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A HISTORICAL RECORD OF MERCURY CONTAMINATION IN SOUTHERN FLORIDA (USA) AS INFERRED FROM AVIAN FEATHER TISSUE

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Abstract—During the late 1980s, the upper trophic-level biota of the Everglades (FL, USA) was recognized as being highly contaminated with mercury (Hg). However, the timing and pattern of that increase is poorly known, and no information is available about mercury contamination in Everglades wildlife prior to 1974. We measured methylmercury concentrations in feathers of white ibises (n = 33), great egrets (n = 7), anhingas (n = 21), and great blue herons (n = 12) from museum specimens collected from 1910 through 1980 and combined them with more recent feather samples collected from live birds (1985–2000, n = 98, 37, 49, and 7, respectively). We found no evidence of contamination of museum samples with inorganic mercuric preservatives (0.01–0.28% of total Hg in feathers). All species showed relatively low concentrations of mercury through the 1970s (<5 μl/L dry wt for anhingas, ibises, and egrets, <10 μl/L for herons). Samples from all species taken during the 1990s showed a large and significant increase (4–5×) in MeHg concentration. This evidence suggests that most of the increase in Hg deposition during the 20th century in south Florida occurred during the last two to three decades, which is consistent with information about local source deposition. Contamination levels prior to the 1970s appear to have been associated with normal reproduction in these birds, suggesting partial evidence for a threshold of reproductive impairment.

Keywords—Mercury Wading bird Everglades Contamination Anhinga

INTRODUCTION

Mercury (Hg) and methylmercury (MeHg) have been documented as contaminants of major importance to wildlife and humans in many aquatic ecosystems [1], such as the Great Lakes [2], the Brazilian Pantanal [3], and the Florida Everglades [4]. In aquatic systems, mercury is likely to impact biota because of high bioaccumulation potential, high bioavailability, efficient recycling of Hg, and high potential for methylation [5–7]. The objective of this paper is to document a long-term history of mercury exposure in the Everglades, using museum feather specimens as a tool.

The Everglades of southern Florida is an ecosystem in which the biota had become highly contaminated with Hg by the 1990s, and a number of detrimental effects on biota and potential threats to human health have been documented [4,8–11]. Between 1994 and 1998, however, a decrease of >74% in total Hg was detected using feathers of nestling great egrets (*Ardea alba*) collected annually in a standardized fashion [12]; similar trends have been found using largemouth bass (*Micropterus salmoides*) fillets [13].

An important source of Hg deposition in the Everglades during the 1980s and 1990s has been aerosolized effluent from local municipal waste incineration [14]. The decrease in Hg in fishes and birds during the 1990s has been hypothesized to be in large part the delayed result of decreased mercury emissions from local municipal incineration, initiated in the early 1990s [15].

Prior to the late 1980s, the levels and dynamics of Hg in south Florida are much less well understood. Studies of soil cores suggest that deposition in the south Florida area has increased by roughly 4.9 times during the 20th century and that local deposition has roughly tracked global patterns [16]. However, it is difficult to closely infer the timing of increases in Hg in Everglades soil because of a high state of flux of near-surface soils caused by frequent wetting, drying, burning, oxidation, and deposition and because of differences in bulk density among sites. So while the soil core information is probably reliable as an indication of the magnitude of change over a period of many decades, the resolution within that period is necessarily poor [17]. It is also currently impossible to translate soil mercury concentrations into estimates of contamination in water or wildlife, which makes time-series comparisons difficult. The only available information about wildlife contamination prior to the 1990s was reported in the mid-1970s [18], at which point hepatic and egg tissues of wading birds were not considered elevated.

The record of mercury contamination in the Everglades is thus limited to the inference that mercury has increased in soils by approximately 4.5 times during the 20th century, that contamination levels in wildlife had reached detrimental levels by the 1990s, and that a large reduction in exposure occurred between the early and late 1990s. The pattern of contamination during the rest of the 20th century remains unknown.

The timing and pattern of mercury contamination in wildlife could have ramifications for understanding fluctuations in Everglades wildlife populations and for determining the timing and potential sources of mercury contamination. Knowledge of the historical pattern may also help to establish thresholds for effects of Hg or MeHg on wildlife in wetland systems. The establishment of such benchmarks for wildlife has been ham-

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pered by several persistent problems, including multiple endpoints [19], wide differences in species-specific sensitivities [6], and the inability to include natural stressors when measuring effect levels [20]. In this context, the establishment of Hg contamination levels during a period when wildlife populations were known to be healthy may be an important source of information for risk assessors.

Following a catastrophic population decline due to plume hunting (1860–1910), Everglades wading bird populations had rebounded by the 1930s to annual breeding populations of 100,000 to 200,000 birds [21]. At some point between the late 1940s and the late 1970s, wading bird populations as a whole then declined by 75 to 90% [21,22]. Thus, the period from 1930 through the late 1940s seems to be generally recognized as a time when breeding populations in the Everglades were not constrained by hunting, contaminants, or human manipulations to the environment.

Bird feathers are excellent indicators of mercury contamination in birds [23,24], and this process is particularly well documented for piscivorous birds [12,23–28]. Historical reconstructions of Hg contamination have been successfully inferred from retrospective analyses of bird feathers in museum collections [26,27]. In this study, we examined historical levels of Hg in piscivorous birds by measuring Hg in feather tissues of museum skins collected in south Florida during the 20th century to test the null hypothesis that mercury exposure has remained constant during this time. We also examine the hypothesis that feathers from museums are typically contaminated with mercuric preservatives.

MATERIALS AND METHODS

Study species

We used great egrets, white ibises (*Eudocimus albus*), great blue herons (*Ardea herodias*), and anhingas (*Anhinga anhinga*) as study species because these four species are typical and representative of the Everglades ecosystem, are common in museum collections, and represent a range of aquatic trophic levels. In addition, feather mercury concentrations exist for these species from the period 1990–2000 [8,10,29; data presented here]. White ibises typically prey on crustaceans, insects, and occasionally small fishes. Great egrets eat almost exclusively small to medium-size fishes [29,30], while great blue herons and anhingas take larger fishes, such as centrarchids [31,32]. Anhingas were also selected because they are found almost exclusively in freshwater marshes and are likely to be resident rather than migratory [31].

Collection of feather samples

We collected feather samples from adult birds only using a variety of techniques. All feather samples from birds representing any period prior to 1990 were collected from museum specimens (see the following discussion). Samples representing birds after 1990 were collected directly from live birds or fresh/frozen carcasses. In the case of white ibises, post-1990 feathers were collected from the scapular region of adult birds live-trapped in the Water Conservation Areas (WCAs) of the central freshwater Everglades (Dade and Broward counties) during January to June of 1998–2001 [28,33]. Post-1990 feather samples of anhingas were also collected from the scapular region of live adults trapped on or near their nests in WCA 2 and 3 during April–May 2002. Post-1990 samples of great blue herons and great egrets were collected from the scapular region of animals that were road-killed, from animals that were brought to rehabilitation centers, or from directly underneath active nests (shed feathers).

We aged birds based on plumage and/or reproductive status. For white ibises, great blue herons, and anhingas, this could be done by plumage alone since juvenile plumage is conspicuous. For great egrets, the presence of breeding plumes was taken as evidence of breeding status. Beyond the age of first breeding (adult status), we did not attempt to age field-collected or museum specimens.

Selection of museum specimens for sampling

We searched the available databases of all major ornithological museum collections in the United States and those of minor collections in Florida for records of specimens of the study species. Specimens had to be identified by date of collection to month and location at least to county or recognizable landmark. The information on specimen tag and database or catalog record had to be consistent. The specimens had to have been collected from the freshwater areas of the Everglades because the recent feather collections were from freshwater areas and because the dynamics of mercury cycling and exposure in wading birds are likely to differ consistently between fresh and estuarine areas [34]. Consequently, we rejected records of ibises and herons unless they indicated that specimens were collected in a clearly noncoastal location. For example, western Palm Beach County was accepted, while Palm Beach County was not, and Royal Palm Hammock, a specific freshwater location, was accepted, while Everglades National Park was not. Unlike herons and ibises, anhingas are limited by habitat to freshwater areas, and we therefore accepted all records of Anhingas from Dade, Broward, western Monroe, eastern Collier, and Palm Beach counties. We were also careful of changes in county boundaries during the 20th century that may have been misleading about the collection location.

Collection of museum-stored feather tissues

We collected contour feathers from the breast of specimens, as mandated by museum policies. Individual feathers were isolated and pulled with clean, acid-rinsed, stainless-steel forceps. We collected approximately 0.2 g of feather tissue from each specimen and placed the feathers in individually labeled paper envelopes using forceps. Feather samples were cataloged and stored at room temperature until analysis.

Mercury determination

Feather samples from each museum specimen were analyzed individually at the Florida Department of Environmental Protection Chemistry Section 1 seven months after collection. Since mercuric preservatives were historically used in the preparation of museum skins, we examined the possibility that feathers from museums had become contaminated with inorganic mercury. We first rinsed the museum feathers with 0.1 N hydrochloric acid and collected the acid rinsate. The acid rinsate was analyzed for total mercury using U.S. Environmental Protection Agency Method 245.1 [35]. The aqueous samples were digested using potassium permanganate and persulfate and analyzed by an atomic absorption spectrometer (Varian 220 AA, Mulgrave, Australia) using cold vapor atomic absorption detection (detection limit = 0.1 mg/L).

Following acid rinse, the feathers were analyzed for methyl mercury. The feathers were first rinsed thoroughly with deionized water to remove acid residues and air dried overnight

Table 1. Concentrations (μl/L dry-wt basis) of total Hg (THg) in acid rinsate and methylmercury in digested feather tissue of museum-collected feathers of wading birds from the Florida Everglades (FL, USA). The THg in rinsate is also represented as a proportion of all mercury in the feather sample. SD = standard deviation

	Mean feather Hg (μl/L)	SD total Hg	Mean THg in rinsate (μl/L)	SD THg in rinsate	Mean proportion THg in rinsate
Great blue heron	3.345	3.202	0.0084	0.0058	0.0025
White ibis	1.042	0.761	0.0029	0.0021	0.0028
Great egret	2.773	3.374	0.0038	0.0019	0.0014
Anhinga	1.862	2.652	0.0018	0.0010	0.0010

on a lint-free towel. The dried feathers were then frozen in liquid nitrogen and ground to a powder using a mortar and pestle. The powdered feathers were first digested/denatured at 90°C in 10 ml of a solution of 25% potassium hydroxide in methanol for 1 to 3 h until all the powder had dissolved. A 50-μl aliquot of the digestate was then added to 100 ml of deionized water and ethylated using sodium tetraethylborate in a zero-headspace volatile organic compound vial. The ethylated product, methylethyl mercury, was purged from solution using a Tekmar 3000 (Mason, OH, USA) purge and trap and trapped in a Tenex trap (Mason, OH, USA). The methylethyl mercury was then desorbed from the trap and separated in a gas chromatograph (HP 5890, 15-m DB-5 column, 0.32-mm i.d.; Hewlett-Packard, Palo Alto, CA, USA) using helium as the carrier gas. The separated volatile (methylethyl mercury) was pyrolyzed in an alumina tube furnace maintained at 850°C to atomic mercury as it exited the gas chromatograph column, mixed with Argon makeup gas, and detected using a Tekran 2500 atomic fluorescence detector (Toronto, ON, Canada). The weights of the feathers ranged from 0.02 g to 0.05 g dry weight. The detection limit for this method is 20 ng/kg (nl/L).

Comparison of feather types

The contour feathers we collected from museum specimens were from a different area (abdomen) than the recent samples (scapular). To determine the effect of anatomical location of the feather sample on mercury concentration, we collected feathers of both types from living adult birds in 2002 (nine white ibises, three great egrets, four anhingas, and one great blue heron, all from the south Florida area). The feathers were analyzed separately by feather type and individual for total Hg using the methods described previously and the results compared by feather type.

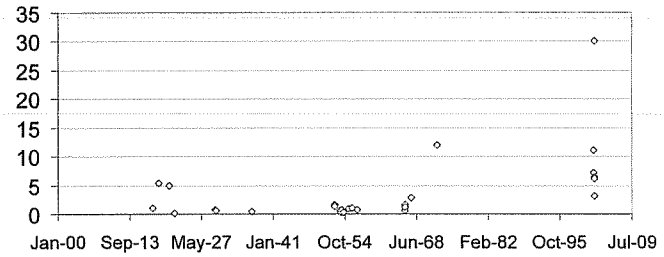

Fig. 1. Mercury concentrations (mg/kg dry wt) in feathers of individual anhinga specimens collected from the Everglades (FL, USA) during the 20th century.

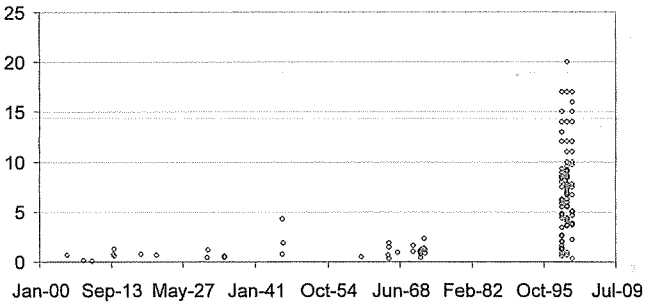

Fig. 2. Mercury concentrations (mg/kg dry wt) in feathers of individual white ibis specimens collected from the Everglades (FL, USA) during the 20th century.

Statistical treatment

Since sample sizes were generally small (<10) and variable across species, we used nonparametric Mann–Whitney *U* tests to test the null hypotheses that mean mercury concentrations in feathers were unaffected by location of feather on the body and that mean feather concentrations were similar in pre-1980 and post-1980 samples.

RESULTS

We found very little evidence of contamination of museum specimens with inorganic mercuric preservatives. The acid rinsate of museum feathers contained 0.0018 to 0.0084 μl/L total Hg (THg, range of species means), representing between 0.10 and 0.28% of the total mercury in the feather samples (Table 1).

Of the 17 birds for which we had both abdominal and scapular feathers, in seven cases abdominal feathers showed higher concentrations of total Hg, and in 10 cases the reverse was true. When comparing these two types of cases, we found no significant differences in the means of the individual differences (e.g., abdominal–scapular) in feather concentration (Mann–Whitney $U = 1.004$, $p = 0.49$). This suggests that although the two types of feathers did not always have equal concentrations of mercury in the same individuals, the direction of differences in concentration between the two feather types was not consistent across individuals. Further, the magnitude of differences within individuals seemed small by comparison with the larger temporal differences in the data set (see Figs. 1–4).

Although the mean MeHg concentrations in feathers were different for the four species (great blue heron > great egret > anhinga > white ibis), the pattern of Hg contamination over time during the 20th century was similar among species (Figs. 1–4). All species showed relatively little variation in concen-

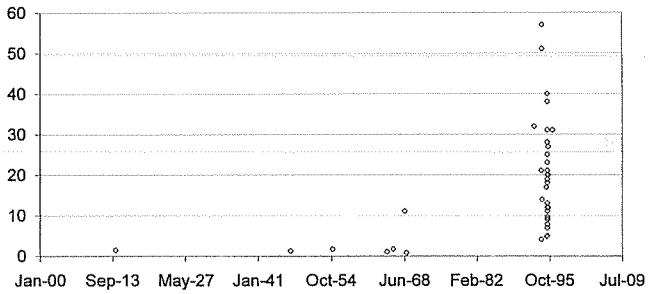

Fig. 3. Mercury concentrations (mg/kg dry wt) in feathers of individual great egret specimens collected from the Everglades (FL, USA) during the 20th century.

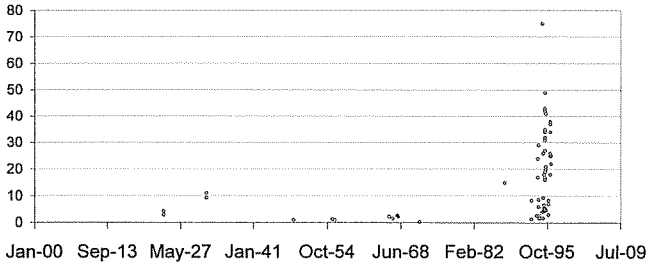

Fig. 4. Mercury concentrations (mg/kg dry wt) in feathers of individual great blue heron specimens collected from the Everglades (FL, USA) during the 20th century.

tration through the late 1970s and a marked (five- to sevenfold) increase in Hg sometime between the late 1970s and the decade of the 1990s. The almost complete lack of specimens during the late 1970s to 1990 appeared to be consistent across museums and probably reflected similar shifts in priorities of curators and collectors. For all four species examined, the mean Hg concentration during the 1990s was significantly higher than the mean of pre-1980 samples (Table 2).

DISCUSSION

We found that mercury concentrations in feathers of aquatic birds from south Florida were consistently low across four species until rapid increases during some point after the late 1970s. Since the findings are similar across species and the differences robust, we interpret this finding as being indicative of the dynamics of mercury exposure to wildlife during this period.

We were not able to include sex, season of collection, or age of specimen (beyond breeding age) as covariates in our analyses. We are unaware of any process that would result in a consistent sex bias in collection of either field-caught or museum specimens. The vast majority of both pre- and post-1980s specimens were collected during the breeding season (December–June, 87 and 97%, respectively), when birds should have been in breeding plumage. Thus, it seems likely that molt status was unlikely to explain the variation seen in mercury concentrations between the two samples. We were unable to evaluate the hypothesis that age affected mercury concentration in these birds.

Table 2. Mercury concentrations (μl/L) in feather samples of piscivorous birds from the Everglades (FL, USA), comparing the same species during two eras: pre-1980s and post-1999. SD = standard deviation

	Anhinga	Great egret	White ibis	Great blue heron
Pre-1980s				
Mean	1.86	2.77	1.04	3.34
SD	2.72	3.64	0.77	3.34
Median	0.87	1.50	0.86	2.35
n	21	7	33	12
Post-1990				
Mean	10.03	19.84	7.47	21.03
SD	9.11	12.45	4.58	15.98
Median	6.50	18.00	7.10	19.00
n	7	37	98	49
Z^a	3.475	3.834	7.508	4.264
p	0.00007	0.00001	0.00001	0.00001

[a] Mann-Whitney *U* test.

We also do not believe that museum storage was likely a source of variation in our samples. The extremely small amounts of THg we found in the acid rinsate from feathers suggests that very little exogenous inorganic Hg (not bound to feather tissue and not originating from the live animal) had become associated with the surface of the feathers. This indicates that the museum specimens came in contact with only small amounts of exogenous Hg during preparation or storage. The possibility of incorporation of inorganic Hg into feathers seems small since nearly all Hg in feathers of museum specimens has been found to be in the methylated form [23,36,37]. Since only inorganic mercury compounds were used by preparers of skins and since methylation is extremely unlikely under museum storage conditions, the only exogenous contamination source would seem to be inorganic mercury. The low variance in inorganic Hg in feathers and the large sample of individual specimens (73), species (four), years (31), and museums (six) suggest that contamination with mercuric preservatives may be an infrequent problem in assaying museum feather samples for mercury.

The pattern of increase in feather mercury across the 20th century in piscivorous birds from south Florida was consistent across four species. The fact that the pattern for anhingas was similar to the other species is also of particular interest. While the other study species may be migratory [30,32] or nomadic [38], anhingas are largely sedentary, especially in the Everglades, where temperatures are appropriate year-round for the peculiar thermoregulatory needs of this species [31]. In addition, the anhingas are almost completely freshwater in distribution, while the herons and ibises may be found in coastal areas. The similarity of the anhinga contamination pattern to the other species suggests that the contamination signal from the heron/egret/ibis samples was representative of the south Florida environment and had not become overwhelmingly confounded through migration/movements or coastal habitat use. The species-specific concentrations we found were expected given the relative trophic levels of the birds, with great blue herons > great egrets > anhingas > ibises.

The timing of increases in mercury contamination is also of interest. For all four species examined, the levels of contamination appear to have been consistent for much of the 20th century. For samples of ibises, anhingas, and great egrets prior to the 1980s, feather mercury remained below 5 μl/L MeHg dry weight with only one exception, and ibises remained below 3 μl/L with one exception. For great blue herons, all but one of the samples were below 10 μl/L. The fact that we had so little similarity between the concentrations pre- and post-1980s suggests that the two periods were fundamentally different in contamination risk.

The consistency of concentrations in the pre-1980s period for all species also suggests that for most of the 20th century, mercury contamination remained at relatively stable levels, and we find no evidence of a monotonic increase that would imply increasing atmospheric deposition. Thus, while the amount of increase in Hg in feathers (five to seven times) during the 20th century is similar to the order of magnitude implied by global deposition [39,40] and by Everglades soil samples (4.9 times), the feather data suggests that most of the increase in the Everglades has happened during the last 20 years of the 20th century, primarily as a result of local, not global, deposition.

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