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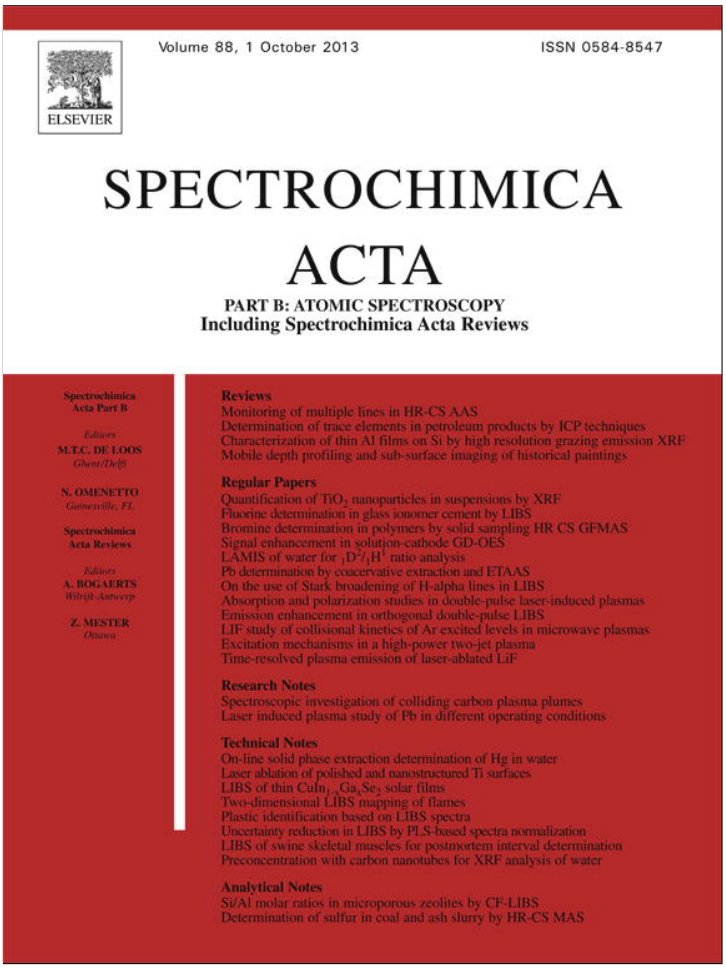

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Technical Note

Ultraviolet vapor generation atomic fluorescence spectrometric determination of mercury in natural water with enrichment by on-line solid phase extraction

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ABSTRACT

A novel method, which coupled an on-line solid phase extraction (SPE) enrichment with ultraviolet vapor generation (UVG) atomic fluorescence spectrometry (AFS), was developed to improve the sensitivity of mercury determination and to remove the interference of some anion and organics to UVG of mercury. A high mercury retention efficiency and maximum exclusion of inorganic and organic matrix in water samples were achieved by using C_{18} SPE mini cartridge modified with sodium diethyldithiocarbamate (DDTC). Fast and efficient elution from the cartridge was found by using L-cysteine mixing solution. Furthermore, through the investigation of different UV reactor designs, the most important factor was the structure of the reactor (which corresponded roughly to the photon flux) wherein the tubing was sintered into the UV lamp to give the highest UV generation efficiency. The second factor was the materials of the tubing (which roughly corresponded to the working wavelength). Synthetic quartz, characterized by the highest transparency at 185 nm, attained the highest UVG efficiency, suggesting that the most favorable wavelength for UVG was 185 nm. Under optimum conditions, the achievable detection limit (3σ) with sample loadings of 10.0 mL was 0.03 ng L^{-1} and 0.08 ng L^{-1} with different manifolds, respectively. The method was successfully applied to the determination of Hg in tap water, river water and lake water samples.

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1. Introduction

Mercury is a major water pollutant because of its high toxicity and bioaccumulation factor (up to 10^6) [1]. Interest in monitoring it in natural waters has attracted considerable attention [1]. Inductively coupled plasma-optical emission spectrometry (ICP-OES) [2,3], inductively coupled plasma-mass spectrometry (ICP-MS) [4,5], vapor generation-atomic absorption spectrometry (VG-AAS) [6,7], and vapor generation-atomic fluorescence spectrometry (VG-AFS) [8,9] are several viable methods for the determination of mercury. However, ICP-OES is not sensitive enough for most water samples; VG-AAS and ICP-MS are too sophisticated to be used in the field. VG-AFS is a low cost and sensitive method for determination of mercury, but the unstable reductant (KBH_4 or $NaBH_4$) frequently used for this method hampers its field use. Fortunately, recent studies [10–13] reporting on ultraviolet vapor generation (UVG) AFS methods without using the unstable reductants, should prove very suitable for the measurement of mercury in the field. However, many anions, such as NO_3^-, SO_4^{2-}, Br^- have been found to interfere with the UVG of mercury [14,15], and no effective masking method has been found. Apart from anions, the presence of organic matter has also been found to affect UVG. For example, it has been reported that low molecular weight organic compounds such as alcohols, aldehydes, or carboxylic acids give different sensitivities with UVG [16], while humic acid causes depression [15]. Though the anions could be removed by pre-treatment [1,17], most of these methods would commonly bring in some other reagents, such as high concentrations of solvents [18,19], high concentrations of acid [20,21] or thiourea [22,23], which seriously decreases the efficiency of UVG. Thus, despite several methodologies for the separation of mercury from other anions [12], no successful application of such separation coupled with UVG of mercury has been achieved.

There remain several inconsistencies with UVG. For example, Matusiewicz [14] reported that longer irradiation times did not decrease the mercury generation efficiency, but Gao's result [12] suggests the opposite; Wang [24] found that L-cysteine could induce the decomposition of organic mercury, but in Yin's paper [25], it could even generate methylmercury directly, though the sensitivity was low. It is possible that these contradictory conclusions were caused by the different structures of the UV reactors or their working wavelengths, which has not yet been systemically examined.

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In this study, a novel on-line method for the separation of mercury from a number of anions and organic species is reported based on selectively trapping mercury in a C_{18} cartridge modified with diethyldithiocarbamate chelating ligand (DDTC). Subsequent determination is achieved using UVG-AFS. The effects of sample pH, eluent components and flow injection procedure were studied to optimize the working conditions. The design of the reactor and UV working wavelength were systematically studied. Furthermore, the application of the method to the determination of mercury in tap water, river water, lake water and sea water samples was investigated.

2. Instrumentation and reagents

2.1. Instrumentation

A Titan 8220 atomic fluorescence spectrometer (AFS, Beijing Titan Instruments Co., Ltd., Beijing, China) fitted with a mercury high intensity hollow cathode lamp (HCL, Beijing Research Institute of Nonferrous Metals, Beijing, China) as the irradiation source was used for the measurement of mercury. The operational parameters were set in accordance with the manufacturer.

To establish the optimum separation and elution conditions, a flow injection system with AFS detection, as shown in Fig. 1, was assembled in our laboratory. The system comprised a peristaltic pump, a 6-port sampling valve and an 8-port selection valve (Beijing Titan Instruments Co., Ltd., Beijing, China). Three Tygon® pump tubes of 1.84 mm ID (Elkay Products, Inc. Worcester, MA, US) were used to deliver the solutions. PTFE tubes of 0.8 mm ID (Cole-Parmer Instrument Company, Verson Hills, IL, US) were used for connections and liquid delivery.

2.2. UV reactor

For this work, several UVG apparatus based on a 19 W low pressure mercury discharge lamp (LPML) were tested; their structures are shown schematically in Fig. 2. Two types of pipeline layout: "OP" denotes a tube that was coiled around the outside of the lamp body; "IP" denotes a tube that was sintered inside and passed through the interior of the lamp directly within the discharge. The tube materials studied comprised non-ozone quartz (185 nm cut, OP2, IP1), ozone quartz (partly transparent at 185 nm, OP3, IP2), synthetic quartz (transparent at 185 nm, OP4, IP3) and polytetrafluoroethylene (PTFE, 185 nm cut, OP1).

2.3. Reagents

All reagents were of chemical pure grade and purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China unless otherwise stated. The C_{18} cartridge was purchased from Agela Technique, Tianjin, China. It was packed with 500 mg C_{18} 200 mesh size resin, resulting in a volume of 0.5 mL. A standard solution (1000 mg L^{-1}) of mercury was purchased from the National Research Center for Certified Reference Materials, China, and diluted as required. Milli-Q water was used throughout the study.

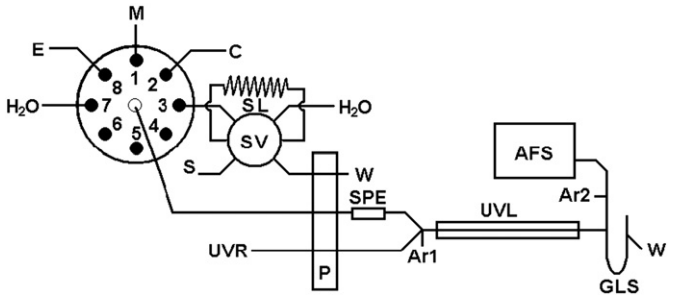

Fig. 1. Schematic diagram of trace mercury analysis systems. 1–8: the ports of the 8-port selection valve; M: cartridge modification solution; C: cartridge washing solution; S: sample; E: eluent; SL: sample loop; SV: sampling valve; H_2O: clean water; SPE: mini on-line solid phase extraction cartridge; UVR: UV reaction solution; UVA: UVA apparatus; Ar1, 2: Ar gas; GLS: gas liquid separator; W: waste; AFS: atomic fluorescence spectrometry.

A resin modification solution was prepared by dissolving 1 g of sodium diethyldithiocarbamate (DDTC) in water, followed by dilution to 1000 mL with Milli-Q water. An eluent was prepared by dissolving 4.62 g ammonium acetate and 0.12 g L-cysteine in water, to which was added 50 mL acetonitrile and the mixture was diluted to 1000 mL with water. A 50% (v/v) acetonitrile solution was used to wash the cartridge.

2.4. Flow injection procedure (Table 1)

The cartridge was activated prior to installation by sequentially passing 10 mL acetonitrile and 20 mL water through it. The cartridge was first rinsed with eluent to remove any carryover of mercury from the previous sample before modified by DDTC; the sample was then pumped through the cartridge, trapping the mercury. After trapping, the cartridge was rinsed by water and 50% acetonitrile solution. Then the collected mercury was eluted, merged with UV reagent and introduced into the UV reactor to generate cold vapor mercury atoms. These were separated from the solution in a gas–liquid separator and swept into AFS detector by argon carrier gas to be measured.

3. Results and discussion

3.1. Retention and elution of mercury on modified C_{18} cartridge

According to Blanco [17], DDTC was used as modifiers for the C_{18} cartridge to selectively trap mercury for analyte enrichment and matrix separation. Moreover, the favorite pH range for the retention to remove the interference by thiuram ligands was studied, and pH = 4–5.4 was chosen.

The trapped mercury could be eluted by using thiourea in an acidic solution or using L-cysteine or mercaptoethanol in a neutral solution. But thiourea hindered the UVG of mercury so severely (see below) that the latter method should be used. In view of the high toxicity of mercaptoethanol, L-cysteine in neutral buffer solution was chosen as

Table 1
Operational sequence of the measurement procedure for mercury determination with on-line enrichment.

No.	Description	Solution	Time (s)	Flow rate (mL min^{-1})	Switch valve position	Sample[a]	Sampling valve
1	Elution	Eluent	30	3	8	1	Loading
2	Modification	DDTC	15	6	1	1	Loading
3	Enrichment	Sample	100	9	3	1	Sampling
4	Water washing	Water	30	5	7	2	Loading
5	Washing	50% (v/v) acetonitrile	30	5	2	2	Loading
6	Elution	Eluent	160	3	8	2	Loading
7	Stop flow	Eluent	15	0	8	2	Loading

[a] For saving sample loading time, the sample was loaded in the previous test procedure after the previous sample was enriched in the SPE cartridge.

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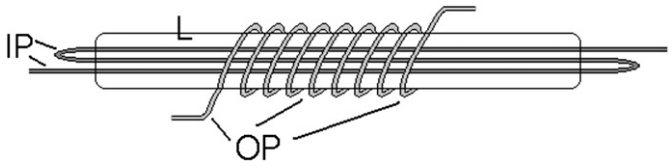

Fig. 2. Schematic diagraph of UV generator, L was the 19 W UV lamp; IP was the pipeline sintered into the lamp; OP was the pipeline coiled around the lamp.

the eluent, and 1 mmol L^{-1} L-cysteine at 3 mL min^{-1} could attain the maximum elution speed.

3.2. Interferences from inorganic and organic species

There was severe interference of UVG from anions and organic materials, especially NO_3^-, ClO^-, NO_2^-, thiourea etc., even at very low concentrations [14–16]. When the UVG was coupled with the above mentioned DDTC modified C_{18} SPE method, the interference was eliminated. Under such conditions, Fe^{2+}, Fe^{3+}, Cu^{2+}, Pb^{2+}, AsO_4^{3-}, humic acid or L-cysteine with concentration 10 mg L^{-1}; Na^+, K^+, Ca^{2+}, $H_2PO_4^-$, $Cr_2O_7^{2-}$, SO_4^{2-}, Br^-, I^-, Cl^-, thiourea or mercaptoethanol with concentration 10 g L^{-1} didn't interfere the recoveries for 100 ng L^{-1} Hg^{2+} (less than 10% deviation with precision less than 3%).

3.3. Response from organic mercury species

With the chemical cold vapor generation AFS method, response from organic mercury species (methylmercury, ethylmercury) was very different from that of Hg^{2+} [26,27], which was with the equivalent of an interference in the measurement of mercury in water by UVG unless pretreatment was undertaken. This was examined in this study by assessing spike recoveries for two organic mercury species in different water samples. Generated with the IP3 UV reactor design, the spike recoveries from different water samples were all in the range of 85–110%, whether HCOOH or L-cysteine was used as the UVG reagent. No significant interference from organomercury species was evident using this method [28,29].

3.4. Selection of UVG reagent

UVG was strongly dependent on the reagents; formic acid, mercaptoethanol, thiourea, DDTC and L-cysteine were investigated in this work. Formic acid gave the highest efficiency, which was equal to conventional borohydride reduction; that for L-cysteine was 1/3 of formic acid; mercaptoethanol was 1/6; DDTC showed little efficiency as blank solution; and thiourea completely inhibited the generation of mercury. All these reagents, except formic acid, could be used to elute mercury that had been trapped on the C_{18} cartridge, suggesting that there were two methods to detect mercury: an extra formic acid solution could be introduced to support UVG post-cartridge for achieving higher sensitivity or the eluent itself could be used as the UVG reagent to simplify the manifold for field use. The eluent containing L-cysteine was selected because of its highest sensitivity.

3.5. UV reactor

There were various contradictions in the field of UVG: Matusiewicz [14] reported that longer irradiation times would increase the generation efficiency of mercury, while Gao's [12] results were just the opposite; Wang [24] found that L-cysteine could only enhance the decomposition of organic mercury, while this work found that L-cysteine could even generate mercury with higher efficiency by UV irradiation. All these contradictions are most likely derived from differences in the UVG reactors. To discover the reason, a group of UVG reactors was examined, all based on a 19 W low pressure mercury discharge lamp (LPML). Their structures were shown schematically in Fig. 2.

The UVG efficiency of each device was shown in Fig. 3. IP3 was the optimum design among the 7 examined and was chosen as the practical design for this work. In addition, whether L-cysteine (Fig. 3a) or HCOOH (Fig. 3b) was to be used as the reagent, the UVG efficiency of 4 OP designs were significantly lower than 3 IP arrangements. These were in accordance with the literature [30], and could be explained by the fact that the interior tubing would sustain higher UV illumination than the outside; this was derived not only from the irradiation within the 360° environment, but also from the enhancement of the actual discharge around the inserted tubing. Moreover, regardless of L-cysteine (Fig. 3a) or HCOOH (Fig. 3b) being used as the UVG reagent, the UVG efficiency of 4 OP designs followed OP4, OP3, OP2, and OP1 in descending order, indicating that the UVG efficiency was affected by the tubing material and followed the order: synthetic quartz > ozone quartz ≫ ozone free quartz ≫ PTFE. For 3 IP designs and L-cysteine serving as the UVG reagent, results were similar to the OP design. When HCOOH was used as the UVG reagent, no difference in efficiency was found among the 3 IP designs. There were two principal emission lines from the LPML in the UV range, 254 nm and 185 nm. All the above mentioned 4 materials were transparent to 254 nm except PTFE, which was only partly

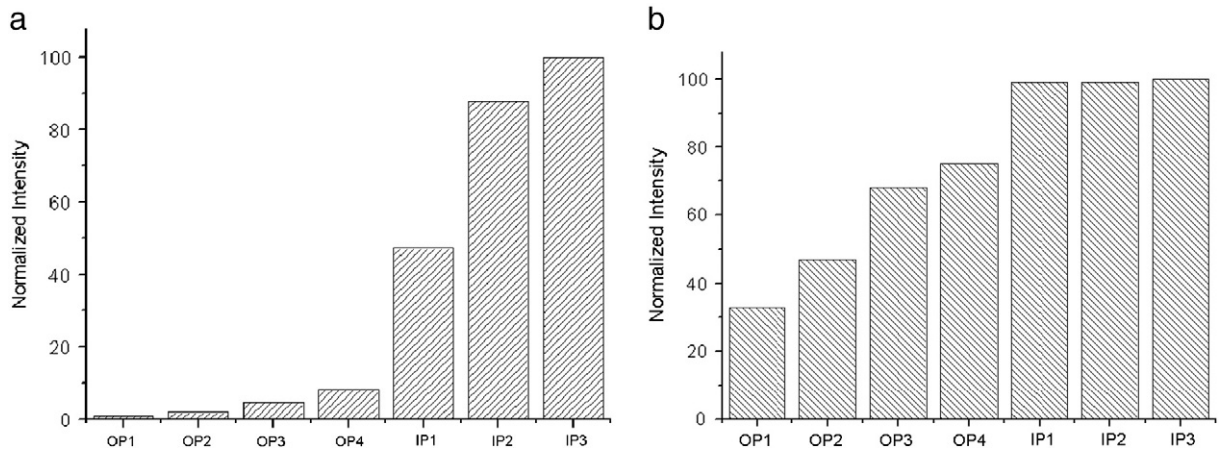

Fig. 3. Effect of different UV apparatus for Hg generation. a: UV generated with L-cysteine; b: UV generated with HCOOH; OP 1 was 0.9 m PTFE pipeline (i.d. 0.8 mm, o.d. 2 mm) coiled on a 19 W UV lamp; OP 2 was 0.9 m none-ozone quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) coiled on a 19 W UV lamp; OP 3 was 0.9 m ozone quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) coiled on a 19 W UV lamp; OP 4 was 0.9 m synthetic quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) coiled on a 19 W UV lamp; IP 1 was 0.9 m none-ozone quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) inserted in a 19 W UV lamp; IP 2 was 0.9 m ozone quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) inserted in a 19 W UV lamp; IP 3 was 0.9 m synthetic quartz pipeline (i.d. 0.8 mm, o.d. 2.5 mm) inserted in a 19 W UV lamp; all intensities of OP apparatus and IP apparatus were normalized by OP 4 and IP 3, respectively.

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Table 2
Test results of different waters.

Description or number	UVG reagent	Certified value (μg L^{-1})	Found[b] (μg L^{-1} or ng L^{-1})	Spiked		
				Value (ng L^{-1})		Recovery (%)[d]
				Added	Found[c]	
GBW (E) 080392	HCOOH	11.0 ± 0.8	11,5 ± 0.5	\	\	\
	None [a]		10.8 ± 0.9	\	\	\
GBW (E) 080393	HCOOH	104 ± 2.0	105 ± 1.1	\	\	\
	None [a]		104 ± 0.7	\	\	\
GSBZ 50016-90 202026	HCOOH	4.12 ± 0.37	4.00 ± 0.2	\	\	\
	None [a]		4.21 ± 0.4	\	\	\
GBW (E) 080042	HCOOH	1.00 ± 0.06	1.05 ± 0.05	\	\	\
	None [a]		0.97 ± 0.07	\	\	\
NRCORMS-3	HCOOH	12,600	12,100 ± 140	\	\	\
	None [a]		13,200 ± 150	\	\	\
River water	HCOOH	\	1.2 ± 0.1	40.0	41.9 ± 0.3	101.6 ± 0.8
	None [a]		1.1 ± 0.2	40.0	41.3 ± 0.4	100.5 ± 1.0
Tap water	HCOOH	\	0.9 ± 0.1	60.0	55.3 ± 1.2	90.7 ± 2.1
	None [a]		1.0 ± 0.2	60.0	54.9 ± 0.7	89.8 ± 1.2
Lake water 1	HCOOH	\	2.1 ± 0.1	20.0	19.5 ± 0.4	86.9 ± 2.0
	None [a]		1.9 ± 0.3	20.0	19.0 ± 0.3	85.5 ± 1.5
Lake water 2	HCOOH	\	3.4 ± 0.2	50.0	50.8 ± 0.8	94.8 ± 1.6
	None [a]		3.6 ± 0.4	50.0	50.4 ± 1.4	93.7 ± 2.9
Sea water 1	HCOOH	\	1.1 ± 0.1	30.0	29.6 ± 0.2	95.1 ± 0.7
	None [a]		1.0 ± 0.2	30.0	29.1 ± 0.4	93.7 ± 1.3
Sea water 2	HCOOH	\	1.4 ± 0.1	100.0	94.6 ± 0.9	93.2 ± 0.9
	None [a]		1.6 ± 0.3	100.0	96.8 ± 1.1	95.2 ± 1.1

[a] At this time, the UV reagent pipeline was taken out, and L-cysteine in eluent was the real UV generation reagent.
[b] Standard deviation for 3 tests, for CRM water samples the unit was μg L^{-1}, for real water samples the unit was ng L^{-1}.
[c] Standard deviation for 3 tests.
[d] Standard deviation for 3 tests.

transparent. For 185 nm, transparency follows the order: synthetic quartz > ozone quartz ≫ ozone free quartz ≫ PTFE, which was in agreement with the order of UVG efficiency. This implied that 185 nm instead of 254 nm was more important for UVG. These results were similar to those of Zheng et al. [31] who reported that Hg^{2+} and MeHg showed different efficiencies for conversion to Hg^0 when irradiated with either a UV lamp or a visible lamp.

3.6. Analytical application

When post cartridge HCOOH was added or L-cysteine was present in the eluent to serve as the UVG reagent, the analytical performance of this method was studied using 10 mL sample volumes. With these two reagents, the linear ranges were both 1–5000 ng L^{-1}, relative standard deviations were both lower than 5% for 100 ng L^{-1}. The detection limit (DL) was 0.03 ng L^{-1} (HCOOH) or 0.08 ng L^{-1} (L-cysteine), respectively. Even the poorer value was sufficient for routine water testing. So, despite the fact that, when HCOOH was used as the UVG reagent, a slightly improved DL can be achieved, L-cysteine remained the best choice because this simplified the manifold and was more suitable for field testing.

The results of the determination of trace mercury in water obtained with the proposed UVG method with the two reagents are summarized in Table 2. The determined values for the CRMs were all located within the certified ranges; the spike recoveries were all in the range 85–110%, suggesting that this method was suitable for routine testing.

3.7. Conclusion

The proposed UVG-AFS method using on-line separation with a DDTC modified C_{18} cartridge enabled efficient elimination of interference on UVG of mercury by anions and some organic materials. On the other hand, the efficiency of UV generation using different reagents showed that in addition to the usually used HCOOH, L-cysteine when present in the eluent was also a powerful UVG reagent. Thus, elution and UV generation could be perfectly combined to yield a trace mercury detector suitable for field testing. The analytical performance and recoveries for CRMs and fortified water samples were all satisfactory.

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