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Analysis of Heavy Metal Content in Odaiba Waters, Japan Using *Inductively Coupled Plasma Mass Spectrometry* (ICP-MS)

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ABSTRACT: This study aims to determine the content of dissolved heavy metals (Mn, Co, Ni, Cu, Zn, Cd, Pb) using *Inductively Coupled Plasma Mass Spectrometry* (ICP-MS). This method was chosen because it has high sensitivity and the ability to detect elements in ultra-low concentrations accurately. Water samples were collected on July 13, 2024, and analyzed using selective resin and UV irradiation for the separation of metals. The results showed that primary metals (Zn, Mn, Ni, Cu) exceeded the CASS-6 reference limits, with Zn having the highest concentration (88.69 ± 1.92 nM). In contrast, minor metals (Cd, Pb, Co) were below the reference values, reflecting a stable distribution of heavy metals. The increase in significant metal concentrations is thought to be related to the timing of sampling in the summer, which coincides with the rainy season, potentially increasing runoff carrying heavy metals from the city center and industry to the waters of Odaiba. The detection results of minor metals in this study indicate that ICP-MS is effective for measuring heavy metal content at very low quantities. Environmental parameters, such as pH, temperature, and salinity, are also believed to influence the solubility and mobility of metals.

Keywords: Heavy metals; ICP-MS; Odaiba waters

INTRODUCTION

Marine environmental pollution is a growing problem as anthropogenic activities increase. One of the most dangerous forms of pollution is heavy metal pollution, namely the accumulation of toxic metals such as lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As), and copper (Cu) in the marine environment, which can harm ecosystems and human health (Elgendy *et al*., 2023; Bibi, 2023). These metals usually enter the water from industrial activities, metal plating, marine transportation, and runoff from urban and agricultural areas (Rastogi and Nandal, 2020). Heavy metals are toxic and can accumulate in organism tissues through bioaccumulation and biomagnification, resulting in increased metal content at higher trophic levels, including in fish consumed by humans (Briffa *et al*., 2020; Balali-Mood *et al*., 2021). The history of heavy metal pollution in Japan has recorded environmental and health tragedies such as Minamata disease and itai-itai disease, both of which stemmed from mercury and cadmium pollution in waterways (Aoshima and Horiguchi, 2019).

In the Tokyo Bay area, particularly in the waters of Odaiba, industrial activity has been ongoing since the Meiji era and continues to increase to this day. Previous studies have shown that the content of heavy metals such as zinc (Zn) and lead (Pb) in sediments in this area has continued to increase, mainly due to waste from electrical coating factories and discharges from large rivers such as the Tama, Edogawa, and Arakawa rivers that flow into the waters of Odaiba (Sakata *et al*., 2018; Masudur & Biswas, 2012). Additionally, the geographical characteristics of Odaiba, as a *semi-enclosed* bay, cause pollutants to accumulate over a prolonged period. The most recent research data on heavy metal content in this region, particularly over the last five to ten years, can be found in Yaida *et al*. (2023), which examined the heavy metal content in the waters of Tokyo Bay and several rivers that flow into Tokyo Bay in 2017. This highlights a data gap and underscores the need to update studies to assess the current state of heavy metal pollution in the Odaiba area. The primary

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questions addressed in this study are the current level of heavy metal content in Odaiba waters and its relationship to the contributing factors in the surrounding area.

In this study, heavy metals are classified into two groups: major metals and minor metals. Major metals refer to heavy metal elements in the research results that exceed the CASS-6 reference, such as Copper (Cu), Zinc (Zn), Manganese (Mn), and Nickel (Ni). These metals are also classified based on their solubility in acidic water (Miranda *et al*., 2022; Anucha *et al*., 2022). The high content of these metal elements is thought to have critical biological functions in marine life; however, they remain potentially toxic when their content exceeds the threshold limit (Georgieva *et al*., 2023; Sutak et al., 2020). Minor metals refer to heavy metal elements in research results under the CASS-6 reference, such as cobalt (Co), cadmium (Cd), and lead (Pb). The low content of minor metal elements is thought to be due to anthropogenic activity, as they have no known biological function and can cause damage to the nervous system, kidneys, and bones, as well as lead to growth and reproductive disorders (Hossini *et al*., 2022; Wu and Zhang, 2023).

In aquatic systems, heavy metals can be present in dissolved form, bound to particles, or accumulated in sediments, depending on pH, redox conditions, and bioavailability (Cleave and Crans, 2019). Therefore, the analytical method used must be sensitive and accurate. *Inductively Coupled Plasma Mass Spectrometry* (ICP-MS) was chosen for this study due to its sensitive detection capability and resistance to interference in heavy metal analysis (Rawat *et al*., 2024; Carter *et al*., 2020). ICP-MS is the most sensitive spectrometry method compared to ICP-AAS and ICP-OES because it uses an inductively coupled plasma to ionize the sample and analyzes ions based on the mass-to-charge ratio (m/z), enabling multi-element and isotopic analysis with detection limits down to the ppt level (Balaram, 2021; Adesina *et al*., 2025). In contrast, ICP-AAS measures light absorption, and ICP-OES detects light emission from excited atoms, with detection limits generally at the ppb level (Albano *et al*., 2022). In addition to being faster and higher capacity, ICP-MS is also capable of analyzing various types of samples, including liquids, solids, and gases. At the same time, the other two methods are limited to liquid samples (Motshakeri *et al*., 2025).

MATERIALS AND METHODS

This study uses a quantitative approach with descriptive methods to describe the concentration of heavy metals in the waters of Odaiba, Tokyo Bay, and compare it with the CASS-6 standard and previous studies. Surface seawater samples were collected on July 13, 2024, at one station at coordinates (Figure 1). Samples were collected using Niskin bottles and stored in decontaminated Nalgene bottles. They were then filtered through a 0.2 µm filter and preserved with 20% hydrochloric acid (HCl).

A total of 30 mL of sample was irradiated with a 400 W UV lamp (λ = 365 nm) for 40 minutes to decompose organic ligands, then adjusted to pH 6.0–6.2 with 3.6 M ammonium acetate buffer and 25% NH_3. Metal extraction was performed using NOBIAS Chelate PA-1 resin that had been conditioned with 0.05 M ammonium acetate buffer. A 25 mL sample was placed in the resin column and eluted with 5 mL of 2 M HNO_3, then stored for analysis. NOBIAS Chelate PA1 resin is a special chelating resin widely used in heavy metal analysis using ICP-MS and ICP-OES due to its ability to concentrate metals while minimizing matrix interference. This resin contains iminodiacetic acid and ethylenediaminetriacetic acid functional groups that act as tridentate ligands, binding transition metal ions such as Ni^{2+}, Cu^{2+}, Zn^{2+}, and Co^{2+} through stable coordination bonds (Biller & Bruland, 2012; Aryee *et al*., 2020; Liu *et al*., 2021).

Heavy metal analysis was performed using Perkin Elmer NexION 2000 ICP-MS with Li, Co, Y, Ce, and Tl standard solutions (1–10 ppb, 2% HNO_3). Calibration was performed using linear regression between signal intensity and concentration, while metal content calculation used a modification of the Robbika *et al*. (2022) formula:

$$y = mx + b$$

Where: y = Number of ions counted per second/Signal intensity (cps); m = Calibration curve slope (slope); x = Concentration; b = *Blank/Background Signal*

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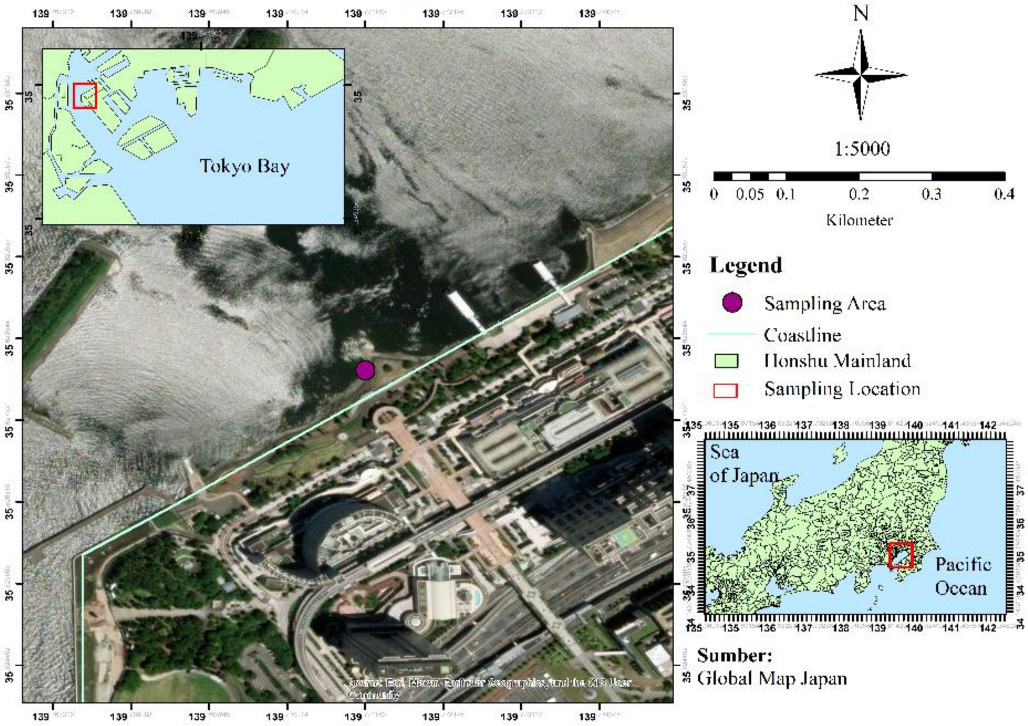

Figure1. Sample Collection Location Map

The analysis data were processed descriptively (mean and standard deviation) and compared with the reference standard to determine the level of pollution in the study waters.

RESULTS AND DISCUSSION

The classification of major heavy metal content consists of Mn, Ni, Cu, and Zn, referring to the metal content of the research samples that exceeded the CASS-6 standard reference, which is a Certified Reference Material (CRM) of coastal seawater prepared by the National Research Council of Canada (NRC) with information on trace element content and other constituents (Brophy *et al.*, 2016). The analysis results show that the major heavy metal content exceeds the CASS-6 minimum reference limit, with a relatively significant average difference. Zinc (Zn) was recorded as the metal with the highest content (88.69 ± 1.92 nM), but it had the most critical measurement variation. Manganese (Mn) ranked second (53.82 ± 1.18 nM) with more moderate variation. Nickel (Ni) showed better data consistency (25.28 ± 0.71 nM), followed by copper (Cu), which had the lowest but most stable content (9.10 ± 0.15 nM). The relatively small standard deviation values, especially for Cu and Ni, indicate a fairly consistent supply of dissolved metals at the research site (Table 1).

Minor heavy metals detected include cobalt (Co), cadmium (Cd), and lead (Pb), with all contents below the CASS-6 minimum reference limit. The average difference between the measurement values and the reference standard is relatively small. Cobalt (Co) showed the highest content of 0.25 ± 0.15 nM, but with relatively high data variation, as indicated by the significant standard deviation. Cadmium (Cd) and lead (Pb) had much lower concentrations, 0.05 ± 0.0012 nM and 0.04 ± 0.0012 nM, respectively, and showed high consistency between repetitions. The small standard deviation values, especially for Cd and Pb, indicate a stable distribution of metals and a relatively uniform supply at the study site.

The ICP-MS analysis results show that the heavy metals detected in Odaiba Waters include the primary metals Zn, Mn, Ni, and Cu, as well as the minor metals Co, Cd, and Pb. Based on

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comparison with the CASS-6 standard (Brophy *et al*., 2016), Zn had the highest content at 88.69 ± 1.92 nM, followed by Mn (53.82 ± 1.18 nM), Ni (25.28 ± 0.71 nM), and Cu (9.10 ± 0.15 nM). The low variation in Cu and Ni data indicates a stable distribution, while the high Zn value is thought to originate from anthropogenic activity around the Tokyo Bay industrial area. Cu showed a value (9.10 nM) similar to that of CASS-6 (8.41 nM), indicating relatively normal water conditions. In contrast, Zn increased sharply to 88.69 nM compared to the reference value of 19.2 nM, indicating potential pollution due to industrial or anthropogenic activities around the coastal area. Although the increase in Cu was still within the measurement error limit, the accumulation of this metal still reflected potential environmental pressure.

.Meanwhile, Mn and Ni also increased from their respective reference values of 40.4 nM and 6.97 nM to 53.82 nM and 25.28 nM, indicating the possibility of heavy metal input from human activities or terrestrial runoff. Natural redox processes and human activities likely contributed to the increase in Mn content. *Runoff* processes accelerate the dissolution of metals such as Zn^{2+}, Cu^{2+}, and Ni^{2+} through acidic rainwater, thereby increasing the mobility of metals in aquatic systems (Gao *et al*., 2023). The interaction between fine particles in sediment suspension and sea ions also causes metal desorption into seawater (Minkina *et al*., 2025). The Keihin industrial area, which encompasses Tokyo, Yokohama, and Chiba, is believed to be a significant contributor to heavy metal pollution through runoff and atmospheric deposition, as indicated by historical evidence from sediment isotope analysis (Sakata *et al*., 2019).

Minor metals such as Cd and Pb showed lower or equivalent concentrations to the reference values, at 0.05 ± 0.0012 nM and 0.04 ± 0.0012 nM, respectively. This indicates that contamination from these two metals has not increased significantly in this region. Co has a relatively large standard deviation (0.25 ± 0.15 nM), which may indicate instability in the content at a single observation point. Meanwhile, the Cd, Pb, and Co content in the samples was well below the CASS-6 values, which are 0.05 nM, 0.04 nM, and 0.25 nM, respectively, compared to the reference values of 0.20 nM, 0.05 nM, and 1.14 nM. These results indicate that the water is relatively clean of these three elements, although the higher variability of Co indicates spatial differences in the sampling locations. The stable distribution of Pb since the 1980s is associated with inputs from the Edo River and industrial activities in surrounding large cities, including battery recycling and non-ferrous metal processing (Hosono *et al*., 2016; Kojima & Jain, 2008). Increased Co content in hypoxic areas is thought to be due to remobilization from sediments and inputs from metal processing and electronics manufacturing industries (Kurian *et al*., 2020; Nitzsche *et al*., 2022).

The results of simultaneous measurements of minor and major metal content also demonstrate the high sensitivity of ICP-MS, as indicated by low standard deviation and the ability to detect many elements simultaneously in a single measurement cycle with a very low detection limit (<1 ng/L) (Michalke, 2022).

The presence of heavy metals in the waters of Odaiba, Tokyo Bay, can be explained both spatially and temporally. Spatially, ocean dynamics, water column stratification, and tidal and seasonal conditions are thought to influence metal distribution. Ocean currents and stratification

Table 1. Heavy Metal Concentrations in Odaiba Waters, Tokyo Bay in 2024 (nM)

Major Metals			Minor Metals		
Heavy Metal Elements	Research Results (nM)	CASS-6 Standard Reference (nM)	Heavy Metal Elements	Research Results (nM)	CASS-6 Standard Reference (nM)
Cu	9.10 ± 0.15	8.41 ± 0.51	Cd	0.05 ± 0.0012	0.20 ± 0.02
Zn	88.69 ± 1.92	19.2 ± 2.73	Pb	0.04 ± 0.0012	0.05 ± 0.02
Mn	53.82 ± 1.18	40.4 ± 2.18	Co	0.25 ± 0.15	1.14 ± 0.09
Ni	25.28 ± 0.71	6.97 ± 0.67			

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processes (Figure 2) promote the dispersion of soluble metals, such as Zn and Mn, while metals that are easily adsorbed onto particles, including Cu, Pb, and Cd, tend to settle (Schindler *et al*., 2021; Ahmed *et al*., 2022). Stratification limits vertical mixing, causing heavy metals originating from surface inputs to remain trapped in the upper layers (Wang *et al*., 2022). Sampling was conducted during the neap tide phase (July 13, 2024), when low tidal conditions reduced the effects of sediment resuspension, thereby potentially providing a more representative picture of heavy metal distribution in relation to local surface conditions (Barbarelli & Nastasi, 2021; Geng *et al*., 2024).

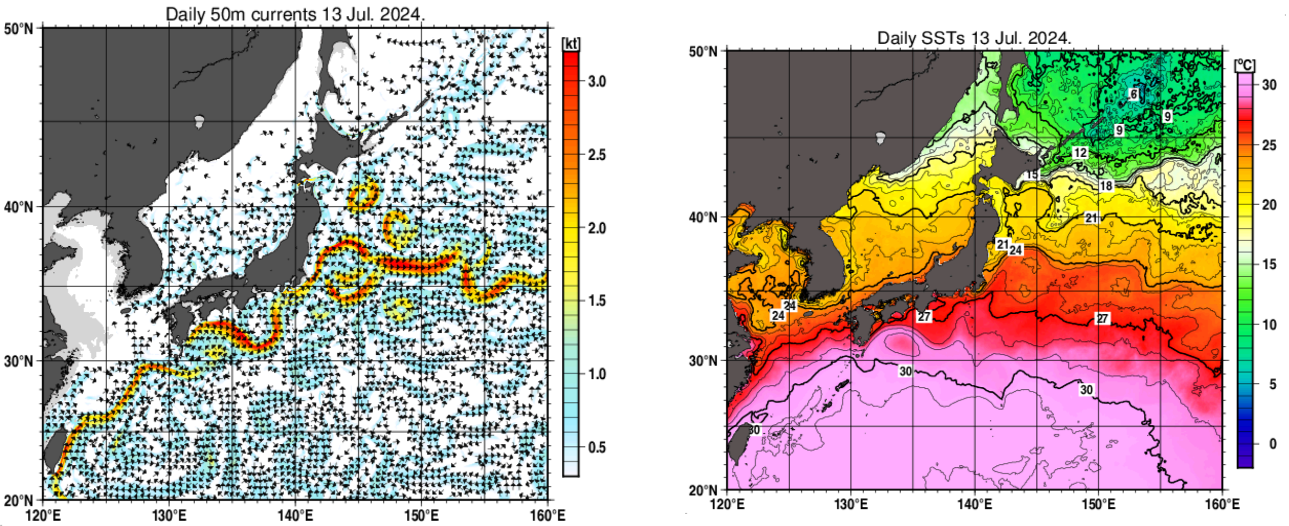

Figure 2. Distribution of Daily Currents and Distribution of Daily Surface Temperature in the Sea of Japan on July 13, 2024 *(Japan Meteorological Agency)*

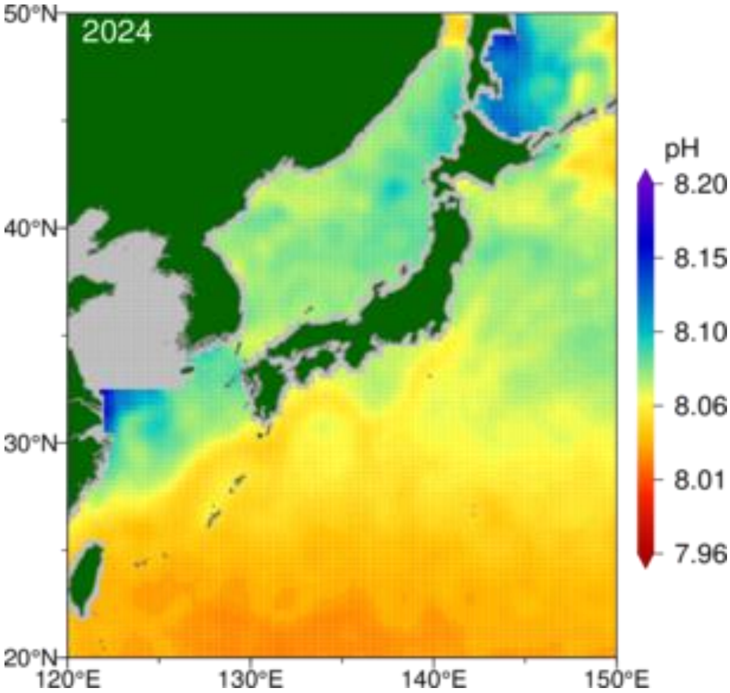

Figure 3. Distribution of pH Content in the Sea of Japan in 2024 (*Japan Meteorological Agency*)

Temporally, the summer season in Japan, characterized by increased stratification, surface temperature, and land runoff due to high rainfall, is thought to enhance the external input of heavy metals such as Zn and Mn into the waters (Garcia-Soto *et al*., 2021; Ohba *et al*., 2024). Mn and Ni concentrations increase during the rainy season and typhoons, presumably due to the influence of particles from river runoff and anthropogenic activities (Islam and Mostafa, 2023; Billah *et al*., 2019), while higher temperatures have the potential to increase the solubility of metals such as Zn^{2+} and Mn^{2+} (Aslam *et al*., 2025). Salinity variations also contribute to metal mobility through changes in speciation and complexation with dissolved organic matter (Mosley & Liss, 2020; Nakano *et al*., 2023).

Environmental parameters such as pH, temperature, salinity, and the physical-chemical conditions of the water are thought to influence the distribution of heavy metals in marine waters. This correlation is consistent with the distribution of ocean pH in 2024 (Figure 3), where eastern Japan exhibits relatively acidic pH levels. Under acidic conditions, heavy metals such as Zn^{2+}, Mn^{2+}, and Ni^{2+} tend to remain dissolved, thereby increasing their content in the water column (Najamuddin *et al*., 2023; Miranda et al., 2022; Anucha *et al*., 2022). Conversely, metals such as Pb, Cd, and Co are thought to form insoluble compounds, such as hydroxides or carbonates, more easily at neutral to alkaline pH, thus tending to precipitate (Esfandiar *et al*., 2022). In addition, pH also has the potential to influence the processes of adsorption, ion exchange, and the formation of metal complexes with dissolved organic matter (Dai *et al*., 2022).

Temperatures reaching 27°C (Figure 2) are believed to accelerate dissolution, desorption, and metal oxidation-reduction reactions, thereby facilitating the release of heavy metals from sediments into the water column. High temperatures increase the solubility potential of metal ions such as Zn^{2+} and Mn^{2+} and accelerate chemical reactions between particles (Ma *et al*., 2024; Aslam *et al*., 2025; Li *et al*., 2022). High surface temperatures have the potential to create thermal stratification, which limits vertical mixing and thereby influences the distribution of metals on the surface more significantly through horizontal currents (Liu *et al*., 2019; Garcia-Soto *et al*., 2021).

Industrial activities and other anthropogenic sources significantly influence the distribution of heavy metals in Tokyo Bay. Industrial waste, urban runoff, and the use of copper (Cu)-based antifouling paints are considered the primary contributors to the increased Cu content in port and estuary areas (Sproson *et al*., 2020; Carić *et al*., 2021). Electroplating and metallurgical activities also have the potential to spread Zn widely through the release of industrial waste and its affinity for organic particles, which are then resuspended due to tides or dredging (Douglas *et al*., 2025).

CONCLUSION

Odaiba waters are contaminated with primary heavy metals, with zinc (Zn) as the highest pollutant (88.69 nM), followed by manganese (Mn: 53.82 nM), nickel (Ni: 25.28 nM), and copper (Cu: 9.10 nM). In contrast, minor metals (Cd, Pb, Co) were recorded at low concentrations (<0.25 nM), below the reference value. Accurate and precise ICP-MS analysis results confirmed an increase in heavy metal content, particularly Zn, Mn, and Ni, which was dominated by anthropogenic inputs from industry and urban runoff. The distribution of these metals is also complexly influenced by environmental factors, including water stratification, temperature, and rainfall. For future research, it is recommended to expand temporal and spatial monitoring, track specific sources of pollutants (especially Zn and Ni), and assess the ecological impacts of metal accumulation.

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