ORIGINAL ARTICLE

Annals of Agricultural and Environmental Medicine 2023, Vol 30, No 3, 455–461
www.aaem.pl

A simplified method for determining potential heavy metal leached from sediments of stormwater and combined sewer systems – importance for public health

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Duda-Saternus S, Kujawska J, Wojtaś E, Kozłowska A, Jamka K, Szeląg B, Babko R, Łagód G. A simplified method for determining potential heavy metal leached from sediments of stormwater and combined sewer systems – importance for public health. Ann Agric Environ Med. 2023; 30(2): 455–461. doi: 10.26444/aaem/170099

Abstract
Introduction and Objective. The identification and understanding of interactions between contaminants present in sediments from stormwater and combined sewer systems is a prerequisite for their proper management, and provides a basis for developing effective strategies to minimize their negative impact on humans and the environment. The study presents the method described in PN-EN 12457-2:2006 as a possible technique for studying the mobility of heavy metals in sediments from stormwater and combined sewer systems.
Materials and method. The presented PN-EN 12457-2:2006 method is a relatively simple technique for preparing extracts for the determination of heavy metals in sediments from stormwater and combined sewer systems, consisting of one-step leaching, which is quick to perform. In addition, it allows determination of the characteristics of the samples to be analyzed, and indicates procedures and tests for evaluating hazardous substances released from solid waste.
Results. The results of the concentrations of leached heavy metals: chromium, copper, nickel, lead and zinc, obtained in the study, corresponded to the concentrations of the exchange fraction of sludge when using the recommended method with sequential extraction (Student's t-test, $p=0.263$). In the literature review conducted, no papers were found on the application of the leaching method to prepare extracts for the determination of heavy metals in sediments from stormwater and combined sewer systems.
Conclusions. The PN-EN 12457-2:2006 method is capable of providing important data on the potential risks to humans and the environment from the presence of contaminants in sewage sludge.

Key words
heavy metals, sediments, sediments of stormwater and combined system

INTRODUCTION

Sediments from stormwater and combined sewer systems comprise solids deposited in the manhole pipes and other facilities. If the sewerage system is operating properly, the sediments move with the wastewater and can be separated by filtration, decantation or centrifugation. Sediments consist mainly of solid organic and inorganic substances in the form of emulsions and suspensions of varying hydration [1]. The operation of the sewer system, which depends on the variability of rainfall conditions, the characteristics of the flowing wastewater, as well as the parameters of pipes, manholes and other facilities located in the system, contributes to the formation of sediments. During the flow of wastewater in a storm, in the combined sewer and sanitary sewer systems there is an increase in the biofilm on the walls of the pipes, which is a reservoir of numerous microorganisms. The processes of transport, transformation and biodegradation of pollutants lead to the precipitation of suspended solids and the formation of sediments [2–4].

Although sediments constitute an integral part of sewage systems, their excessive presence can lead to serious problems. These can include reduced capacity of the pipes, leading to flooding of passageways or lower rooms in buildings, as well as periodic deterioration of wastewater quality which, in turn, hinders its treatment. Moreover, sediments can carry

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Received: 30.06.2023; accepted: 27.07.2023; first published: 11.08.2023

pollutants that can enter the environment, for example, through leaching from storm sewers or combined sewers [5–7].

Sediments in storm sewers are an integral part of the system; in order to maintain its proper functioning, the sediment must be regularly removed and properly managed or disposed of [8]. Monitoring the quality of stormwater sediments is necessary for a number of reasons, both in an environmental and health context. Knowledge of the composition of these sediments, including the content of mineral compounds, organic compounds and pollutants of anthropogenic origin, is essential for their proper management in the catchment. It is also important to assess the extent to which sediments from storm sewers are contaminated and whether they pose a threat to the environment and human health.

The chemical composition of stormwater drainage sediments is diverse and can include a number of different contaminants [9, 10]. These pollutants depend on a number of factors: catchment development and imperviousness of its surface, structure, type of road network, traffic volume, terrain and weather conditions [11]. Sediments can contain components that have harmful effects on water quality and ecosystems. There are studies devoted to new, significant threats to human health and ecosystems found in sediments. These include chemicals – such as toxic trace elements – Se, Ag, Ti; nanoparticles; aromatic hydrocarbons; polychlorinated biphenyls; chemical surfactants, polycyclic compounds, siloxanes, pesticides, phenols, personal care products and pharmaceuticals, as well as hormones and numerous biological pathogens (viruses, parasites and their eggs, pathogenic bacteria and others) [Tab. 2, 3, 12–17].

Heavy metals are some of the most important pollutants found in the sediments from stormwater and combined sewer systems. Previous studies show that the amount of metals in sludge varies. Table 1 shows the concentration ranges of heavy metals found in sediments from stormwater and combined sewer systems. The concentration ranges of metals in sediments vary widely, for example, from 9 – 456 mg/kg in the case of lead, indicating the need for regular monitoring studies. Distinguishing between these metal concentrations is crucial for environmental quality and public health management. Significantly variable concentrations mean that one-off measurements may not be sufficient to fully understand potential health risks. As cities and towns grow and develop, regular monitoring and management of heavy metals becomes even more important. Proper monitoring and better management strategies effectively minimizing exposure to these heavy metals can ensure the reduction of potential risks to public health.

The content of heavy metals in sediments is a particularly serious environmental problem due to their lack of biodegradability, high toxicity and tendency to bio-accumulate [19]. In addition, heavy metals such as cadmium, chromium and nickel can be dangerous even at very low environmental concentrations [20]. These metals, which enter the biological system through different trophic levels, can bio-magnify and pose significant risks to human health [21]. Furthermore, the need for effective monitoring and surveillance of heavy metal concentrations in sediments cannot be over-emphasized [22]. This issue has escalated to become a matter of concern at multiple scales – local, regional and national – due to the implications associated with the concentration, effects, distribution and origin of these heavy metals [23]. The bio-accumulation of heavy metals critically affects the life processes of a wide range of organisms [24]. Furthermore, their effects on human health are of particular concern as they can lead to a range of toxicities targeting organs, such as the kidneys (nephrotoxicity), nervous system (neurotoxicity), liver (hepatotoxicity), skin (dermatotoxicity) and cardiovascular system [25].

Table 1. Concentration of heavy metals from more than 100 reservoirs located in a stormwater drainage system [18]

Metals	Range [ppm]
Fe	360–46,100
Zn	12–4,940
Mn	25–1,850
Cu	6–1,090
Pb	9–456
Ni	4–159
Co	2–31

Scientific data indicate that cadmium exposure causes lung cancer. Chromium, especially Cr(VI), has harmful effects on the central nervous system; moreover, it can cause kidney and liver damage. In turn, long-term exposure to nickel can lead to cancer, damage to the nervous system and reduced cell growth [26]. A significant source of heavy metals is the run-off of rainwater, which mainly originates from the sources associated with road traffic and industry, primarily automotive [27–30]. Identification and understanding of the interactions between the contaminants present in sediments is a step towards their proper management, and provides a basis for developing more effective strategies to minimize their negative impact on humans and the environment.

To date in Poland, no legal regulations have been issued on the quality of sediments from stormwater and combined sewer systems. In order to assess them, it is recommended to test the physico-chemical properties of sediments using accredited methods and methods specified in Polish standards. In practice, these mainly involve the methods related to sludge, for which the Sewage Sludge Directive (86/278/EEC) indicates only permissible limits for Zn, Cu, Ni, Pb, Cd, Cr and Hg and pathogens. The directive also regulates the recovery of sludge on land under certain sanitary and environmentally friendly conditions [8, 19, 31]. This adds to the challenge of managing sediments from storm and combined sewer systems, and emphasizes the need for further research and standardization in this area. This is due to the fact, among others, that the sewage sludge generated in treatment plants is characterized by high homogeneity and the absence of problematic solids, which have been removed within the mechanical part of the treatment plant by straining (screens, grids, etc.) and falling into the gravity field (sand traps).

On the other hand, stormwater sediments are characterized by a very wide range of consistency, density and content of very different sizes and materials of solid parts, e.g. rubble, gravel, sand, leaves and tree needles, bottle caps, cigarette butts, and many other solid wastes, on which various fractions of mineral and organic suspensions can adhere, being carriers of the afore-mentioned metals, petroleum substances, parasite eggs, bacteria, viruses, etc. [3, 5].

In view of the above, there is a need to continue research on the chemical composition of sediments from stormwater and combined systems. It is critical that studies focus not only on identifying the various contaminants in these sediments, but also on understanding the processes affecting their transport and distribution.

The authors propose using following method:

PN-EN 12457–2:2006: Characterization of Waste – Leaching – Compliance Test for Leaching of Granular Waste Materials and Sludges; Part 2: One Stage Batch Test at a Liquid to Solid Ratio of 10 L/Kg for Materials with Particle Size Below 4 Mm (without or with Size Reduction).

This is an effective and economical method for ecotoxicological evaluation in sediments analysis [32, 33] which provides extracts that can identify key contaminants and pollution indicators found in sediments, such as heavy metals, polycyclic aromatic hydrocarbons, biological oxygen demand, as well as forms of nitrogen and phosphorus.

Expanding research in the area of sediments from stormwater and combined sewer systems will allow the development of innovative methods for its disposal, and thus reduce its negative impact on various elements of the environment and, consequently, human health. The method has the potential to provide important data on the potential environmental risks of the presence of contaminants in these sediments. Knowing and understanding the interactions between the contaminants present in the sediments is a step forward toward proper management of these sediments, and provides a basis for developing more effective strategies to minimize the negative impact of sediments from stormwater and combined sewer systems on humans and the environment.

The aim of the current study is to develop and validate a simplified method for determining the potential load of heavy metals leached from sediments of stormwater and combined sewer systems, as well as to assess its significance for public health. To achieve this aim, the following tasks were set:
1) to undertake a comprehensive analysis of the scientific literature on current knowledge of heavy metals in stormwater and combined sewer system sediments;
2) to develop a simplified method for determining heavy metal loads in sediments;
3) to analyze the results and compare them with available literature data to assess the effectiveness of the new approach.

MATERIALS AND METHOD

Speciation analysis is commonly used to evaluate the mobility of heavy metals in sewage sludge. This process involves the sequential use of different solvents to extract specific forms of metals. Each extraction step (i.e., each use of a different solvent) is designed to extract a specific 'fraction' of a chemical compound, such as an exchangeable form, a form bound to organic matter, a form bound to metal oxides, or a residual form [34]. For example, in the case of heavy metals in sediments, sequential extraction can be used to determine which fractions of metals are most mobile and potentially bio-available, which is crucial for environmental risk assessment.

Sequential extraction, proposed by Tessier, Campbell and Bisson in 1979, is one of the most widely used methods for fractionating heavy metals in environmental samples, such as soils or sediments. It consists of five sequential extraction steps, each designed to isolate a specific 'fraction' of metals:
1) *exchangeable fraction:* extraction with magnesium sulfate at low pH aims to isolate the metals that are easily exchangeable and can be quickly released into the environment;
2) *organic carbon and sulfide bound fraction:* extraction with a solution of acetic acid and sodium sulfate aims to isolate metals bound to organic matter and sulfides;
3) *fraction bound to metal oxides:* extraction with a solution of hydrogen peroxide and ammonia aims to isolate metals bound to metal oxides, such as iron oxide and manganese oxide;
4) *mineral-bound fraction:* extraction with nitric acid aims to isolate metals bound to minerals;
5) *residual fraction:* extraction with a mixture of nitric acid and hydrofluoric acid, aims to isolate the 'residual fraction', which contains metals bound to highly resistant minerals [35].

The study presents and recommends the PN-EN 12457-2:2006 method as an alternative to a commonly used procedure, the Tessier sequential extraction method. The PN-EN 12457-2:2006 method specifies the procedures for conducting tests used to evaluate the release of hazardous substances from solid waste.

Tests were conducted on sediments from stormwater and combined sewer systems, which was extracted with deionized water at a ratio of 1/10 after mixing on a shaker for 24 hours. Heavy metals were determined in the extracts by inductively coupled plasma emission spectrometry using Agilent's JY 238 Ultrace ICP Spectrometer with radical plasma (Horiba Jobin Yvon). Determination of metals by ICP-MS was performed in accordance with PN-EN ISO 17294-2:2006 – Water quality – Application of inductively coupled plasma mass spectrometry (ICP-MS). Part 2 – Determination of selected elements including uranium isotopes [36]. Detection limits were set individually for each series of measurements for the metals under study. The limits were determined at different emission bands and specific wavelengths (Tab. 2).

Figure 3 shows a comparison of the results of own tests, the concentrations of heavy metals leached from stormwater sediments, obtained according to the procedures proposed in the PN-EN 12457–2:2006 method, with the results of heavy metal concentrations obtained using sequential extraction, as observed in other studies.

The results of the concentrations of the heavy metals tested: chromium, copper, nickel, lead and zinc according to the PN-EN 12457–2:2006 method corresponded to the concentrations of the exchange fraction from the Tessier

Table 2. Wavelengths (nm) and limits of detection (ppb) used in metal determination using ICP [32]

Metal	Wavelengths [nm]	LOD [ppb]	Metal	Wavelengths [nm]	LOD [ppb]
Zn	213.856	6.77	Fe	259.94	0.92
Cd	228.802	0.24	Mn	257.610	4.02
Co	228.616	0.62	Mo	202.030	0.92
Cr	267.716	0.39	Ni	221.647	0.40
Cu	324.754	2.02	Pb	220.353	0.38

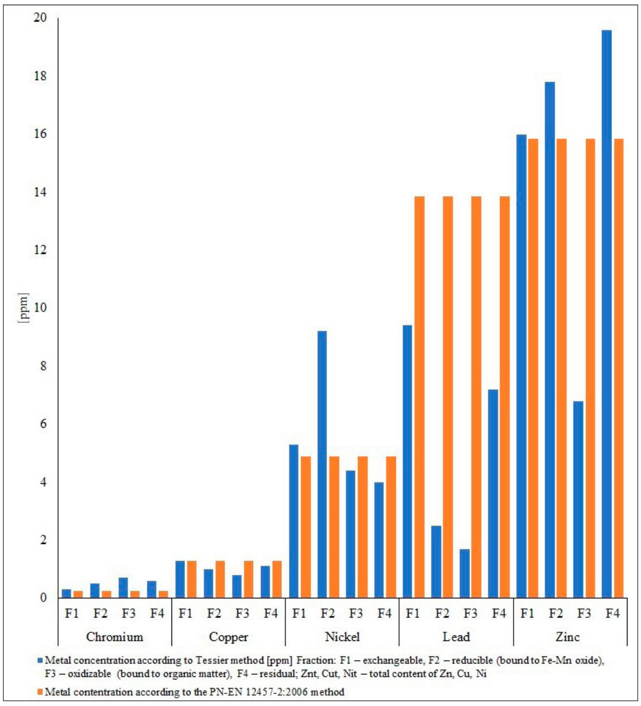

Figure 1. Comparison of the results of own tests of metal content in stormwater sediments obtained according to the PN-EN 12457-2:2006 method and the method using sequential extraction [33]

sequential extraction method, obtained in other studies [37]. The results show no statistically significant differences between the presented leaching test results (Student's t-test, p-value =0.263).

RESULTS AND DISCUSSION

Although sequential extraction is a commonly used technique to assess the forms and availability of contaminants in environmental samples, the method has several limitations. Sequential extraction is based on the assumption that different solvents are able to selectively extract specific fractions of contaminants. However, in practice, some solvents can extract more than one fraction, which can lead to inaccuracies in determining fractions. Sequential extraction is a multi-step process that is time-consuming. Due to the lengthy and complicated process, there is a risk of sample contamination during the various steps of sequential extraction. The sample is destroyed during extraction. This makes it impossible to repeat the analysis on the same sample [38].

In addition, sequential extraction relies on chemical reagents other than those found in the environment, consequently, some researchers have questioned the use of these chemical reagents to quantify the bio-available fraction [39, 40]. There are also limited opinions in the available literature on the relationship between the sorption behaviour of heavy metals, and the quality of the run-off wastewater (e.g., dissolved organic carbon content and pH).

It seems that dissolved organic matter and pH are the most important solution parameters affecting the mobility of metals from sediments [41]. Dabrowska (2015) [37] conducted a comparative study of the fractionation of heavy metals (Zn, Cu, Ni, Pb, Cd, Cr) from bottom sediments, using two types of sequential extraction applied – Tessier and a three-step extraction known as the BCR procedure (the solvents used were acetic acid, ammonium hydroxide, oxidized water, as well as a mixture of concentrated nitric and hydrochloric acids). In the bottom sediments, the cadmium content determined in the exchange-carbonate, organic-sulfide and residue fractions (practically insoluble compounds) after extraction by the Tessier method, did not coincide with the values obtained after extraction by the BCR method. This was also true for the zinc and lead content of the iron and manganese oxide fractions. The reasons for the discrepancies in the results obtained may have been both the extraction reagents used and the extraction conditions (different reagents, temperature and time) [37]. This confirms the importance of choosing the right extraction method depending on the purpose of the speciation analysis being conducted, and the chemical forms of heavy metals being analyzed.

Bojakowska et al. (2012) [42] conducted an assessment of sediments at selected sites in Warsaw. Among the heavy metals tested, the highest concentrations were recorded for zinc – in the range of 44 – 458 mgZn/kg. Copper was present in the range of 5 – 72 mgCu/kg, which slightly exceeded Class II according to the German LAWA classification used to assess sediments quality. Lead concentrations ranged from 3 – 62 mgPb/kg, classifying the sediments as uncontaminated to moderately contaminated. Elevated levels of cadmium were noted in almost all measurement samples – above 1 mgCd/kg [42]. Heal et al. (2016) [39] characterized sediments at selected sites in Dunfermline, Scotland. The highest concentrations of heavy metals were recorded for nickel (63.6 – 89.3 mg/kg), which, according to the LAWA classification, classifies the sediments in purity class II. Elevated concentrations were also observed for chromium (118 mg/kg) [43].

These studies shed light on the diversity of heavy metal concentrations in sediments, which emphasizes the need for further, detailed research to assess their mobility, toxicity and impact on the environment and public health. The authors therefore propose using the EN 12457–2:2006 method which involves one-step leaching and has advantages that make it a viable alternative to more complicated and time-consuming procedures, such as sequential extraction. The method is recommended because it is relatively simple and quick to perform. Samples of the sediments are mixed with deionized water in a specified ratio and shaken under specified conditions; then, they are analyzed for the presence of various substances. This offers the possibility of collecting reliable data for calibrating mechanistic models of stormwater and combined sewer catchments. The method also provides a fairly good representation of turbulent flow conditions, which can be more representative of the conditions that prevail in real sewer systems during leaching and transport of non-homogeneous sediments. Thus, the results obtained using it can better reflect the potential impact of sediment on the environment – surface waters that most often receive rainwater and general runoff discharged through storm overflows [44]. This method also has the potential to be applied to ecotoxicological studies, which would allow a more accurate understanding of the effects of various contaminants in sediments [45].

However, like all methods, EN 12457–2:2006 has its limitations and does not provide a complete picture of all the forms in which heavy metals may be present in sediments. Hence, it can be used in conjunction with other analytical techniques, depending on the specific research objective.

Calibration of the above method with more accurate multi-step methods would allow faster, more environmentally friendly (no need for chemical reagents) and broader screening. This leaching test is essential, as it helps to determine the amount of heavy metals that can be leached from sediments under certain conditions. However, to gain a more complete understanding of the heavy metals present in sediments, their forms and the risk they pose, it may be beneficial to use additional analytical techniques. Such techniques include inductively coupled plasma mass spectrometry (ICP-MS) or atomic absorption spectroscopy (AAS), X-ray diffraction (XRD), scanning electron microscopy (SEM) or transmission electron microscopy (TEM) coupled with energy dispersive X-ray analysis (EDX). Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Atomic Absorption Spectroscopy (AAS) can be used to determine the total concentration of heavy metals in sediments, giving a complete picture of the heavy metal load.

In turn, EN 12457–2:2006 provides information on the leachable fraction, ICP-MS or AAS can provide information on the total amount of metals, including those that may be tightly bound to the sediment matrix and not easily leached. XRD can be used to determine the mineralogical composition of the sediments. Understanding the mineralogy can help predict the behavior of heavy metals, as certain minerals can adsorb or sequester heavy metals. Scanning electron microscopy (SEM) or transmission electron microscopy (TEM) coupled with energy dispersive X-ray analysis (EDX) can provide information on the morphology of the sediments and the spatial distribution of heavy metals at the micro- and nanoscale. This can help to identify potential 'hot spots' of heavy metal concentrations in stormwater and combined systems.

The final interpretation of the results requires a full understanding of the limitations of each method and the potential errors that can result from the extraction process. All of this emphasizes the complexity of the problem of analyzing heavy metals in sediments from storm and combined sewer systems, as well as the need for conducting further research in this area. In the literature review performed, no studies were found on the application of the leaching method to the preparation of extracts for the determination of heavy metals in sediments. It is worth considering a broader introduction of the method into sediments research. It is necessary to determine the water-sediment relationship. Water extracts from stormwater sediments beside of analysis of heavy metal amount can additionally be studied using arrays and immersion probes that are part of electronic sensing, in this case an e-tongue. The results of the analyses may constitute material for studies related to risk assessment of the aquatic environment and public health risks.

Such studies continue to be relevant, since the processes associated with the spread of heavy metals and their impact on the environment and living organisms are very complex and dependent on many factors [46, 47]. When heavy metals enter the aquatic environment, they are absorbed by microorganisms. Later, they enter the organisms of aquatic invertebrates and fish through food webs, where they accumulate in the vital organs, affecting the growth and reproduction of these organisms. They can also threaten human health through the food chain, causing a variety of diseases [48]. Thus, heavy metal pollution carries potential risks for the environment and human health.

Risk assessment is becoming an increasingly important issue in the face of ongoing climate change and apparent global trends in rainwater management and use in various industries. Climate change is leading to increasingly unpredictable weather events, including changes in rainfall patterns. Increased rainfall may result in greater surface run-off into stormwater systems, leading to increased transport of sediment and associated pollutants. These changes could potentially lead to higher pollutant loads entering water bodies, with implications for both the environment and public health.

The proposed simplified method for determining potential heavy metal loads leached from sediments can make a significant contribution to risk assessment efforts. By providing a simplified method for measuring heavy metals in sediments, the presented work can contribute to a better understanding of contaminant dynamics under different climatic conditions and management strategies. This knowledge could be valuable in predicting and managing the risks associated with heavy metal contamination under changing precipitation patterns due to climate change.

The above-mentioned information is important for understanding the mechanisms that occur in sewer networks, in the context of building and calibrating computational models. Reliable research results, and thus reduction of their uncertainty in the context of conducting multi-year analyses, provide the opportunity to expand the scope of environmental monitoring, which is important for the development of methods for assessing the impact of anthropogenic factors on the quality of surface water and aquatic ecosystems [49–51]. Hence, it is also important to determine the role of weather conditions and pollutants contained in particulate matter that have a major impact on the health and mortality of the population [52], as well as the processes of deposition and leaching from urban catchments [53].

CONCLUSIONS

In conclusion, it can be stated that the sediments from stormwater and combined sewer systems are significant media which can carry a variety of pollutants, including heavy metals. They require constant monitoring, and the proper analysis of these sediments is crucial for environmental quality management as well as public health protection. Therefore, this study focused on the development and validation of a simplified method for the determination of heavy metal loads in these sediments.

The presented simplified method, based on PN-EN 12457–2:2006, was found to be effective in identifying key pollutants and pollution indicators present in sediments, such as heavy metals and most probably polycyclic aromatic hydrocarbons, biological oxygen demand, as well as forms of nitrogen and phosphorus. This suggests that the proposed method can be used as an effective and economical tool for assessing the impact of sediments on the environment and human health.

This conclusion has potentially significant implications for environmental quality management and public health practice. Continuous monitoring and management of heavy metals is becoming increasingly important as cities and towns develop. Through proper monitoring, better management strategies can be ensured that effectively minimize exposure to these toxins and reduce potential health risks.

These findings also highlight the need for continued research into the chemical composition of stormwater and combined sewer system sediments. The focus should not only be on identifying different contaminants in these sediments, but also on understanding the processes that affect their transport and distribution.

Future research will look at other types of contaminants, such as pesticides, pharmaceuticals and microplastics, as well as hazardous pathogens in sediments water extracts. Expanding the range of pollutants tested will help in a better understanding of the overall impact of sediments form stormwater and combined systems on the environment and public health.

Acknowledgements

This study was partially supported by the Polish Ministry of Education and Science within Grant Nos. FD-20/IS-6/999 and FD-20/IS-6/019.

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