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Open-File Report 2007–1077

U.S. Department of the Interior
U.S. Geological Survey

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U.S. Geological Survey, Reston, Virginia 2007

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Suggested citation:
May, T.W., and Brumbaugh, W.G., 2007, Determination of total mercury in fillets of sport fishes collected from Folsom and New Melones Reservoirs, California, 2004: U.S. Geological Survey Open-File Report 2007–1077, 8 p.

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Conversion Factors

Multiply	By	To obtain
	Mass	
gram (g)	0.03527	ounce (oz)
milligram (mg)	.000035	ounce (oz)
	Length	
millimeter (mm)	0.0394	inches (in)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:
°F=(1.8×°C)+32

Concentrations of chemical constituents in solid materials are given in microgram per gram (µg/g).

Determination of Total Mercury in Fillets of Sport Fishes Collected from Folsom and New Melones Reservoirs, California, 2004

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Abstract

This report presents the results of a study by the U.S. Geological Survey, done in cooperation with the U.S. Bureau of Reclamation, to determine mercury concentrations in selected sport fishes from Folsom and New Melones Reservoirs in California. Fillets were collected from each fish sample, and after homogenization and lyophilization of fish fillets, mercury concentrations were determined with a direct mercury analyzer utilizing the process of thermal combustion-gold amalgamation atomic absorption spectroscopy. Mercury concentrations in fish fillets from Folsom Reservoir ranged from 0.09 to 1.16 micrograms per gram wet weight, and from New Melones Reservoir ranged from 0.03 to 0.94 microgram per gram wet weight. Most of the fish fillets from Folsom Reservoir (87 percent) and 27 percent of the fillets from New Melones Reservoir exceeded the U.S. Environmental Protection Agency's fish consumption advisory of 0.30 microgram per gram wet weight.

Introduction

Mercury is a byproduct of the extensive gold mining performed in the Sierra Nevada Mountains of California in the nineteenth century. After being discarded by miners, the residual mercury was gradually released into the downstream environment, including Folsom and New Melones Reservoirs. These two reservoirs are created by Folsom and New Melones Dams, which are managed by the U.S. Bureau of Reclamation (USBR) for multiple uses, including water supply and recreation. Folsom Reservoir is on the American River northeast of Sacramento, whereas New Melones Reservoir is located on the Stanislaus River 60 miles upstream from the confluence of the San Joaquin River. Both of these reservoirs support sport fisheries for several species; the concern is that mercury residues now approach or exceed guidelines for human consumption.

The U.S. Geological Survey (USGS) conducted a study in cooperation with the USBR to determine the concentrations of mercury in selected sport fishes from Folsom and New Melones Reservoirs. The State of California and USBR also are cooperating to address this concern. The California Department of Fish and Game and USBR personnel conducted all fish collections. The USBR will use the data from this study to make future management decisions concerning fish harvests from these reservoirs. The data will be provided to state and local agencies responsible for evaluating the potential risk to the public from fish consumption. Fish health advisories will be issued if necessary.

Sample History

A shipment of 36 whole-body fish and 4 dry tissue powder samples collected by USBR from Folsom Reservoir were received by the USGS on August 3, 2004. Upon receipt, the samples were assigned the USGS batch number 1036 and USGS identification numbers 32439–32478. A second shipment arrived on August 12, 2004, from USBR scientists, which consisted of 4 whole-body fish from Folsom Reservoir, 33 whole-body fish from New Melones Reservoir, and 5 dry tissue powder samples. This shipment was assigned USGS batch number 1041 and USGS identification numbers 32530–32571. A third shipment from USBR consisted of 6 whole-body fish from Folsom Reservoir, 12 whole-body fish from New Melones Reservoir, and 3 dry tissue powder samples. This final shipment was assigned USGS batch number 1054 and USGS identification numbers 32740–32760. The USBR requested that the USGS conduct a survey of mercury contamination in the edible portions (fillets) of selected sport fishes from the reservoirs.

Methods

Homogenization and Lyophilization

Filleting of whole-body fish samples was conducted with a titanium knife. A fillet was collected from one side of all fish, except for one small sample, which had fillets removed from both sides to provide a more suitable biomass. The method used to homogenize fish fillets was based on the size of the fillet sample. Fillet samples > (greater than) 200 g (grams) were processed through a Hobart® meat grinder, whereas fillet samples from 100 to 200 g were homogenized using a Kitchen Aid® blender with an attached meat processor unit. Fillet samples < (less than) 100 g were minced with a titanium knife. Homogenized samples were lyophilized with a Virtis Genesis® 35EL freeze dryer and percent moisture was determined as part of the lyophilization procedure. For dry tissue powders, residual percent moisture was determined by heating a 1-gram subsample for 4 hours at 90 to 95 °C (Celsius) in a gravity convection oven. Once dried, fillet samples were further homogenized using a Bamix® Mixer/Blender. All dried samples were stored in glass vials in a desiccator.

Instrumental Analysis and Data Reporting

Mercury was determined with a direct mercury analyzer. With this method, a dried fish sample of approximately 50 to 100 mg (milligrams) was combusted in a stream of oxygen. All mercury in the sample was volatilized and trapped by amalgamation on a gold substrate and thermally desorbed and quantitated by atomic absorption spectrophotometry (U.S. Environmental Protection Agency, 2003). The entire sequence was conducted with a Milestone Direct Mercury Analyzer (DMA-80) equipped with an automated sample carousel. Duplicate determinations were conducted for each sample and the mean of the two analyses was reported; however, if the relative percent difference (RPD) among duplicates exceeded 20 percent, an additional analysis was performed and the concentration expressed as the mean of all three analyses. The concentrations of mercury measured in dried fillet samples was converted to wet weight for reporting based on moisture contents determined by lyophilization, but concentrations of samples received as dry powders were reported “as received.” Based on our historical measurements, residual moisture of homogenized, desiccator-stored fish fillet tissue typically is between 2 and 3 percent, but we made no attempt to apply a correction for residual moisture of individual dry

samples because the mercury analyzer is calibrated with dried certified reference tissues having similar ranges of moisture content. The uncertainty of reported wet-weight concentrations, potentially because of moisture differences among the individual dried unknown samples and the certified reference samples used for the calibration, is expected to be considerably less than the overall method uncertainty [about ± (plus or minus) 5 to 10 percent for mean concentrations well above the quantitation limit]. Furthermore, the uncertainty associated with moisture variation among dry sample determinations should be no greater than the uncertainty associated with moisture variation among fresh or frozen fish fillet sample analyses.

Results and Discussion

Percent moisture and mean concentrations of total mercury measured in fish fillet samples collected from various sport fishes of Folsom Reservoir are presented in table 1. Percent moisture in fillets ranged from 75.0 to 85.4 percent and averaged 79.4 percent. Mercury concentrations ranged from 0.09 to 1.16 μg/g (micrograms per gram) wet weight and averaged 0.58 μg/g wet weight. Out of 46 fish fillet samples, 40 (87 percent of the sample set) contained mercury concentrations exceeding the U.S. Environmental Protection Agency fish consumption advisory of 0.30 μg/g wet weight (U.S. Environmental Protection Agency, 2001).

Percent moisture in fillets of fish from New Melones Reservoir ranged from 66.4 to 81.8 percent and averaged 75.9 percent (table 2). However, all of the salmon submitted from New Melones Reservoir (32747–32754; 32756–32759) were in poor condition; for example, the flesh appeared to be partially decomposed and the moisture contents for these particular fish (66.4 to 73.3 percent) were lower than normal. Adjustment to a normal moisture content might be preferable if the mercury concentrations in these fillets are to reflect "fresh" fish samples. Total mercury concentrations in fish fillet samples from New Melones Reservoir (table 2) ranged from 0.03 to 0.94 μg/g wet weight and averaged 0.23 μg/g wet weight. Out of 45 fish fillet samples, 12 (27 percent of the sample set) exceeded the 0.30 μg/g fish consumption advisory guideline (U.S. Environmental Protection Agency, 2001).

Percent moisture and concentrations of total mercury in submitted dry tissue are presented in table 3. Percent moisture ranged from 3.1 to 4.3 percent. Mercury concentrations ranged from 2.83 to 2.94 μg/g dry weight and 5.09 to 5.32 μg/g dry weight. As mentioned earlier, mercury concentrations were not corrected for residual moisture because the DMA-80 was calibrated using dried reference tissue powders with comparable residual moisture values.

Quality Control

The samples were handled in five groups or blocks through the instrumental analysis. Quality control included blanks, replicates, pre-combustion spikes, and tissue reference materials. During the instrumental run, additional quality control included independent calibration verification checks.

For each group or block of samples, an independent calibration verification sample [National Research Council Canada (NRCC) DOLT-2] was analyzed at the beginning and end of the instrumental run to confirm the calibration status of the DMA-80 system; each measured calibration sample was within ±10 percent of the certified concentration. Four reference tissues were analyzed for mercury: NRCC DORM-2 [n=9 (9 samples)], National Institute of Standards and Technology (NIST) RM50 (n=5), International Atomic Energy Agency (IAEA) 407 (n=5), NRCC DOLT-2 (n=5); recoveries of mercury were within certified or recommended ranges. Method precision can be estimated either from the RPD from the duplicate analysis of tissue samples or as percent relative standard deviation (RSD)

Table 1. Total mercury concentrations in fillets of sport fishes from Folsom Reservoir, California, 2004.

[USGS, U.S. Geological Survey; ID, identification; n, number; µg/g, micrograms per gram wet weight]

USGS ID number	Field/lab ID number	Percent moisture	n	Mean total mercury (µg/g)
32439	FLF 001	78.0	2	0.51
32440	FLF 002	80.3	2	0.47
32441	FLF 003	83.4	2	0.65
32442	FLF 004	81.7	2	0.56
32443	FLF 005	80.6	2	0.37
32444	FLF 006	81.5	2	0.59
32445	FLF 007	77.8	2	0.50
32446	FLF 008	81.2	2	0.49
32447	FLF 009	80.0	2	0.51
32448	FLF 010	80.7	2	1.04
32449	FLF 011	75.9	2	0.71
32450	FLF 012	77.4	2	0.94
32451	FLF 013	78.7	2	0.56
32452	FLF 014	79.4	2	0.36
32453	FLF 015	75.9	2	0.59
32454	FLF 016	80.3	2	0.38
32455	FLF 017	77.2	2	1.02
32456	FLF 018	79.0	2	0.65
32457	FLF 019	79.4	2	0.43
32458	FLF 020	76.6	2	0.80
32459	FLF 021	80.4	2	0.38
32460	FLF 022	78.0	2	0.64
32461	FLF 023	77.7	2	0.87
32462	FLF 024	76.2	2	0.83
32463	FLF 025	82.0	2	0.18
32464	FLF 026	84.4	2	0.09
32465	FLF 027	85.4	2	0.33
32466	FLF 028	80.3	2	0.51
32467	FLF 029	78.2	2	0.60
32468	FLF 030	78.5	2	0.56
32469	FLF 031	82.3	2	0.47
32470	FLF 032	81.2	2	0.98
32471	FLF 033	79.6	2	0.92
32472	FLF 034	76.5	2	1.16
32473	FLF 035	78.7	2	0.74
32474	FLF 036	75.3	2	0.45
32567	FLF 038	78.7	2	0.85
32568	FLF 039	78.4	2	1.00
32569	FLF 040	75.0	2	0.47
32570	FLF 041	83.7	2	0.12
32740	FLF 043	78.2	2	0.10
32741	FLF 044	80.0	2	0.46
32742	FLF 045	83.2	2	0.91
32743	FLF 046	75.9	2	0.81
32744	FLF 047	77.8	2	0.11
32745	FLF 048	82.2	2	0.22

Table 2. Total mercury concentrations in fillets of sport fishes from New Melones Reservoir, California, 2004.

[USGS, U.S. Geological Survey; ID, identification; n, number; µg/g, micrograms per gram wet weight]

USGS ID number	Field/lab ID number	Percent moisture	n	Mean total mercury (µg/g)
32530	NMRF 001	78.8	2	0.24
32531	NMRF 002	75.6	2	0.11
32532	NMRF 003	78.1	2	0.28
32533	NMRF 004	78.5	2	0.09
32534	NMRF 005	77.5	2	0.08
32535	NMRF 006	76.9	2	0.15
32536	NMRF 007	76.8	3	0.19
32537	NMRF 008	75.5	2	0.11
32538	NMRF 009	79.0	2	0.06
32540	NMRF 011	77.5	2	0.49
32541	NMRF 012	79.7	2	0.31
32542	NMRF 013	78.3	2	0.45
32543	NMRF 014	81.8	2	0.94
32544	NMRF 015	79.7	2	0.54
32545	NMRF 016	79.6	2	0.60
32546	NMRF 017	80.2	2	0.40
32547	NMRF 018	79.0	2	0.33
32548	NMRF 019	78.9	2	0.34
32550	NMRF 021	72.2	2	0.09
32551	NMRF 022	79.0	2	0.05
32552	NMRF 023	71.1	3	0.16
32553	NMRF 024	73.7	2	0.05
32554	NMRF 025	80.0	2	0.18
32555	NMRF 026	70.6	2	0.18
32556	NMRF 027	78.3	2	0.12
32557	NMRF 028	78.7	2	0.05
32558	NMRF 029	80.6	2	0.03
32560	NMRF 031	80.1	2	0.68
32561	NMRF 032	80.3	2	0.41
32562	NMRF 033	78.7	2	0.27
32563	NMRF 034	78.8	2	0.27
32564	NMRF 035	78.7	2	0.53
32565	NMRF 036	80.1	2	0.24
32747	NMRF 038	72.7	2	0.08
32748	NMRF 039	72.2	2	0.09
32749	NMRF 040	69.8	2	0.11
32750	NMRF 041	66.4	2	0.13
32751	NMRF 042	67.9	2	0.12
32752	NMRF 043	69.9	2	0.14
32753	NMRF 044	68.3	2	0.11
32754	NMRF 045	67.9	3	0.11
32756	NMRF 047	71.6	2	0.09
32757	NMRF 048	71.4	2	0.11
32758	NMRF 049	73.3	2	0.07
32759	NMRF 050	72.4	2	0.09

Table 3. Total mercury concentrations in dry tissue powders submitted with whole-body fish samples, 2004.
[USGS, U.S. Geological Survey; ID, identification; n, number; µg/g, micrograms per gram dry weight]

USGS ID number	Field/lab ID number	Percent moisture	n	Mean total mercury (µg/g)
32475	FLF 005A	3.8	2	2.94
32477	FLF 025A	4.3	2	2.84
32549	NMRF 020	4.1	2	2.83
32571	FLF 042	3.1	2	2.88
32760	NMRF 051	4.0	2	2.87
32476	FLF 015A	4.1	2	5.29
32478	FLF 037	3.9	2	5.15
32539	NMRF 010	3.7	2	5.11
32559	NMRF 030	3.5	2	5.14
32566	NMRF 037	3.2	2	5.21
32746	FLF 049	4.1	2	5.09
32755	NMRF 046	3.5	2	5.32

from triplicates. The overall mean RPD ± the standard deviation for 103 duplicate pairs of fish fillets (n=91) and fish powders (n=12) was 4.7 percent ±4.5. RPDs were <10 percent for all samples except seven, the latter ranging from 10.3 to 23 percent. Samples with RPDs >20 percent (n=3) were analyzed again to obtain a triplicate analysis. Percent RSDs from triplicate analyses of these and other arbitrarily selected samples (n=13) used for spike recovery analysis were ≤13 percent. Percent recovery of methylmercury hydroxide from pre-combustion tissue spikes (n=10) ranged from 106 to 125 percent, and averaged 113 percent. Although all spike recoveries were above 100 percent, the mean recovery was well within quality control protocol target limits (80 to 120 percent). Moreover, spike recoveries are not necessarily the best accuracy indicator for the DMA-80 method because the instrumental response for liquids is sometimes slightly different than for solids. Blank equivalent concentrations (BECs) for total mercury exceeded method detection limits for three of the five sample blocks, but the lowest sample concentration (0.03 µg/g wet weight) was about 200 times the highest BEC concentration (0.00014 µg/g wet weight). The instrument detection limit was 0.0053 µg/g dry weight mercury. The method detection limits ranged from 0.00004 to 0.00021 µg/g wet weight mercury, and limits of quantitation ranged from 0.0014 to 0.0077 µg/g wet weight. Overall, the quality control was within acceptable limits as specified by the USGS.

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