

Transport stability of pesticides and PAHs sequestered in polyethylene passive sampling devices

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Abstract Research using low-density polyethylene (LDPE) passive samplers has steadily increased over the past two decades. However, such research efforts remain hampered because of strict guidelines, requiring that these samplers be quickly transported in airtight metal or glass containers or foil wrapped on ice. We investigate the transport stability of model pesticides and polycyclic aromatic hydrocarbons (PAHs) with varying physicochemical properties using polytetrafluoroethylene (PTFE) bags instead. Transport scenarios were simulated with transport times up to 14 days with temperatures ranging between -20 and 35 °C. Our findings show that concentrations of all model compounds examined were stable for all transport conditions tested, with mean recoveries ranging from 88 to 113 %. Furthermore, PTFE bags proved beneficial as reusable, lightweight, low-volume, low-cost alternatives to conventional containers. This documentation of stability will allow for more flexible transportation of LDPE passive samplers in an expanding range of research applications while maintaining experimental rigor.

Keywords PAH · Pesticide · LDPE · Passive sampling device · Transport stability · Storage

Introduction

Passive sampling devices made from low-density polyethylene (LDPE) or other polymers have been used for over two decades to sample the freely dissolved fraction of organic contaminants in numerous environmental media (Huckins et al. 2006; Mills et al. 2013). LDPE passive samplers have been used to sample non-polar and semipolar compounds in air (Paulik et al. 2015; Tidwell et al. 2015), water (Allan et al. 2012; McDonough et al. 2014) and sediment porewater (Fernandez et al. 2014; Liu et al. 2013). Contaminants diffuse into passive samplers, and concentrations increase until equilibrium is reached with the sampled matrix. The first generation of samplers, called semipermeable membrane devices (SPMDs), consisted of LDPE strips containing a volume of triolein to retain sequestered hydrophobic contaminants (Huckins et al. 1990). Recent single-phase variations without triolein afford simpler extraction and analytical cleanup (Adams et al. 2007; Anderson et al. 2008; Mills et al. 2013). LDPE passive samplers are constructed from low-cost materials and are often more cost-effective compared to active sampling methods (Melymuk et al. 2014; US Environmental Protection Agency 2012). Additionally, performance reference compounds (PRCs), also called depuration compounds, are infused into the passive sampler material before deployment. The rate at which PRCs diffuse from the material into the surrounding environment, either air or aqueous, corresponds to the rate at which compounds are sequestered from that surrounding environment (Huckins et al. 2002; Melymuk et al. 2014). The use of these PRCs, along with solvent extraction and instrumental analysis, allows for determination of

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time-weighted averages of bioavailable freely dissolved or vapor-phase environmental concentrations.

Guidelines proposed by the US Environmental Protection Agency (2012) and the US Geological Society (Alvarez 2010) indicate that field-deployed LDPE passive samplers or SPMDs should be stored immediately in airtight cans or jars and transported frozen or near frozen via overnight courier or as soon as possible. Overnight frozen shipping can be expensive or logistically unattainable from some locations (Anderson et al. 2014). Moreover, airtight canisters are suggested for passive sampler transport to and from the study site as a means to suspend sampling and to prevent loss of compounds by volatilization. Recommended canister materials are either glass or metal to limit compound absorption to canister surfaces (Huckins et al. 2006). Rigid canisters add volume and weight that may increase shipping costs. Other transportation guidelines propose wrapping passive samplers in clean aluminum foil and subsequently placing them in plastic bags (US Environmental Protection Agency 2012). While plastic bags are more amenable to shipping, a barrier of pre-cleaned aluminum foil is needed to prevent direct exchange of compound between the passive sampling material and the polymer of the transport bag, often polyethylene. Additionally, polyethylene bags are neither airtight nor chemically impervious, and vapor-phase chemicals can potentially diffuse through the polyethylene bag and be captured by the LDPE passive sampler during transport. The polyethylene bag itself may also sequester contaminants that volatilize from the passive sampler. Alternatively, bags made of polytetrafluoroethylene (PTFE) would provide an airtight, lightweight, low-volume, and chemically inert solution for cost-effective shipping. The use of such PTFE bags is only supported by limited data regarding silicone, rather than LDPE passive samplers (O'Connell et al. 2013). To the authors' knowledge, there are no studies of transport of LDPE passive samplers in PTFE bags. Data-based criteria for transport conditions will increase the utility of passive sampling techniques in an expanding range of applications.

Transportation at ambient temperatures in lightweight, durable bags would allow more cost-effective shipping or transport compared to airtight metal cans or glass jars shipped overnight on ice. In contrast to samples wrapped in aluminum foil and enclosed in polyethylene bags, the PTFE bags are airtight and chemically inert, eliminating the need for foil. We hypothesize that less stringent transport conditions will have no effect on concentrations of commonly studied contaminants sequestered in LDPE passive samplers. The aim of this work is to demonstrate the stability of model pollutants in LDPE passive samplers under simulated transport in PTFE bags, with temperatures between -20 and 35 °C and for durations between 10 h and 14 days. These conditions were chosen to mimic a worst-case scenario of a 14-day transport from a hot climate. Model compounds include organochlorine and

organophosphate pesticides and polycyclic aromatic hydrocarbons (PAHs).

Materials and methods

Standards, solvents, and materials

Pesticide (alachlor, alpha-BHC, chlorpyrifos, and endrin ketone) and PAH (anthracene, benzo[ghi]perylene, chrysene, and fluoranthene) compounds were selected to represent a range physicochemical properties (Table 1). All were of purity ≥ 98 % (AccuStandard, USA). Tetrachloro-meta-xylene and PCB-209 (AccuStandard, USA) were used as extraction surrogate standards for pesticides, and phenanthrene-d10, fluoranthene-d10, chrysene-d12, and benzo[ghi]perylene-d12 were used for PAHs (CDN Isotopes, Canada). Internal standards 4,4'-dibromooctafluorobiphenyl (Supelco Analytical, USA) and perylene-d12 (Chemservice, USA) were added immediately before instrumental analysis to correct for instrument variation (Table 2). Hexane solvents were Optima™ grade or better (Fisher Chemical, USA). PTFE transport/storage bags and Clip N Seal closures were purchased from Welch Fluorocarbon, Inc. (USA). LDPE lay-flat tubing used to make passive samplers was purchased from Brentwood Plastics, Inc. (USA). Average width of tubing is 2.7 cm, average membrane thickness is 75–95 μm , and average transient polymer cavity size is 10 Å (Anderson et al. 2008).

Sample preparation

Passive samplers were constructed from LDPE tubing cut into 100-cm lengths. Each LDPE strip was pre-cleaned to remove potential chemical interferences with three successive conditioning washes in 100 mL of hexanes, each for 24 h. After drying, each strip of tubing was heat-sealed at one end, infused with <100 μL of target compound solution in *n*-hexane (200–600 ng of each compound per strip), and then heat-sealed at the remaining end. Pressure was applied lengthwise between (gloved) thumb and index finger to uniformly disperse target compound solution throughout the sealed LDPE sampler. The same target compound solution was used in all LDPE strips, and all were constructed in one batch. This method of infusion and heat sealing was chosen because it requires less solvent than equilibration techniques as in Booi et al. (2002). Unlike SPMDs which can contain 1 mL of triolein in each strip of tubing (Huckins et al. 1990), the constructed strips contained only a small volume and are considered single-phase samplers. Each passive sampler strip was placed in an individual PTFE bag (Fig. 1). Samples were immediately moved to dark, temperature-controlled environments at -20 , 4, 20, or 35 °C. Ambient light was minimized during laboratory preparation steps. The PTFE bags used in this study

Table 1 Model compounds used in transport stability analysis

		CAS number	Molecular weight (g mol ⁻¹) ^a	log K_{ow} ^a	log K_{oa} ^a	IDL (ng/mL) ^c
Pesticide	Alachlor	15972-60-8	269.77	3.52	10.0 ^b	0.5
	Alpha-BHC	319-84-6	290.83	3.72	8.84	2.0
	Chlorpyrifos	2921-88-2	350.59	4.66	8.88	0.5
	Endrin ketone	53494-70-5	380.91	4.99 ^b	11.1 ^b	1.0
PAH	Anthracene	1719-06-8	178.23	4.45	7.55	1.7
	Benzo[ghi]perylene	191-24-2	276.33	6.70 ^b	12.0	1.7
	Chrysene	218-01-9	228.29	5.81	9.48 ^b	1.7
	Fluoranthene	206-44-0	202.25	5.16	8.88	1.7

^a US Environmental Protection Agency (2015)

^b Estimated value

^c Instrument detection limits (IDL) for extracts of LDPE are determined as three times the standard deviation of seven runs of the lowest standard, expressed as concentration

are translucent and attenuate UVA and UVB transmittance by 49 % (Supplementary Information Fig S1). UV degradation of chemicals was not examined in this study but is expected to be minimal based on previous findings of reduced rates of photodecomposition of PAHs when adsorbed to coal ash (Korfmacher et al. 1980) and silicone passive sampling devices (O'Connell et al. 2014).

Eight samplers were extracted immediately following preparation to represent the $t=0$ treatment. Four samplers from each temperature treatment were extracted at 10 h, 1.5 days, 3 days, and 7 days. An additional four samplers at 35 °C were extracted after 14 days. Passive samplers

were extracted with two 40 mL *n*-hexane dialyses following the addition of extraction surrogate standards. Dialysates were combined and quantitatively reduced to a volume of 1 mL. Extracts were stored in the dark in amber glass vials at -20 °C until analyzed.

Instrumental analysis

Instrumental analysis for each of the model compounds was performed on two methods (Table 2). Pesticides were quantified with gas chromatography with electron capture detectors (GC-ECD). PAHs were quantified with gas chromatography

Table 2 Analytical parameters

	Pesticide method	PAH method
Extraction surrogate standards	Tetrachloro-meta-xylene, PCB-209	Phenanthrene-d10, Fluoranthene-d10, Chrysene-d12, Benzo[<i>a</i>]pyrene-d12
Internal standard	4,4'-Dibromooctafluorobiphenyl	Perylene-d12
Gas chromatograph	6890 N (Agilent)	7890A (Agilent)
Detector(s)	2× micro-electron capture detectors	5975C mass spectrometer (Agilent)
Column(s)	DB-XLB and DB-17MS (both Agilent)	DB5-MS (Agilent)
No. of calibration points ($R^2 > 0.98$)	5	6 or 7
Temperature program	110 °C, 1 min 4 °C/min to 300 °C, hold 10 min	60 °C, 1 min 30 °C/min to 180 °C 3 °C/min to 230 °C, hold 5 min 28 °C/min to 280 °C, hold 10 min 8 °C/min to 310 °C 16 °C/min to 350 °C, hold 5 min
Reference	Anderson et al. (2014)	Allan et al. (2012)



Fig. 1 LDPE passive sampling strip in PTFE bag

with mass spectrometry (GC-MS). All concentrations were quantified by the relative response of the internal standard to target compounds in a five- to eight-point calibration curve (all $R^2 > 0.99$). Instrument detection limits are given in Table 1, and analytical parameters are given in Table 2.

Statistical analysis

Treatment recoveries were scaled as a percentage of the mean control ($t=0$) treatments. Mean percent recoveries were analyzed by one-sided Dunnett's tests. Significance for all tests was set at $\alpha=5\%$. Statistical analyses were performed with JMP Pro 11.2.0 and Microsoft Excel 2013.

Quality control

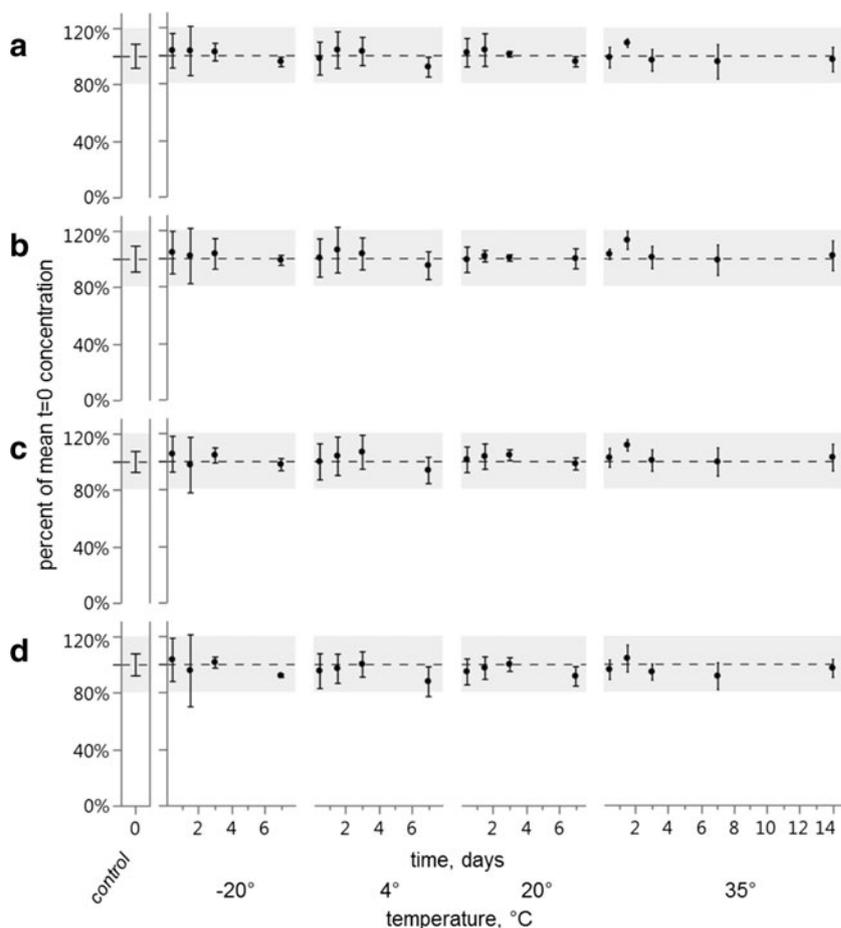
Over 30 % of the samples analyzed were quality control samples. Blank LDPE samples were pulled during the pre-cleaning and construction phases and retained as quality control samples. The extraction process was performed without LDPE for a solvent extraction blank. Injections of *n*-hexane solvent represented instrument reagent blanks and were included in all analytical batches to demonstrate that the instruments had low background responses. All target compounds were below detection limits in all blank quality control samples. Continuing calibration verifications consist of a solution of known concentration of all target compounds to monitor instrument performance and were within 20 % of known value for all target compounds. Extraction surrogate standards were added to passive samplers prior to extraction in order to quantify procedural recoveries. Pesticide surrogate recoveries averaged 92 % (standard deviation = 10 %), and concentrations were not corrected for procedural losses. Recoveries of PAH surrogate standards averaged 65 % (standard deviation = 11 %), and PAH concentrations were corrected for losses.

Results and discussion

Overall mean recovery was 101 % (standard deviation = 6 %) of $t=0$ across all time and temperature treatments for all pesticides (Fig. 2) and PAHs (Fig. 3). The lowest mean recovery among all time/temperature treatment groups was endrin ketone at 88 % (95 % confidence interval (CI) = 77–98) for the 7 days, 4 °C treatment, and the highest mean recovery was for alpha-BHC at 113 % (95 % CI = 106–119) for the 1.5 days, 35 °C treatment. Mean recoveries and standard deviations for these and other compounds and treatment conditions are given in Supplementary Information Table S1. Average relative standard deviation (RSD) for pesticides was 7.4 %. Average RSD was lower for PAHs at 4.9 %, likely because PAH concentrations were corrected for sample preparation losses while pesticide concentrations were not. No mean recovery was less than mean $t=0$ treatment (one-sided Dunnett's test, all p values < 0.05), and therefore, there was no effect of transport on target compound concentrations for any conditions tested.

The model pesticides and PAHs in this transport study exhibited no decrease in recovery after 14 days of simulated transport conditions in temperatures as high as 35 °C. As the selected model compounds span a range of physicochemical properties, these data suggest that similar compounds would also exhibit no decrease in concentration. Care should be taken in extending the inferences to more extreme conditions, as effects may exist that were not detectable within the given experimental design. The transport stability findings presented here suggest that researchers performing targeted analysis on PAHs and pesticides can do so using more flexible transport conditions. However, if the intended chemical analysis is non-targeted, then expedient transport at or near freezing is a conservative approach to ensure recovery. Huckins et al. (2006) caution that in SPMDs, high-fugacity compounds such as naphthalene can be lost if samplers are not kept under freezing conditions within hours of retrieval. The compounds selected for this study ($\log K_{oa}$ range 7.55–12.0, Table 1) are comparatively less volatile than naphthalene ($\log K_{oa}$ 5.19) (US Environmental Protection Agency 2015). We did not observe any trend between compound volatility and recovery loss, because no recovery loss was observed for any compound in any treatment. If compound loss were to occur under the conditions mimicked in this study, it would be limited to compounds more volatile than the pesticide alpha-BHC, the three-ringed PAH anthracene, or compounds that have lower thermal stability, a chemical characteristic not examined in this study. Biodegradation was also not examined in the present study. LDPE that is deployed in water can develop a

Fig. 2 Mean recoveries of pesticides **a** alachlor, **b** alpha-BHC, **c** chlorpyrifos, and **d** endrin ketone. Concentrations are represented as a percent of control treatment ($t=0$). No recovery is less than control (one-sided Dunnett's test). *Gray area* highlights $\pm 20\%$ of control. *Error bars* represent 95% confidence intervals of the means ($n=8$ for $t=0$ control, and $n=4$ for all other treatments)



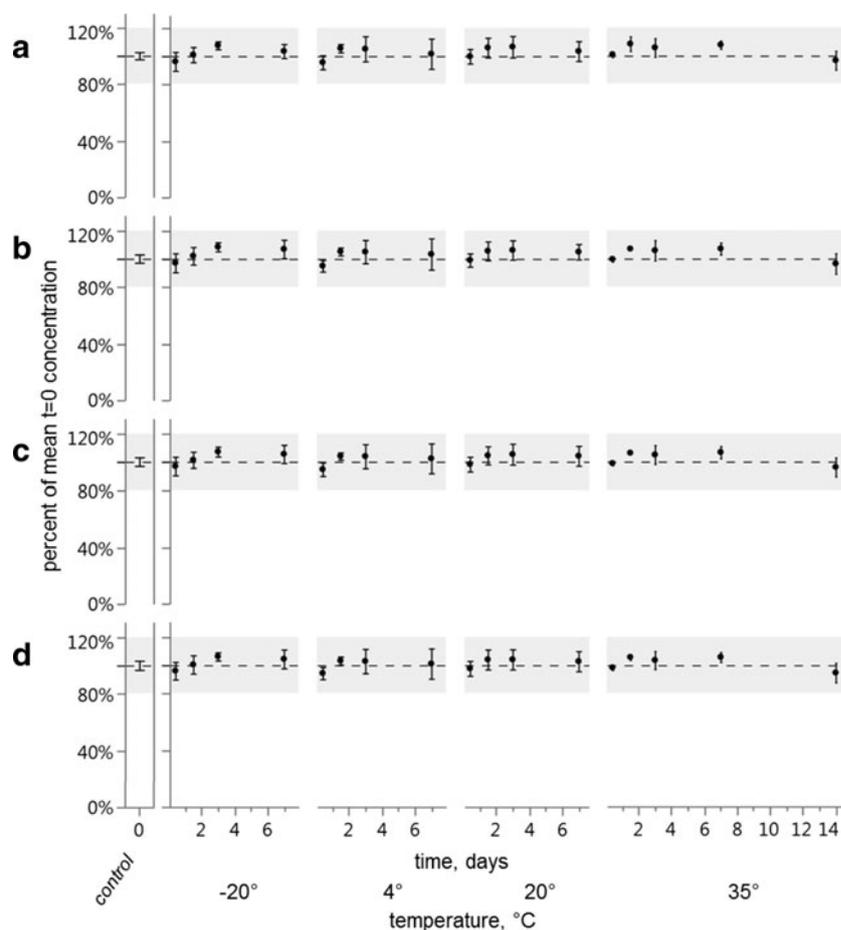
biofilm (Anderson et al. 2014; Huckins et al. 2006) that might favor biodegradation. Passive samplers deployed in air are unlikely to develop a biofilm. Booij et al. (2006) demonstrated that biofouling does not drastically affect target compound uptake while the passive samplers are deployed in water, but biodegradation resulting from biofouling is not well described. Careful selection of PRCs allows researchers to estimate potential effects from biofilms, including biodegradation (Ghosh et al. 2014). During retrieval, the LDPE passive samplers can be cleaned in water from the sampling location to remove biofouling and limit biodegradation during transport. In addition to thermal stability and biodegradation, the effects of more extreme transport durations or temperatures for other classes of semivolatile organic compounds in passive samplers are also worthy of future study.

The LDPE tubing strips selected for this study have an average thickness of 75–95 μm , a thickness that has been used previously in passive sampling techniques (Adams et al. 2007; Anderson et al. 2008; Booij et al. 2002; Rusina et al. 2007). However, LDPE sheets nominally 50 μm (Khairy et al. 2014; Liu et al. 2013; Oen et al. 2011) or 20–30 μm (Alvarez et al. 2014; Fernandez

and Gschwend 2015) are also used. Equilibrium partition coefficients are not affected by LDPE polymer thickness (Lohmann 2012), but it is expected that thinner polymers reach equilibrium faster. We conclude that compounds in the present study reached equilibrium quickly with the small volume of air in the airtight PTFE bag because concentrations did not change across temperature or time. Similarly, we hypothesize concentrations of compounds sequestered in thinner LDPE to also exhibit stability, because equilibrium is expected to be reached quickly.

Accelerated stability tests have been used in chemical standard and pharmaceutical industries as a means to estimate long-term storage stability albeit on a shorter time-scale. In such studies, the storage temperature is increased by at least 20 $^{\circ}\text{C}$ and recoveries are evaluated at standard time intervals (Rueck and Hellriegel 2014). Deviations from acceptable stability in accelerated tests give an early indication of shorter shelf life and inform study design in subsequent long-term studies (Bajaj et al. 2012). Typically, for every 10 $^{\circ}\text{C}$ increase, the rate of degradation doubles (International Conference on Harmonisation of Technical Requirements for Registration of

Fig. 3 Mean recoveries of PAHs **a** anthracene, **b** benzo[ghi]perylene, **c** chrysene, and **d** fluoranthene. Concentrations are represented as a percent of control treatment ($t=0$). No recovery is less than control (one-sided Dunnett's test). Gray area highlights $\pm 20\%$ of control. Error bars represent 95 % confidence intervals of the means ($n=8$ for $t=0$ control, and $n=4$ for all other treatments)



Pharmaceuticals for Human Use 2003; Rueck and Hellriegel 2014). The design of the present study represents accelerated stability tests across a temperature range of 55 °C or the equivalent of about 634 days (14 days $\times 2^{5.5}$). Using the principles of accelerated stability tests, the present study suggests that these compound concentrations are expected to be stable in cold storage for about 2 years.

The compound stabilities tested herein support the use of PTFE bags as a reliable alternative to glass jars, metal canisters, or aluminum foil and plastic bags when transporting LDPE passive samplers. The burden of cost in passive sampling campaigns is in extraction and analysis, while the materials and preparation of an LDPE passive sampler are comparatively inexpensive. In one cost analysis for polychlorinated biphenyl analysis, the US Environmental Protection Agency (2012) reported that an LDPE passive sampler costs only \$5 USD to prepare but costs about \$375 USD for extraction and analysis. The PTFE bags used in the present study cost approximately \$5 USD each. Similarly, pre-cleaned glass jars with PTFE liners cost \$3–8 USD each, depending on the volume. Both PTFE bags and glass jars may be solvent-

cleaned and re-used and therefore have similar costs for repeated uses. The PTFE bags have lower risk of breakage during transport or shipment and cost less to ship because they weigh less. Another transport option is to wrap the passive sampler in aluminum foil and transport on ice, optionally stored in a plastic bag. While this method is more cost-effective than jars or PTFE bags, it does not prevent analytes from partitioning out of the sampling material into or through the plastic bag, if used. As demonstrated in this work, PTFE bags allow for lower cost, chemically inert transport at ambient temperature without increasing material costs.

Passive samplers have been gaining utility in recent decades as a cost-effective means of detecting low concentrations of hydrophobic contaminants in a variety of environments. The present study documents an additional benefit of LDPE passive samplers when studying environmental contaminants represented by the chosen model pesticides and PAHs—that they may be transported in the dark in lightweight PTFE bags at ambient temperature up to 14 days at 35 °C.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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