 5.0× 10^{-7} M	The RSD of the sensor was 5.25% for 10 replicates indicates the good reproducibility	Tap and river water samples	Cd^{2+}, Ba^{2+},Pb^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+}, Ca^{2+}, Co^{2+}, Mg^{2+}, Ag^{+} were not interfere upto 250nM in presence of 50 nM of Hg(II)	144
Hg(II)	Electrochemical	PVC membrane sensor	Potentiometer	3.2× 10^{-9} M	1.0× 10^{-8} - 5.0× 10^{-3} M	RSD values for synthetic samples measurements were less than 3.10%	Wastewater samples	The selectivity co-efficient for various ions were in the range of 1.0× 10^{-4}-4.5× 10^{-4} M	145
Hg(II)	Biosensor	B-cyclodextrin	Differential pulse and cyclic voltammeter	5.0× 10^{-15} M	1.0× 10^{-14}- 2.0× 10^{-12} M	SRM of water sample GSB04-1729-2004 was analyzed	Tap and rain waters	100 times of Cd^{2+}, and Pb^{2+}, 200 times of Ni^{2+} and Ag^{+}, 300 times of Zn^{2+}, Cu^{2+}, Cr^{3+} and Co^{2+} were not interfere in the determination of Hg^{2+}	146
Hg(II)	Electrochemical	Copper film	Anodic stripping	0.1 µg L^{-1}	10-100 µg	The RSD values for 12	-	Simultaneously mercury and lead	147

		electrode	voltammeter		L^{-1}	replicates of Hg determination was 4.5%		are determined.	
Hg(II)	Electrochemical	Carbon nanotubes	Anodic stripping voltammeter	0.025 μM	0.1-100 μM	The RSD value for six replicates was 1.93%	River and industrial waste waters	Up to 200 folds of Pb^{2+}, Cu^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+} and Mn^{2+} were not interfere in the determination of Hg(II)	148
Hg(II)	Electrochemical sensor	Mesoporous carbon nano fibre	Anodic stripping voltammeter	0.3 nM	5-500 nM	The RSD values in the determination of Hg(II) in real samples was less than 2.3%	Yellow reiver, China	The proposed electrode avoid the interferences of Cd^{2+}, Pb^{2+} and Cu^{2+}	149
Hg(II)	Potentiometric sensor	MWCNTs	Potentiometer	3.1× 10^{-9} M	4.0× 10^{-9} - 2.2× 10^{-3} M	The recoveries of Hg(II) were in the range of 99-102%	Aqueous samples	The proposed has high selectivity towards the determination of Hg(II) with the infeted by some ions in aqueous samples	150
Hg(II)	Electrochemical biosensor	Polythymine-methylene blue	Square wave voltammeter	0.1 nM	0.2-100 nM	The RSD values for the determination of Hg(II) in real samples were less than 9.6%	Water and fish samples	The voltammetric signal of Hg(II) was 5-10 times higher than other metal ions and makes the method highly selective	151
Hg(II)	Electrochemical	Rotating silver electrode	Square wave voltammeter	4.61× 10^{-8} M	1.0× 10^{-7} -8.0× 10^{-4} M	The RSD for seven replicates was 2.19%	Milk and breast milk	No interferences of copper, cobalt, iron and zinc were observed	152
Hg(II)	Electrochemical	Graphene modified with silver	Differential pulse voltammeter	3.38× 10^{-8} M	5.0× 10^{-8} -1.0× 10^{-4} M	The RSD for eight replicates was 2.25%	Leachate samples	Even 200 times excess of Al^{3+}, Cd^{2+}, Co^{2+}, Ni^{2+}, Pb^{2+}, Fe^{2+}, Fe^{3+}, and Zn^{2+} were not interfere	153

MWCNTs: multi-walled carbon nanotubes;

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Hg(II)	Electrochemical	Graphene oxide	Cyclic voltammeter	0.035 nM	0.1-100 nM	The RSD value in the reproducible test was 4.5%.	River water samples	Even 10 times higher concentrations of Co^{2+}, Mn^{2+}, Pb^{2+}, and Fe^{3+} were not interfere in the determination of Hg(II)	154
Hg(II)	Electrochemical	Gold nano particles	Differential pulse anodic stripping voltammeter	0.03 μg L^{-1}	0.1-10 μg L^{-1}	Recoveries of Hg(II) in real samples was in the range of 87-	Tap and lake waters, milk and soils	1000 folds of Zn^{2+}, Cd^{2+}, Pb^{2+}, Mn^{2+}, Co^{2+}, and Cu^{2+} were not interfere in the determination of Hg(II)	155

						102%			
Hg(II)	Electrochemical	Gold nano particles	Stripping Voltammeter	1 μm L^{-1}	-		Water samples	-	156
Hg(II)	Electrochemical	Graphene-Au modified electrode	Square wave voltammeter	0.001 aM	1.0 aM-100 nM	The RSD values for triplicate measurements was less than 4.46%	Spiked tap and river waters and landfill leachate samples	Even 500 nM of Cd^{2+}, Co^{2+}, Cr^{2+}, Cu^{2+}, Mn^{2+}, Ni^{2+}, Pb^{2+}, Zn^{2+}, Al^{3+}, and Fe^{3+} were not interfere in the determination of 10 nM of Hg(II)	157
Hg(II)	Electrochemical	Graphene/CeO$_2$	Differential pulse anodic stripping voltammeter	2.187 × 10^{-11} M	0.002-0.12 μM	-	Wastewaters	Simultaneously Cd^{2+}, Pb^{2+}, Cu^{2+}, Hg^{2+} were determined	158
Hg(II)	Electrochemical	Graphene quantum dots	Anodic stripping voltammeter	0.02 nM	0.02-1.5 nM	Recoveries from spiked samples were in the range of 96.6-101%	Spiked samples	Cu^{2+} was also determined along with Hg(II)	159
Total Hg	Liquid-liquid microextraction	Screen printed carbon electrodes	Square wave anodic stripping voltammeter	0.2 μg L^{-1}	0.5-10 μg L^{-1}	The recoveris in the determination of mercury in real samples were in the range of 95-108%	Tap, river, bottled and industrial waste waters	-	160
Total Hg	Electrochemical sensing	Zinc oxide quantum dots	Linear sweep voltammeter	5 ppb	5 ppb- 50 ppm	-	River and groundwaters	Except Cd^{2+}, the other ions, such as Zn^{2+}, Pb^{2+}, and As^{3+} were not interfere	161

Table 4. Analytical parameters of reviewed research papers about the speciation and determination of mercury by miscellaneous techniques

Analyte	Method	Supporting media	Analytical Instrument	LOD	Linearity range	QA/QC studies	Analyzed Samples	Interference study	Ref
Hg(II)	Colorimetric	Gold nano-particles	Dark-field microscope	1.4 pM	-	Recoveries were 98.3 and 110.0% for river and industrial waste water, respectively	River and industrial wastewater	25nM concentrations of Pb^{2+}, Ni^{2+}, Fe^{2+}, Cd^{2+}, Zn^{2+}, Co^{2+}, and Mn^{2+} were not interfere in the determination of Hg(II)	162
Hg(II)	Electrochemi luminiscence	Gold nano-particles	Potentiostat PG340	5.1pM	-	Results were compared with AFS measurements	Tap and lake waters	The method was selective for Hg determination in presence of Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}, Mg^{2+}, Mn^{2+}, Pb^{2+}, Al^{3+}, Fe^{3+}	163
Hg(0)	Thermal desorption	-	Direct Milestone Analyzer	-	-	Accuracy was verified with testing the SRM (NIST-2711) and CRM (GBW-GBW 08301 RCV 8221	Soil samples	-	164
Hg(II)	Electrochemi luminiscence	γ-poly glutamic acid-grapheme -luminol	Chemi-luminescence analyzer	0.001 ppb	0.002-20 ppb	The RSD values for reproducibility of biosensor were 6.2%. The results were compared with ICP-MS	River water samples	No interference of Pb^{2+}, Zn^{2+}, Cu^{2+}, Mg^{2+} and Cd^{2+} were observed	165
Hg speciation	Liquid-liquid-liquid micro extration	18-crown-6	Electrophoresis	1.1-6.0 (Hg^{2+}), 0.8-5.5 (Me Hg), 0.2-1.8 (PhHg) μg L^{-1}	2.5-200 μg L^{-1}	The RSD values of the reproducibility tests were less than 13.0%	Hair and water samples	-	166
Hg(0)	UV-light genaration	Multimode diode lasers	Photomultiplier modules	0.12 μg m^{-3}	0-60 μg m^{-3}	The co-efficient of linear regression was obtained as R^2=0.998	-	-	167
Total Hg	-	-	Direct mercury	-	-	-	Fish samples	Vaandium also determined along with mercury	168

			analyzer						
MeHg	ISO-17025	-	Advanced mercury analyzer	0.009 ng	0.09-3.0 ng	CRMs (IAEA-436, DOLT-2, TORT-2, IAEA-452) were analyzed. The RSD values were in the range of 1.7-4.5%	Marine biota samples	-	169
Total Hg	-	-	Direct mercury analyzer	2.7 μg/kg	2-150 ng	Recoveries of Hg were in the range of 98.9-106.1%. CRM (DORM-3) was analyzed	Animal tissues	-	170

Analyte	**Method**	**Supporting media**	**Analytical Instrument**	**LOD**	**Linearity range**	**QA/QC studies**	**Analyzed Samples**	**Interference study**	**Ref**
Total Hg	-	-	Direct merucy analyzer (DMA-80)		0-50 ng	SRM (NIST-1633b) and Rice fluor-NIES-10 (Japan) were analyzed	Human hair and nails	-	171
Total Hg	Platinum trap	-	Combustion mercury analyzer (MA 3000)	0.27 ng/g	-	CRMs (INCT-PVTL-6) and STRP-IS3 were analyzed	Tobacco samples	-	172
Total Hg	Colorimetric	Lysine	Anisotropic gold nanoparticles	27 pM	0.01-1.0 nM	-	Deionized and tap waters	No interference of As^{3+}, Cr^{3+}, Cd^{2+}, Pb^{2+}, Ni^{2+}, Zn^{2+} and Ba^{2+}were observed	173
Total Hg	US EPA method 7473		Direct mercury analyzer	-	-	Method was compared with TD-AAS results	Sediments	-	174
Hg(II)	Electrochemi luminescent	Graphene coupled quantum dots	MPI-A multifuntional electrochemical analytical system	0.06 ng mL^{-1}	0.2-1000 ng mL^{-1}	The RSD values in the determination of Hg(II) real samples were in the range of 2.4-7.5%	Spiked and real water samples	No interferences of Cu^{2+}, Pb^{2+}, Ni^{2+}, and Cd^{2+} was observed	175
Hg(II)	Photoeelctro chemical	CdS quantum dots	Atomic force microscope	6.0×10^{-10} M	3.0×10^{-9} - 1.0×10^{-7} M	-	-	100 folds of Cr^{3+}, Fe^{3+}, Pb^{2+}, Cd^{2+}, Cu^{2+}, Mn^{2+}, Zn^{2+}, Al^{3+}, and Co^{3+} were not interfere	176

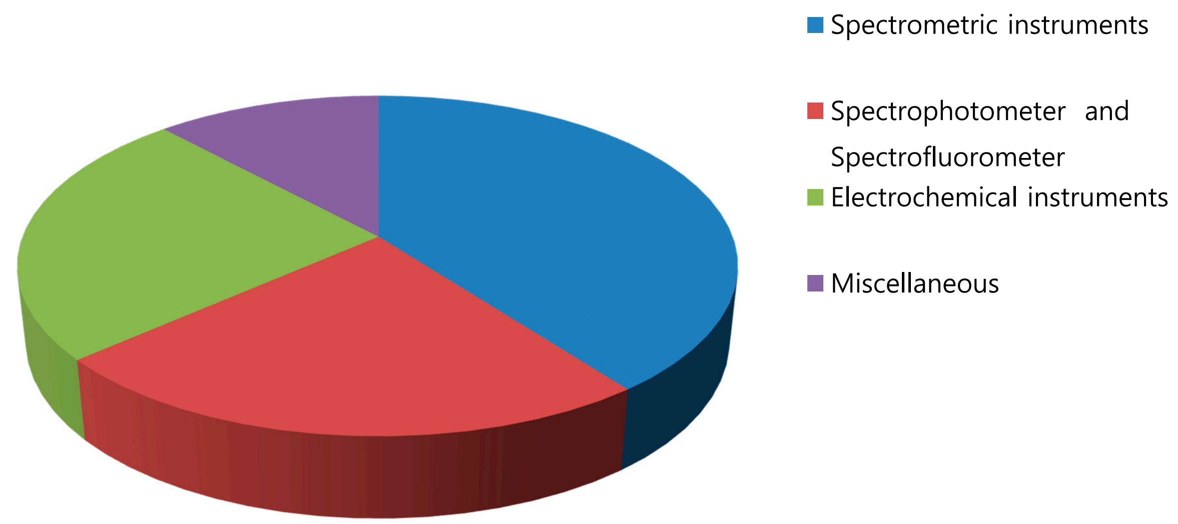

Figure 1. Determination and Speciation of mercury using various analytical instruments

The analytical figures of merit, such as limit of detection and linearity, quality control and quality assurance studies, applicability to natural samples and interference studies indicates the validity of the method. Detection limit indicates the lowest level of analyte can determine using the method conditions. A few studies [55, 71, 78, 85, 91, 146, 157, 162, 163, and 173] reported the lowest levels up to pico grams of mercury. These studies can be considered as highly sensitive due to lower detection limits. The lower detection limits were obtained mostly with ICP-MS and CV-AFS instruments than others. On the other hand, spectrophotometer and spectrofluorometer instruments can give reasonable sensitivity but they are inexpensive compare to ICP instruments. Linearity range describes the range of method stability to determine the analyte concentrations. Most of the electrochemical methods and spectrophotometry and spectrofluorometry methods were determine the linearity range of analyte concentrations.

A very important study in analytical parameters is quality assurance (QA) and quality control (QC). The validity and reliability of the data produced by the researchers are depends on this study. Quality assurance studies can performed with the measurement of accuracy of the data obtained with standard reference materials (SRMs) provided by the National Institute of Standards and Technology (NIST, USA) or certified reference materials (CRMs) provided by various reputed institutes or organizations. Quality control can be studied by measuring the precision of the data (repeatability and sensitivity) obtained in their method. The precision of the data can be obtained in many ways, such as with the analysis of number of replicates, inter-lab comparison of the data, and relative standard deviation (RSD) of blank and/or standard material analysis [177]. Regarding QA studies in the reviewed papers, a few [53, 55, 58, 70, 77, 79, 81, 90, 98, 101, 146, 164, 171] were reported the analysis of SRMs to compare the recoveries of their methods. The recoveries obtained with the measuring of SRMs gives validity to the obtained data. But the purchase of SRMs is expensive and not easy for economic laboratories. The other alternative to measure the accuracy of the data is analysis of CRMs. A large number of studies [54, 56, 58, 63, 68, 72, 78, 79, 80, 81, 82, 85, 87, 91, 93, 94, 97, 98, 115, 164, 169, 170, 172] reported the analysis of CRMs to validate their data. Regarding QC studies, most of the papers were reported the RSD values of replicate sample analysis and/or standard materials analysis. In overall, most of the researchers were aware about the quality of the data but few [60, 71, 75, 107, 110, 118, 121, 123, 124, 126, 127, 129, 130, 135, 136, 139, 140, 156, 158, 161, 168, 173, 176] were not reported any QA/QC studies which impact the reliability of their data.

The validity of analytical method can be enhanced with its applicability to the natural samples. Regarding the analysis of natural samples, most of the reviewed papers were analyzed water samples, such as river, lake, sea water, ground water, spiked and wastewaters. After water samples, the researchers were analyzed mercury in sea food samples, such as fish, shrimps, and sea weeds. A few studies reported the determination of mercury in various environmental samples, such as petroleum hydrocarbons [61], human hair [56, 62, 166, 171], phosphate fertilizers [68, 70], glycerin [69], sediments [78, 79, 85, 95, 174], cosmetics [106], living cells [114, 116, 170], and tobacco samples [172]. But few methods [51, 67, 112, 124, 137, 139, 147, 167, 176] were not reported their applicability to the natural samples. This review predicts that a very few authors [55, 71] were determined mercury content in the atmosphere. Due to the difficulty in sampling and analysis most of the authors were not concentrated on this issue.

Another important aspect of analytical parameters of the methods is interference study. Interferences of other ions in the determination of mercury are very important particularly when those methods applied to the analysis in natural samples. The natural samples usually are complex of different ions, the selectivity of the method is very important in the determination of mercury in environmental samples. In this aspect, electrochemical methods were reported higher degree of selectivity of their methods and not interfered from other ions in the determination of mercury. Interference study was not performed by a large number of authors [55, 61, 64, 69, 70, 71, 72, 74, 75, 76, 79, 80, 81, 84, 86, 87, 91, 93, 94, 95, 97, 99] who determined mercury with spectrometric instruments. But using electrochemical instruments and spectrophotometer, spectrofluorometer most of the authors were reported the interfering ion(s) studies.

Toxicity of mercury depends upon its chemical form for example; methylmercury is more toxic than inorganic mercury. The speciation studies reveal the exact toxicity of mercury in environmental samples. But very few authors [54, 61, 62, 68, 76, 81, 95, 96, 97, 98, 103, 166] were reported the speciation studies of mercury but most of the authors were determined inorganic mercury. More than 90% of electrochemical methods and spectrophotometer, and spectrofluorometer methods were determined divalent inorganic mercury and not reported speciation studies. But few were reported [60, 63, 64, 70, 73, 74, 79, 80, 82-88, 90, 99, 100, 161, 168, 170-174] the total mercury content in various samples, which doesn't predicts the accurate toxicity based on its concentrations.

4. Conclusions

The present study reviewed the research articles published in recent years (2015-2016) about the determination and speciation studies of mercury using various analytical instruments. Approximately, 126 research papers were reviewed in this regard and all the analytical parameters established in their studies are tabulated. This study concludes that most of researchers are using spectrometric instruments for the determination of mercury in different environmental samples. This study predicts the quality of the data based on reported quality assurance and quality control data by the authors. Another important prediction from this review is most of the researchers were measured inorganic mercury and/or total mercury but only few were reported speciation of mercury. Speciation studies are very important to predict the accurate toxicity of mercury in the environment because its toxicity depends on its chemical form. Due to the difficulty in sampling and analysis most of the researchers were not reported the concentrations of mercury in the atmosphere. This review suggests the speciation studies and the determination of mercury in the atmosphere has to concentrate more in future.

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