Continuous flow system for arsenate uptake by horticultural peat impregnated with Fe

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ABSTRACT

The objective of this research was to employ horticultural peat impregnated with Fe in column experiments to remove arsenic from water to a level of 10 μ g/L or less. The point of zero charge and the cation exchange capacity of the peat and important elements and metals contained in the peat were determined. Batch tests revealed that the optimum Fe impregnated peat for the column studies was the peat impregnated with 0.54 M Fe. It was possible to reduce the arsenic concentration exiting the column to < 10 μ g/L and to a minimum of 0.61 μ g/L. During the column tests the pH increased and the leaching of Mn, Ca, Mg, Cu, Pb, and Br from the peat decreased.

RÉSUMÉ

L'objectif de cette recherche était d'employer de la tourbe d'horticulture imprégnée de Fe dans des installations en colonne pour éliminer l'arsenic de l'eau et atteindre un niveau égal ou inférieur à 10 μ g/L. On a déterminé le point de charge zéro et la capacité d'échange de cations de la tourbe ainsi que d'importants éléments et métaux contenus dans la tourbe. Des essais par lots ont révélé que la valeur optimale de la tourbe imprégnée au Fe était celle qui était imprégnée de 0,54 M Fe dans les tests sur les colonnes. Il a été possible de réduire la concentration d'arsenic qui sortait de la colonne jusqu'à < 10 μ g/L et d'atteindre un minimum de 0,61 μ g/L. Pendant les tests sur les colonnes, le pH a augmenté et le lessivage du Mn, Ca, Mg, Cu, Pb et Br contenus dans la tourbe a diminué.

1 INTRODUCTION

In Canada and the United States, sources of drinking water from groundwater that contain arsenic above the 10 µg/L limit must be treated. This may be expensive for small rural communities and so more economical arsenic removal is needed. There is also an interest in better understanding the conditions that contribute to arsenic release into waters used for human consumption.

Aqueous phase arsenic exists as arsenate, As (V) and arsenite, As(III) (Lorenzen et al., 1995). Soluble arsenite can be oxidized to arsenate which can be adsorbed to soils and then become mineralized (Environment Canada, 1995). Arsenate exists in water as H_3AsO_4 (at < pH 2.2), H_2AsO_4 (from pH 2.2 – 6.7) and $HAsO_4$ (from pH 6.7 – 11.6) (Chuang et al; 2005).

Arsenic is effectively removed from water by adsorption onto activated alumina, granular activated carbon (AC) and engineered oxides of Fe, Al and Ti, and by ion exchange, Fe oxide precipitation, lime softening, coagulation/filtration, membrane filtration, osmosis, and reverse electro-dialysis (Berg et al., 2006; García et al., 2004; Partey et al., 2008; Poots and McKay, 1979) though methods employing Fe oxides are most common (García et al., 2004). Employing energy intensive processes (such as AC adsorption or reverse osmosis) may not be sustainable (Høibye et al., 2008; Jones et al., 2007) or generally affordable (Bert et al., 2006).

Adsorption has often been preferred for arsenic removal (Pokonova, 1998) but inexpensive and appropriate sorbents are needed. Peat is abundant and economical, and as with AC has a high organic content, but there are limited studies of arsenic adsorption with peat.

Under oxidizing conditions arsenic is taken up predominantly with Fe forming scorodite, FeAsO $_4$ ·2H $_2$ O and under reducing conditions (in aquifer walls) arsenic is incorporated into sulfide minerals such as orpiment, As $_2$ S $_3$ and realgar, AsS. Arsenic commonly forms inner sphere complexes with Fe oxy-hydroxides and oxide surfaces and is attracted to the positively charged surfaces of AC below pH 7 – 7.5, and Fe impregnated AC below pH 8.2 – 8.7 (Budinova et al., 2006; Deutsch, 1997; Evans, 1989; Reed et al., 2000). Adsorption of arsenate by oxide surfaces is maximized near pH 4 and competes with antimony, silica and phosphorus for adsorption sites (Dixit and Hering, 2003; Ngo et al., 2002).

When the peat is impregnated with Fe, it is oxidized to form Fe oxy-hydroxide or FeOOH and ligand exchange (and uptake) of arsenic with the OH (carboxyl and hydroxyl) functional groups is promoted (Gu et al., 2005; Lorenzen et al., 1995; Manju et al., 1998; Subramanian et al., 1997; Viraraghavan and Kapoor, 1995).

The Fe concentration in solution influences impregnation. Initially the Fe may be bonded organically with the peat but as organic sites become saturated and if oxidizing conditions are encouraged, Fe oxide precipitation may occur (Henrot and Wieder, 1990). In

this study horticultural peat impregnated with Fe was investigated with the aim of achieving arsenic removal to $< 10 \mu g/L$ and understanding the factors important in arsenic uptake.

2 MATERIALS AND METHODS

Horticultural peat was obtained from Traverse Gardens Plant Nursery in Torbay, Newfoundland. The peat had a pH of 4.2 and was classified on the Von Post scale as 3H mixed with some 6H (10H represents the greatest decomposition) (Asapo and Coles, 2008).

2.1 Peat Impregnation

To impregnate the peat the procedure of Gu et al. (2005) was followed with changes being that a 10:1 weight ratio of impregnating solution to peat was employed, four solution concentrations of $FeCl_2$ (tetra hydrate 99+%, Fisher Scientific) of 0.054, 0.108, 0.54, and 1.08 M were used, 10 ml of sodium hypochlorite (NaClO) were added every 6 hours, and the solutions were shaken continuously. After the more soluble Fe^{2+} diffused through the peat it was oxidized to Fe^{3+} (to form stronger complexes with peat ligands) using NaClO in a 9-12 % solution with active chloride of 5.68 % (A&C American Chemicals Ltd.). At pH > 3, Fe^{3+} can be easily hydrolyzed to Fe oxy-hydroxide which has a strong affinity for arsenate (Gu et al., 2005; Brown et al., 2000). The Fe impregnated peat is hereafter referred to as the Peat-Fe.

HCl and NaOH (A&C American Chemicals Ltd.) were used to adjust solution pH to between 4.5 and 5 for maximum solubility of ${\rm Fe}^{2+}$ during the first 6 hours and above pH 5 for the remainder of the impregnation process.

2.2 Characterization of untreated peat and Peat-Fe

The point of zero charge (PZC) was determined by titration with acid and alkali (Tschapek et al., 1974) as described by (Coles and Yong, 2002) except that 2 ml of 0.1 N HCl or 4 ml of 0.01 N NaOH were added every 15 min while recording the change in pH. The cation exchange capacity (CEC) was measured in triplicate following the Ca(OAc) 2 - CaCl2 method (Analytical Methods Manual, 1984) but with quantities reduced to one third. To prepare the peat samples (untreated peat and Peat-Fe) for the detection of Fe, arsenic and other elements they were digested according to the US EPA Method 3050B "Acid Digestion of Sediments, Sludge and Soils" (American Chemical Society, 1986). To obtain the moisture content of the untreated peat, six samples of 15 g each were heated at 110 Co for 24 hours (Mondal et al., 2007; Poots and McKay, 1979).

2.3 Batch and column tests

Batch tests in 10:1 and 10:2 weight ratios of arsenic solution to dry peat were conducted on peat soils that had been impregnated with four different concentrations of Fe to determine the best Peat-Fe sample to be used in the

column studies. Blank and spiked batch tests employed 6.1 µg/L and 60 µg/L arsenic solutions.

Leaching column tests were conducted with the Peat-Fe soil that retained the most arsenic during the batch tests. Vertical Plexiglas cylinders 12.8 cm high by 6.4 cm in diameter were fitted with 0.635 cm thick and just less than 6.4 cm in diameter porous ceramic plates at the upper inlet and lower outlet. The ceramic plates (Hoskin Scientific) had a porosity of 0.5, a maximum pore size of 6 μm , a saturated hydraulic conductivity of 3.11 \times 10 $^{-5}$ cm/s and a flow through of 180 mL/h/cm²/14.7 psi. The column was manufactured by Technical Services at Memorial University of Newfoundland and the influent supply was 1.55 m above the inlet.

After Fe impregnation and before acid digestion or use in the batch and column tests, the Peat-Fe samples were oven dried, crushed, washed with distilled water to remove excess Fe, air-dried for 24 h, and ground to a powder. For the column tests the powdered sample was placed in the cell, one liter of distilled water was passed through the cell, and after the sample expanded the extra soil was removed. The effluent collected during the first 5 hours was discarded as it was mixed with distilled water.

All solutions were filtered through a 0.45 μm filter paper and acidified with HNO₃ before detection of elements with a Hewlett-Packard 4500 series ICP-MS.

2.4 Statistical Analysis

Minitab was employed during the early stages of the column tests to find correlations between the effluent pH and arsenic concentrations and other element concentrations in the effluent.

3 RESULTS

3.1 Peat characterization

The PZC and CEC were determined for the untreated peat and the four Peat-Fe soils impregnated with 0.054, 0.108, 0.54 and 1.08 M FeCl $_2$ and the PZC values occurred at pHs of 2, 3, 2.9, 3 and 3.3 respectively, and the average CEC values were 274, 22.5, 17.4, 17.1 and 8.62 meq/100 g respectively.

Iron impregnation of the peat samples raised the PZC from pH 2 to an average of pH 3.05 and significantly reduced the CEC values compared with the untreated peat. Displacement of H⁺ ions on the functional groups of peat during the Fe impregnation process could have prevented much exchange of H⁺ ions by the Ca²⁺ ions used to measure the CEC. A CEC of 246 meq/100 g for a sphagnum moss horticultural peat from the Ottawa, Ontario area (Warith, 1996) is comparable to the CEC of this untreated horticultural peat of 274 meg/100 g.

Iron, arsenic and other selected element contents of the untreated peat and the initial Peat-Fe soils are shown in Table 1. The concentrations of Fe in the Fe impregnated samples do not increase in proportion to the level of Fe impregnation, but reach a maximum in the peat impregnated with 0.54 M Fe²⁺ as FeCl₂ (Peat-Fe 0.54 M). The percentages of Fe taken up by the samples

Table 1. Initial metal and arsenic contents of five peat soils (in mg/kg).

Peat Soils	As	Fe	Mn	Ca	Mg	Cu	Pb	Br
Untreated peat	ND⁵	946	27.9	2,514	NA ^c	0.86	3.37	108.1
Peat-Fe 0.054 M ^a	0.52	54,100	19.9	410	NA	6.28	3.02	NA
Peat-Fe 0.108 M ^a	0.69	49,400	14.1	440	NA	4.26	3.36	NA
Peat-Fe 0.54 M ^a	0.63	136,000	24.9	250	NA	3.71	4.22	NA
Peat-Fe 1.08 M ^a	0.53	114,000	21.9	200	NA	4.66	5.06	NA

^aPeat soils impregnated with indicated molar solutions of Fe²⁺ as FeCl₂

Table 2. pH and arsenic and Fe contents (in μg/L) of the influent to, and the effluent from, the batch tests.

Spiked tests				Spiked tests			Blank tests		
Peat soils	10:1 solution:soil ratio			10:2 solution:soil ratio			10:1 solution:soil ratio		
	As	рН	Fe	As	рН	Fe	As	рН	Fe
Influent solution	60	5.9	589	60	5.9	185,000	6.1	5.9	190
Peat-Fe 0.054 M ^a	64.0	3.0	1,130,000	117	2.9	660,000	141	3.0	209,000
Peat-Fe 0.108 M ^a	67.9	2.5	1,730,000	181	2.3	140,000	106	2.6	147,000
Peat-Fe 0.54 M ^a	29.8	3.1	275,000	90.1	3.1	211,000	32.9	3.2	68,000
Peat-Fe 1.08 M ^a	44.7	3.2	96,000	88.4	2.8	275,000	138	3.3	191,000

^aPeat soils impregnated with indicated molar solutions of Fe²⁺ as FeCl₂

impregnated with 0.108, 0.54 and 1.08 M Fe²⁺ were 82, 45 and 19% respectively. The optimum Fe concentration for impregnating the peat was probably surpassed at 1.08 M Fe²⁺. Smaller percentages of metals adsorption by peat at higher metal dosing concentrations have been documented for Cu, Zn, and Ni (Viraraghavan and Dronamraju, 1993). The Ca was much more abundant in the untreated peat (made possible by the higher CEC) and was probably leached out by the acidic Fe impregnating solution.

The 15 g samples of horticultural peat had a moisture content of 80% as they weighed an average of 3.01 g after drying.

3.2 Batch Tests

Batch tests with the four Peat-Fe soils revealed that the Peat-Fe 0.54 M sample retained the most arsenic overall as seen from Table 2. For this reason it was this soil that was selected for the column experiments and it was more economical than the Peat-Fe 1.08 M.

The arsenic may not have been as effectively retained at the highest level of Fe impregnation in the batch tests because blocking of inner pores of peat by Fe oxyhydroxide formation can occur if the Fe levels in the impregnating solution are too high (Gu et al., 2005).

3.3 Column Tests

Column tests with untreated peat and Peat-Fe 0.54 M were conducted to show the sorption of arsenic in a flow through system. The untreated peat was ineffective in removing arsenic as an initial arsenic concentration of 62.9 μ g/L was reduced to 2.65 μ g/L in the effluent at 12 h but increased to 42.6 μ g/L at 108 h when only 1L of

solution had passed through the column. This is illustrated in Figure 1.

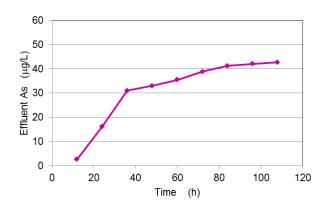


Figure 1. Effluent arsenic concentrations (μ g/L) as a function of time (h) in the untreated peat column test.

The column tests employing the Peat-Fe 0.54 M were conducted with a blank solution that had an average influent arsenic concentration of 6.1 $\mu g/L$ and with a spiked solution that had an average influent arsenic concentration of 58.1 $\mu g/L$. Since similar trends were observed for both column tests, only data for the spiked column test are reported and can be found in Table 3.

The top row of Table 3 gives the average values for the influent to the spiked column test, since there were slight variations in the arsenic and other element concentrations in each liter that was mixed. During the first 72 h of the column test and for first 18 measurements significant correlations of \geq 0.444 (Vincent, 1995) between

^bNot detected

^cNot available

Table 3. Influent and effluent element concentrations (in μg/L) for the Peat-Fe 0.54 M spiked column test, and significant correlations among arsenic, pH, and other elements.

Time (h)	As	рН	Fe	Mn	Ca	Mg	Cu	Pb	Br
Influent	58.1	5.9	287	8.7	83.8	11.7	25.2	1.8	10.6
2	39	3.4	283,000	380	18,300	14,200	14,700	5,200	620
4	35	3.6	344,000	390	17,400	13,600	12,000	3,400	720
6	28	3.7	393,000	320	14,000	10,400	8,610	2,010	730
8	17	4	224,000	230	9,260	9,910	6,300	1,570	540
10	12	4.1	279,000	150	6,110	4,620	6,410	1,470	380
12	11	4.3	157,000	130	5,030	3,890	5,490	1,560	320
14	7	4.5	165,000	90	3,360	2,550	3,540	1,710	210
16	4	4.6	124,000	60	2,290	1,760	4,160	1,780	150
18	4	4.6	114,000	60	2,190	1,490	4,970	1,780	130
24	7	4.1	144,000	50	1,350	1,220	8,630	1,210	320
30	7	4.5	88,100	10	430	320	3,740	1,040	50
36	4	4.3	56,600	10	290	230	2,820	840	25
42	2	4.2	38,000	10	220	170	2,180	550	10
48	3	4.5	30,800	10	160	140	2,100	500	10
54	0	4.3	22,900	10	220	110	2,000	410	10
60	3	4.6	34,900	10	380	450	4,820	600	60
66	37	4.9	60,200	30	1,080	1,420	6,370	420	150
72	16	4.5	24,600	20	480	610	2,700	290	50
96	0.61	4.2	16,400	40	1,810	410	2,900	680	0
120	2.92	4.2	25,000	30	1,820	450	2,500	910	0
144	4.93	4.1	22,600	20	1,810	310	3,220	0.69	0
168	2.95	4.3	15,500	10	420	220	1,140	20	0
192	3.32	4.7	4,720	10	470	640	3,430	70	0
240	3.32	4.5	16,800	10	420	240	5,200	60	0
264	3.33	4.6	19,500	10	420	210	4,800	40	0
288	5.78	4.7	20,000	10	420	170	3,490	30	10
	Significa	nt correlati	ions within the	first 72 h of	arsenic and p	H with the p	arameters i	ndicated	
Arsenic	1	-0.5	0.62	0.76	0.76	0.79	0.79	0.63	0.73
pН	-0.52	1	-0.80	-0.87	-0.88	-0.87	-0.75	-0.78	-0.84

arsenic and pH and other elements (Fe, Mn, Ca, Mg, Cu, Pb and Br) were noted and these correlations are recorded in the bottom two rows of Table 3.

At 66 and 72 h the arsenic concentrations in the effluent had become greater than 10 μ g/L and shrinkage of the soil column was noticed and so after 72 h more (dry) peat was added at the top of the column and compacted into the cell. To increase the residence time after 168 h (dry) peat was again added to the cell and compacted. At both times the peat that had been originally in the column was partially air-dried and the remaining moisture was enough to compact the drier peat being added. The times of 96 and 192 h (which are bolded) indicate the times of the first measurements taken after fresh peat had been added.

At 14 h and pH 4.5 the arsenic concentration decreased to 7 $\mu g/L$. From 14 to 60 h four of the first five liters of solution were treated to < 10 $\mu g/L$ of arsenic. Following the compaction after 72 h, arsenic in the effluent decreased to 0.61 $\mu g/L$, and subsequently five more liters were passed through the column at an average rate of 0.0231 L/h until 288 h when the arsenic concentration in the effluent reached only 5.78 $\mu g/L$ as also shown by the breakthrough curve in Figure 2.

During the first 16 to 18 h of the column test the greatest increase in effluent pH occurred and it coincided with the first large reduction of the arsenic concentration in the effluent. Between 18 and 24 h a small pH drop in the effluent coincided with a small arsenic increase. From 24 to 60 h the arsenic concentration in the effluent

remained low (< 10 μ g/L) and the pH fluctuated between 4.1 and 4.6. After the peat shrinkage in the column around 66 and 72 h followed by the addition of more fresh peat, there was a small dip in pH that coincided with the lowering of the arsenic concentration in the effluent. From 96 h onward the pH ranged from 4.1 to 4.7 and the arsenic concentration in the effluent remained low (< 10 μ g/L), possibly helped by the second addition of more fresh peat after 168 h.

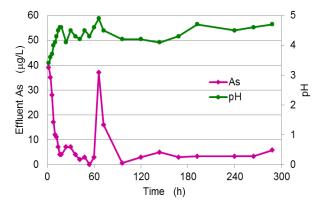


Figure 2. Effluent arsenic concentrations (μg/L) as a function of time (h) in the spiked peat column test.

The net surface charge on the peat was always negative above pH 3, the PZC. With regards to the effluent from the column test during the first 72 h, the positive correlation between arsenic and Fe and the negative correlations between pH and arsenic, and between pH and Fe suggest that as pH increased, this may have helped to stabilize the impregnated Fe. and arsenic bonding increased possibly by complexation with Fe and or co-adsorption with Fe onto the peat. Mn, Ca, Mg, Cu, and Pb were also significantly positively correlated with arsenic and significantly negatively correlated with pH in the spiked column test (and in the blank column test). The increasingly negative character of the Peat-Fe surfaces with increasing pH could have increased the retention of Mn, Ca, Mg, Cu, and Pb and permitted the co-adsorption of arsenic with these other metals.

Though not shown because the trends were not similar in both the blank and spiked column tests, AI was significantly positively correlated with arsenic and significantly negatively correlated with pH in the spiked column test, and Zn, Cr and Co were significantly positively correlated with arsenic and significantly negatively correlated with pH in the blank column test.

Other studies have likewise found that Mn, Ca, Mg, Cu and Pb may influence arsenic uptake/release. Fe, Al, Mn, Ca and Mg may have promoted complexation of arsenate with humic substances (Lin et al., 2004) and Cu and Pb appeared to assist humic acid enhanced mobilization of arsenic from mine tailings (Wang and Mulligan, 2009). Ca improved ferric hydroxide precipitation and removal of arsenic (Ruiping et al., 2007) and Ca and Mg increased the positive character of ferric chloride and countered the silicate ion to increase arsenate removal (Meng et al.,

2000). Selective adsorption of Pb, Cu, Ca, Mg, Zn, and Fe onto different types of peat are mentioned in one or more of the studies reviewed by Brown et al. (2000).

Br was significantly positively correlated with arsenic and significantly negatively correlated with pH in both the blank and spiked column tests.

In coastal eastern Newfoundland enhanced leaching of arsenic into groundwater may have been promoted by Br captured in rainfall (Rageh et al., 2007). A significant and simultaneous increase in the arsenic and Br concentrations was noted in the Bandaiko hot spring waters, Gunma, Japan in the mid 1980's (Kikawada et al., 2004). Br usually occurs as Br (Davis et al., 1998) and in this study the arsenic occurred as arsenate or H₂AsO₄ (Chuang et al., 2005). Similar and competitive uptake/release mechanisms for arsenate and Br may be suggested by the findings in this study.

Table 3 shows the Peat-Fe was able to remove arsenic to very low concentrations and leached both essential ions and Pb into the water. At 288 h and pH 4.7 the Pb concentration was three times the acceptable drinking water level of 10 μ g/L. However, the trends are suggesting that column tests at a higher pH would reduce arsenic and Pb contents in the effluent.

At the end of the spiked column test peat samples from the top, middle and bottom of the leaching cell were analyzed for their contents of arsenic, Fe, and other elements and the results are presented in Table 4. Element concentrations for the untreated peat and the Peat-Fe 0.54 M soil before the leaching column test are also included to make it easier to compare the results. The Fe impregnation process increased the Fe concentration in the peat by more than two orders of magnitude though some of the Fe was washed out during the column test.

The Ca was most noticeably removed from the peat during Fe impregnation with an acidic solution that reduced the CEC and increased the PZC. The arsenic tended to accumulate at the upper layer of the soil column while there appeared to be downward migration of both Ca and Pb. Fe and Mn showed the lowest concentrations at the middle of the leaching cell (where the conditions might be the most oxygen deprived if this factor could be considered significant).

The Mn levels found at three locations in the soil column at the end of the test are comparable amongst themselves but at least one order of magnitude too high when compared with the rest of the Mn data. The ICP-MS used in this study is also used by others and the authors think that there may have been some carry over from another's prior sample(s). Except for these three readings that were taken at the same time, the Mn concentrations in Tables 1, 3, and 4 and in the blank column test appeared to be consistent.

5 CONCLUSIONS

In the batch tests the peat-Fe 0.54 M retained the most arsenic. Pore blockage by Fe oxy-hydroxide formation of the peat impregnated with the maximum FeCl₂ may have reduced some arsenic uptake. Low

Table 4. Concentrations of selected elements (mg/kg) in the untreated Peat, the Peat-Fe 0.54 M before the spiked column test, and the Peat-Fe 0.54 M after the spiked column test and at three column locations.

Sample and Location	As	Fe	Mn	Ca	Mg	Cu	Pb	Br
Untreated Peat	ND^a	946	27.9	2,514	NAb	0.86	3.37	108.1
Initial Peat-Fe	0.63	136,000	24.9	250	NA	4.66	4.22	NA
Final Peat-Fe, Top	4.39	123,000	909	128	150	11.3	7.1	NA
Final Peat-Fe, Middle	2.03	112,000	631	136	170	11.1	12.2	NA
Final Peat-Fe, Bottom	0.61	124,000	858	190	180	12.7	38.3	NA

^aNot detected ^bNot available

concentrations of Fe impregnating solution achieved higher percentages of Fe impregnation.

During the column tests with the Peat-Fe 0.54 M, lower levels of arsenic in the effluent and prolongation of the column life were achieved by allowing the soil in the column to partially dry and adding more dry soil at the top of the column and compacting all of the soil. Arsenic removal to < 10 $\mu g/L$ and better was achievable. Analyzing column sections at the end of the test also indicated that the arsenic tended to accumulate at the top of the column suggesting that the soil had not yet been completely exhausted.

To some extent, during the first 72 h of the column tests, it appears that as effluent pH increased the arsenic, Fe, Br, Mn, Ca, Mg, Cu and Pb in the effluent decreased. It is possible that as the peat surfaces became more negatively charged there was greater uptake of the divalent metal cations (Fe, Mn, Ca, Mg, Cu and Pb) and correspondingly better removal of the monovalent arsenate and Br. Br appears to be important in the uptake/release of arsenic though it might be more so in coastal regions such as Eastern Newfoundland and Japan.

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