



National Oceanic
and Atmospheric
Administration

National Marine
Fisheries Service

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MEMORANDUM FOR: Rachel Friedman, F/NWO5
Steven Landino, F/NWO5

FROM: Tracy Collier, F/NWC5,
Ecotoxicology Program Manager

THROUGH: John Stein, F/NWC5,
Division Director

SUBJECT: Submission of 'white paper' concerning relationship between sediment levels of tributyltin (TBT) and adverse effects to salmonid prey species.

Enclosed is our report to your office concerning a technical analysis of the levels of TBT in sediments that are associated with adverse biological effects to salmonid prey species. A previous version of this document was peer reviewed and we have incorporated several changes in response to comments received. Dr. James Meador is the senior author of this report. Please contact any of us with questions you may have concerning the contents of this report.

An analysis in support of a sediment quality threshold for tributyltin to protect prey species for juvenile salmonids listed by the Endangered Species Act.

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Abstract

The National Marine Fisheries Service has authority under the Endangered Species Act to protect listed salmonid species from any adverse actions that may jeopardize the population's ability to recover and increase to sustainable levels. Some listed salmonid species in the Northwest United States are known to travel through urban areas in their migration from river to ocean. Species such as the chinook salmon (*Oncorhynchus tshawytscha*) can spend several weeks in these estuaries during their physiological adjustment from freshwater to marine water. It is during this residence that individuals receive their highest exposure to urban-related contaminants that occur in sediment and the invertebrate prey species they consume. The concern is that these contaminants are bioaccumulated to levels that may directly or indirectly alter the ability of individual salmon to grow and mature normally. This paper provides a framework for determining the tissue and sediment concentrations of tributyltin for protection against severe adverse sublethal effects in invertebrate species, whose decreased abundance would indirectly affect listed salmonid species.

Two approaches for determining adverse sediment concentrations due to tributyltin (TBT) contamination are presented here. The first is the equilibrium partitioning (EqP) approach, which relies on a sediment-water partition coefficient and toxicological data for water exposures. The EqP approach utilizes the large water quality database that has been generated over the last two decades for TBT and provides strong evidence for adverse effects at low exposure concentrations. The second approach involves determination of a tissue residue that is considered harmful for most species, which is then used to predict the sediment concentration that would likely produce this adverse tissue concentration. Both approaches are presented here because they generally support each other. Based on the information presented below, and the inherent difficulty in measuring porewater concentrations, the tissue residue approach is the recommended method for determining adverse sediment concentrations. Using this analysis, the sediment concentration for TBT proposed here is 6,000 ng/g organic carbon. This concentration may ensure adequate

abundance of salmonid prey species, however, it may not be low enough for the protection of sensitive benthic species.

Background

Before its use was restricted, tributyltin (TBT) had been widely used as an antifoulant on ships, nets, piers, buoys, and other nautical devices to retard the growth of fouling organisms. In 1988, the United States Congress passed the Organotin Antifouling Paint Control Act (OAPCA) (U.S. Congress 1988) to limit the use of TBT, making it the only pesticide to be specifically regulated by the U.S. Congress. Water concentrations of TBT dropped dramatically in the years subsequent to OAPCA (Valkirs et al. 1991); however, sediment concentrations have shown only modest declines (Valkirs et al. 1991). Recent surveys show TBT concentrations in sediment over time are relatively constant (Krone et al. 1996, Fent 1996); hence sediments may have become a source, whereas they were once a sink for TBT. Several benthic areas in Puget Sound have been examined and environmental levels of TBT in sediment were found to be in the 100 ppb (ng/g) to low ppm ($\mu\text{g/g}$) range (Krone et al. 1989, Krone et al. 1996, Collier et al. 1998, EVS 1999). (Note: All concentrations reported here are based on TBT ion, not TBTCl or TBT as Sn.)

Equilibrium Partitioning

Equilibrium partitioning (EqP) was developed to explain and predict the partitioning behavior between sediment, water, and tissue for neutral hydrophobic organic compounds (HOCs), such as polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) (McFarland 1984, Di Toro et al. 1991). The basic premise for EqP is that when sediment and water are in equilibrium, the organism receives an equivalent exposure from each phase allowing predictions of the accumulated dose using either phase. The organismal lipid, total organic carbon (TOC) in sediment, and water can be considered as three phases that exhibit predictable concentrations at equilibrium due to equal chemical activity or fugacity. Because of this assumption, the route of exposure (e.g., water ventilation or prey/sediment ingestion) is immaterial because at equilibrium the

concentration in each phase is a function of the thermodynamic properties, not the kinetics of accumulation. EqP has generally been successful in predicting sediment-water partitioning and bioaccumulation for the non-metabolized, neutral HOC compounds, but has not been explored for ionizable organic compounds or organometallics, such as TBT.

Sediment-Water Partitioning

The K_{oc} is the sediment-water partition coefficient that has been normalized to the organic carbon content of sediment. This normalization greatly reduces the variability in the sediment-water partition coefficients observed among different sediment types. K_{oc} values are chemical specific and are related to the compound's affinity for organic carbon. The equation is:

$$K_{oc} = \frac{[sediment]/f_{oc}}{[water]} \quad (1)$$

where [sediment] is the sediment concentration in dry weight, [water] is the observed water concentration, and f_{oc} is the fraction of organic carbon (g/g dry weight). [sediment] and [water] are in the same units (ppb or ppm).

Several studies show the $\log_{10} K_{oc}$ value for TBT to be in the range of 4.2 to 5.0 (Maguire and Tkacz 1985, Unger et al. 1988, Tas 1993, Meador et al. 1997, Poerschmann et al. 1997, Day et al. 1998). Regression analysis of the sediment-water partition data provided by these studies determined the $\log_{10} K_{oc}$ to be 4.5 (= 32,000) and supports the hypothesis that organic carbon in sediment controls the amount of TBT in water (Meador 2000).

It has been shown for many neutral hydrophobic compounds that the octanol-water partition coefficient (K_{ow}) is a good predictor of the K_{oc} . Several authors have developed equations that predict K_{oc} values from the K_{ow} for various hydrophobic compounds (see Meador 2000). These studies show the K_{oc} for HOCs to range from $0.4 \cdot K_{ow}$ to $1.0 \cdot K_{ow}$. The $\log_{10} K_{ow}$ for TBT of 4.4 and the mean $\log_{10} K_{oc}$ of 4.5 are very close and support the utility of these equations as predictors of sediment-water partitioning. Because TBT in marine systems occurs predominantly as the hydroxide, the partitioning behavior of TBT may be similar to that observed for neutral hydrophobic compounds and predictions generated by EqP may be valid.

Tissue-sediment partitioning

Bioaccumulation factors for HOCs are generally expressed as the tissue to sediment concentration ratio (BAF; equation 2) or the biota-sediment accumulation factor (BSAF; equation 3). The BSAF is the lipid and organic carbon normalized bioaccumulation factor:

$$\text{BAF} = \frac{[\textit{tissue}]}{[\textit{sediment}]} \quad (2)$$

$$\text{BSAF} = \left(\frac{[\textit{tissue}]}{f_{\textit{lip}}} \right) / \left(\frac{[\textit{sediment}]}{f_{\textit{oc}}} \right) \quad (3)$$

where [tissue] and [sediment] are in dry weight (ppb or ppm), $f_{\textit{oc}}$ is the dry-weight fraction organic carbon in sediment (g/g), and $f_{\textit{lip}}$ is the dry-weight fraction of lipid in tissue (g/g). The convention is to express all BSAF components as dry weights (Lee et al. 1993). In this paper, all tissue and sediment concentrations are in terms of dry weight, unless noted. Several factors, such as variable uptake and elimination rates, reduced bioavailability, and insufficient time for sediment-water partitioning or tissue steady state can affect bioaccumulation and ultimately the BSAF. For neutral hydrophobic compounds the theoretical maximum BSAF is unity (Di Toro et al. 1991) and the empirical maximum values generally range from 2 to 4 (USEPA/USACE 1991, Boese et al. 1995) for HOCs that are not metabolized. Because of the amounts of chemical expected in lipid and organic carbon, it is generally believed that HOCs will produce predictable levels of bioaccumulation.

Tributyltin is an ionizable, polar organometallic compound that does not bioaccumulate in organisms according to the EqP approach, which predicts one BSAF value for all species that do not metabolize the compound of interest. A review of the available data (Meador 2000) indicates that species-specific toxicokinetics can predict the bioaccumulation of TBT, not thermodynamic partitioning between tissue and environmental concentrations (e.g., water or sediment). For many species, the bioconcentration factor (BCF) or biota-sediment bioaccumulation factor (BSAF) is far above that predicted based on the expected thermodynamic maximum, which is determined by the lipid content in tissue. Therefore, because lipid does not appear to control TBT bioaccumulation, it is not needed for making predictions. However, because the organic-carbon normalized sediment

concentration (sed_{oc}) is related to the amount of TBT bioaccumulated and the BSAF equation is convenient for expressing species-specific bioaccumulation, the equation can be used in conjunction with an average lipid content for each species. Alternatively, a bioaccumulation factor with only the sediment component normalized to organic carbon (BAF_{oc}) may be used to describe TBT bioaccumulation and would be more appropriate for those species where lipid content is highly variable:

$$BAF_{oc} = \frac{[tissue]}{[sediment]/f_{oc}} \quad (4)$$

It should be noted that this equation will produce smaller values than those obtained with the BAF or BSAF equation. The following equation can be used for converting the BSAF to the equivalent BAF_{oc} :

$$BAF_{oc} = BSAF * f_{lip} \quad (5)$$

There are very few studies on TBT that report BSAF values for invertebrates; however, a few have reported BAF values. For invertebrates, the BAFs from studies of field-contaminated sediments range from 3 to 100 (Langston et al. 1987, Bryan et al. 1989, King et al. 1989, Bryan and Gibbs 1991, Langston and Burt 1991, Kure and Depledge 1994, Stäb et al. 1996). The mean BAFs in Langston et al. (1987) varied from 3 for *Nereis diversicolor* to 78 for *Mya arenaria*. Because of the low lipid content (0.7 – 1 % wet wt.) reported for these species, their BSAFs could be relatively high. For example, if the sediment TOC content was 2.0%, the dry-weight BSAF for *M. arenaria* would be approximately 40. A field study of TBT concentrations in a freshwater lake reported BSAFs for several invertebrate species in the range of 3 to 10 and even higher for some fish (Stäb et al. 1996). Meador et al. (1997) determined BSAF values for 3 marine invertebrates ranging from 0.4 to 4.6 in laboratory exposures, which were expected to be even higher when steady state between tissue and sediment occurred. For these species the BAF_{oc} values ranged from 0.02 for a tolerant species to 0.5 for one that was sensitive to TBT exposure. Relatively high bioaccumulation values for TBT are generally characteristic of species with a high rate of uptake or a low rate of metabolic conversion and elimination. Based on this information, it was concluded that TBT bioaccumulation factors for benthic organisms are generally high compared to other compounds.

TBT Toxicity in Aquatic Organisms

Exposure-based toxicity

Several studies demonstrate that TBT is very toxic to marine invertebrates (Maguire 1987, Cardwell and Meador 1989, Heard et al. 1989, Fent 1996) and because of its persistence in sediment, there is still a concern about its impact on organisms. It is known that marine species exhibit a range in responses when exposed to TBT in water (Cardwell and Meador 1989, Meador 1997, Fent 1996); however, their response to sediment-associated TBT has not been fully studied. Mortality responses for TBT in water exposures have been reported from approximately 0.5 ng/ml to over 200 ng/ml, a range of about 400 fold (Cardwell and Meador 1989). Most of this variability is due to differences in the uptake and elimination kinetics between species; however some of these values underestimate the toxic response because of insufficient time for exposure. For example it takes approximately 75 days for tissue concentrations to reach steady state in *Eohaustorius washingtonianus*, an amphipod with a slow rate of elimination (Meador 1997). Consequently, many of the reported values underestimate the true response because of presteady-state conditions (Meador 2000).

Sublethal responses to water exposure are also highly variable ranging from the low (0.01 ng/ml) to high (0.5 ng/ml) parts per trillion (Cardwell and Meador 1989, Fent 1996). The most common sublethal endpoints measured are growth inhibition, shell chambering in oysters, histological and behavioral abnormalities, and imposex in prosobranch gastropods. In terms of exposure concentrations and sublethal effects, it is clear that molluscs are the most sensitive taxon to TBT, primarily due to their weak ability to metabolize this compound and their high rate of uptake. It should be noted that many of the sublethal responses noted for TBT exposure would eventually lead to death of the organism in the environment.

The data for adverse effects due to sediment exposure are much less abundant; however, a few studies indicate that sediment concentrations in the 100 to 1000 ng/g range can have severe effects. For example Fent and Hunn (1995) noted that clams had disappeared in areas where sediment TBT exceeded 800 ng/g dry weight and Meador and Rice (20010) noted moderate to severe reductions in growth for the polychaete *Armandia*

brevis for sediment concentrations in this range (100 - 1000 ng/g dry wt.) Bryan and Langston (1992) and Langston and Burt (1991) also suggested that some populations of bivalves (*Macoma balthica* and *Scrobicularia plana*) have disappeared in locations with TBT sediment concentrations over 700 ng/g dry wt.

TBT toxicity based on tissue-residues

The critical body residue (CBR) method predicts toxicity in terms of tissue concentrations (McCarty 1991). Several studies have found that when toxicity is expressed as a tissue residue, the variability between species, time periods, and exposure conditions is greatly reduced (McCarty 1991, van Wezel and Opperhuizen 1995, Meador 1997). The observed data indicate that for a specific toxicant, or class of toxicants, there is a narrow range in tissue concentrations across species that is associated with a given mode of action. Several chemical classes representing different modes of action, including narcotics, respiratory uncouplers, acetylcholinesterase inhibitors, dioxin, and others have been examined and the tissue concentrations for effects have been defined (McCarty and Mackay 1993). Sublethal effects, such as growth or reproductive impairment can occur at tissue concentrations that are from 10 to 100 times lower than those for lethality, with a typical acute to chronic ratio of 10 (McCarty and Mackay 1993). While there is some variability in the tissue residue effect number for a given mode of action, a large percentage of this can be due to variable lipid content found in organisms (van Wezel et al. 1995).

Acute TBT toxicity is likely caused by uncoupling of oxidative phosphorylation. Several studies have examined the lethal response for TBT and report that mortality occurs at a relatively constant whole-body tissue residue of 30 – 115 µg/g dry wt. (Meador 2000). Based on these studies, the mean LR50 (lethal residue affecting 50% of the population) for 11 species (including fish) exposed to TBT was determined to be approximately 48 µg/g dry wt. (Meador 2000).

The main sublethal responses reported for TBT include growth inhibition and imposex in molluscs. These organismal responses to TBT are a manifestation of one or several modes of action and they are generally associated with tissue concentrations that occur at 10 to 100 times lower than those reported for the lethal response, which is in

agreement with the data of McCarty and Mackay (1993). A recent review article (Meador 2000) demonstrated a relatively consistent tissue concentration (lowest observed effect residue; LOER) associated with reduced growth (3 µg/g dry wt.) and reproductive impairment (0.5 µg/g dry wt.) for several species.

Predicting Adverse Sediment Concentrations

Because there is a general lack of sediment quality criteria or guidelines for tributyltin, there is a need to be able to characterize the potential to cause ecologically significant effects to organisms living in proximity to contaminated sediment. Approaches for determining such sediment quality guidelines should include components of the EqP, toxicokinetic, and critical body residue approaches because of their utility in predicting bioaccumulation and biological effects. The goal is to protect the sensitive species against sublethal effects, such as altered growth or impaired reproduction, which may affect population dynamics and their ability to thrive.

Equilibrium partitioning (EqP) approach

One of the main advantages of EqP is to utilize the very large water-quality database developed over the years and consider biological responses in terms of the concentration of a contaminant found in sediment (Di Toro et al. 1991). Because toxicity of TBT in sediment is not as well studied as toxicity caused from exposure to TBT contaminated water, the principles of EqP can be applied to determine equivalent sediment concentrations for regulatory purposes.

Water quality data can be converted to sediment concentrations with the following equation:

$$[\textit{sediment}] = K_{oc} * f_{oc} * [\textit{water}] \quad (6)$$

[sediment] and [water] are concentrations associated with biological responses (e.g., lowest observed effect concentration; LOEC, or a water quality criteria), K_{oc} is the organic-carbon normalized sediment-water partition coefficient and f_{oc} is the fraction of organic carbon in sediment (= %TOC/100). Because of the large variability found among species in their

response to environmental concentrations of TBT, only sensitive species with high bioconcentration or bioaccumulation factors should be considered when determining the effect concentration.

Tissue residue approach

An alternate method to the one above utilizing water concentrations for generating sediment quality guidelines would be based on tissue concentrations. A tissue residue deemed to be protective for most species (e.g., LOER or NOER; lowest or no observed effects tissue residue), that is determined from well-controlled laboratory studies, would be converted to a sediment concentration by utilizing the BAF_{oc} value for an appropriate and sensitive test species. The following equation can be used to determine the organic-carbon normalized sediment concentration (sed_{oc}) for use in regulating exposure to TBT in sediment:

$$[sed_{oc}] = \frac{[tissue]}{BAF_{oc}} \quad (7)$$

where [tissue] is the tissue residue used for protection (LOER or NOER) and sed_{oc} is the organic-carbon normalized sediment concentration. As stated earlier, the BSAF is a convenient bioaccumulation factor, even though organismal lipid content does not affect TBT bioaccumulation. A BSAF value could be used to determine the $[sed_{oc}]$ because lipid content cancels out and is not needed; however, use of this equation would require a relatively constant lipid content.

$$[sed_{oc}] = \frac{[tissue]}{BSAF * f_{lip}} \quad (8)$$

where f_{lip} is the average lipid content for this species.

Even though the effect concentration is based on tissue residues rather than environmental exposure, this CBR needs to be related to an environmental concentration for regulatory purposes. Because the organic carbon content of sediment is correlated to the amount of TBT accumulated by a species, the BAF_{oc} can be used to relate tissue concentrations to that found in sediment.

Because of differences in ability to bioaccumulate TBT from sediment, the sediment quality value would have to be determined with sensitive test species (based on exposure concentration) for protection of most species that would inhabit the impacted site. In other words, only species that exhibit a high steady-state BAF_{oc} should be selected when using equation 7 to determine the sediment concentration that is to be considered harmful. Assuming steady state for sediment-water-tissue partitioning and a consistent tissue-residue effect concentration, a sensitive species with a higher BAF or bioconcentration factor (BCF), will respond to a much lower sediment concentration. For example, *Eohaustorius washingtonianus*, which has a high uptake clearance (k_1) and slow rate of elimination (k_2) (Meador et al. 1997) exhibits a high steady-state BSAF (12) ($BAF_{oc} = 0.50$) and is affected by low sediment concentrations of TBT ($LC50 = 260$ ng/g sediment) (Meador 2000).

Analysis

This analysis focused on salmonid prey species, specifically benthic invertebrates. Impacts on salmonids are expected to occur at higher TBT sediment concentrations than those for invertebrates, primarily due to their enhanced metabolism of this compound, reduced exposure to sediment, and the general lack of biomagnification from one trophic level to the next. Because very little research has been conducted on the response of fish to TBT, the above assumptions may be altered in light of new information.

EqP approach

The EqP approach was used only to predict water concentrations from the sediment-water partitioning characteristics of TBT and relate those concentrations to controlled laboratory toxicity experiments based on water exposure. As noted earlier, the EqP approach is useful for predicting sediment-water partitioning for TBT and the amount available for uptake, but not the amount bioaccumulated and that expected at steady state.

A compilation of all available data (51 studies) on TBT determined the median water concentration for chronic toxicity to be 0.22 ng/ml; the 5th percentile concentration was 0.02 ng/ml (Weston 1996). Most of the results for these 51 studies are LOECs based on growth impairment. Using the K_{oc} value of 32,000 determined in Meador (2000), the median water concentration of 0.22 ng/ml would be expected from a sediment concentration (normalized to organic carbon) of 7,040 ng TBT/g organic carbon. For a TOC of 2%, this would equal a concentration of 141 ng/g sediment dry weight. Because this value is derived from the median concentration for all sublethal effect studies, some adverse responses would be expected for this level of exposure.

Several areas in Puget Sound have TBT sediment concentrations that exceed this value. In a recent study of TBT around the Harbor Island Superfund site (EVS 1999), 95% of the sediment samples exceeded the 7,040 ng/g OC value. Also, 60% of the stations sampled produced porewater concentrations above the median water concentration (0.22 ng/ml) associated with all sublethal effects. These concentrations were measured in an estuary where listed chinook salmon transition from freshwater to marine water. Other studies

(Krone et al. 1989, 1996) also indicate that tributyltin concentrations in Puget Sound sediments routinely exceed 7,040 ng/g OC.

The median concentration of tributyltin (TBT) found in the Hylebos Waterway, another Superfund site in Puget Sound where listed salmonids occur, is 329 ng TBT/g dry wt. (Collier et al. 1998). This concentration is in the range needed to produce a lethal response for a sensitive invertebrate such as the amphipod *Eohaustorius washingtonianus* (Meador et al. 1997). Based on the K_{oc} (32,000), this sediment concentration (329 ng/g dry weight) at 2% TOC would produce an interstitial water concentration of 0.51 ng/ml, which is not far below that needed to produce a lethal response in juvenile starry flounder (Meador 1997). (The LC10, or concentration expected to cause mortality in 10% of individuals is 1.5 ng/ml). The starry flounder spends most of its time on the sediment surface and is likely to ventilate water that is high in TBT, in addition to that accumulated by ingestion of prey and sediment. Because sublethal effects are expected to occur at concentrations at least 10 times lower than those causing mortality, these water concentrations may produce a sublethal response in this important flatfish.

It should be noted that the proposed U.S. EPA water quality criterion for TBT is 0.01 ng/ml, which was generated to protect at least 95% of the species from chronic (long term) effects. Chambering in oysters and imposex in snails were the main responses that were responsible for this low value.

Tissue residue approach

If the tissue residue approach is adopted, a tissue concentration of 3 µg/g (dry wt.) is recommended as the level for adverse sublethal effects in salmonid prey. This tissue concentration was recently selected as the “tissue trigger level” for remediation of sediments at the Harbor Island Superfund site (U.S. EPA 1999) and it has been correlated with reduced growth in at least 6 different species (Meador 2000). The tissue residue associated with reproductive impairment (0.5 µg/g dry wt.) was not selected because most of the studies used to derive this value were on stenoglossan gastropods, which are not usually prey for juvenile salmonids.

This tissue concentration (3 µg/g dry wt.) is approximately 15 times less than the lethal tissue concentration (Meador 2000), which is a factor that is almost identical to the computed ratio of acute (lethal) effects to chronic (generally sublethal) effects for water exposures published in a recent risk assessment for TBT (Cardwell et al. 1999). As stated above, most invertebrates exhibit BSAFs for TBT in the range of 2 to 10 or higher. For example, even a species such as *Armandia brevis*, which is not considered highly sensitive to TBT, exhibits a steady-state BSAF of 4.2 ($BAF_{oc} = 0.2$) (Meador et al. 1997). For determination of the threshold sediment concentration, a BAF_{oc} of 0.5 was selected as representative for sensitive species. This is comparable to a BSAF of 10 for a sensitive invertebrate with a lipid content of 5% dry wt., which is a reasonable average lipid content for invertebrates (Boese and Lee 1992). Based on equation 7 or 8, using the appropriate bioaccumulation factor, the resulting sed_{oc} is 6,000 ng/g organic carbon. For a sediment with 2% TOC, this would equal 120 ng TBT/g sediment dry weight.

Summary

Based on the tissue residue approach (recommended) and the available data, protection against severe adverse sublethal effects for many, but not all salmonid prey species, should be achieved with a TBT sediment concentration of **6,000 ng/g OC**. For a sediment with 2% TOC, this would equal **120 ng/g dry weight**. This value is based on a tissue residue approach using a LOER of 3 µg/g dry weight and a $BAF_{oc} = 0.5$ ($BSAF = 10$). In general, the EqP and tissue residue approaches support each other, indicating adverse effects in benthic species at relatively low exposure concentrations. At this sediment concentration, no adverse effects on migrating salmon are expected; however, if substantial tissue residues are detected (e.g., > 500 ng/g dry wt.) in juvenile salmon, then these recommendations should be reconsidered. The goal here is to protect salmonid prey against severe effects; however, at this sediment concentration some sublethal effects on benthic invertebrates, especially molluscs, are expected. If the intent was to protect all benthic species against chronic effects, a value approximately ten times lower would be more appropriate.

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