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TECHNICAL REPORT

ORGANOCHLORINE CONCENTRATIONS IN BURBOT (*Lota lota*) LIVERS FROM FAIRBANKS, ALASKA, AND KANUTI, TETLIN AND YUKON FLATS NATIONAL WILDLIFE REFUGES, ALASKA, 1998

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EXECUTIVE SUMMARY

This study was conducted by U.S. Fish and Wildlife Service biologists during 1998 and 1999. Burbot liver was the tissue of choice for this study because chlorinated hydrocarbons, or organochlorine compounds, are lipophilic and burbot use the liver as their main somatic fat reserve (Love 1980). In addition, burbot consume high on the aquatic food chain and, therefore, are subject to the effects of biomagnification. The objectives of this study were to: determine the concentrations of organochlorine compounds in burbot livers at four sample sites in Interior Alaska; determine the significance of these data by comparing organochlorine concentrations in burbot livers with regional data for organochlorines in burbot and concentrations shown to cause effects in fish; and determine if further investigation of organochlorine contamination in biota of interior Alaska is warranted.

Organochlorines are a particularly onerous group of compounds because they are lipophilic, persistent in the environment, bioaccumulate and biomagnify, and are neuroactive agents (Hoffman et al. 1995). Examples of organochlorine compounds are polychlorinated biphenyls (PCBs), DDT and its degradation products DDE and DDD, variations and degradation products of chlordane, and chlorinated benzenes such as hexachlorobenzene (HCB). With some exceptions, e.g., PCBs (Loganathan and Kannan 1994), concentrations of organochlorines in biota are generally declining (Schmitt et al. 1999) due to numerous prohibitions on their use and production.

Burbot liver samples were collected from the Tanana River below Fairbanks, Kanuti National Wildlife Refuge at Bettles (Koyukuk River), Tetlin National Wildlife Refuge (Tanana River), and Yukon Flats National Wildlife Refuge at Beaver (Yukon River). Twenty-nine burbot were collected. Liver samples were dissected using hexane-cleaned stainless steel instruments at a Fish and Wildlife Service laboratory in Fairbanks.

In general, there were greater contaminant concentrations from the site below Fairbanks and Yukon Flats than from Tetlin and Kanuti. Analysis of the data was complicated by differing lipid concentrations in samples, differing fish weights among sites, and by a low sample size at Yukon Flats. Lipid concentrations of samples from Fairbanks and Yukon Flats were significantly greater than those of samples from Kanuti ($F_{3,25} = 8.5$, $P < 0.001$). There were greater concentrations of DDT and its metabolites at Fairbanks than at other sites, probably reflecting historical use of that pesticide within the city of Fairbanks and at nearby military bases. Concentrations of ∑DDT from Fairbanks are up to two orders of magnitude greater than from five of six studies in Canada. The range of ∑PCB concentrations from our study are similar to those from four of six Canadian studies cited and were generally less than laboratory-derived effects values. Toxaphene concentrations from our study were generally low. Further studies would help illuminate whether the concentrations we found at Fairbanks and Yukon Flats are of concern to fish and wildlife resources.

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ACKNOWLEDGMENTS

The authors express their sincere thanks to Patrick Sousa, Neesha Wendling, Philip Johnson, U.S. Fish and Wildlife Service, and Phyllis Weber Scannell and Matt Evenson, Alaska Department of Fish and Game, who provided valuable reviews of this report. In addition, we thank Carl Lunderstadt, Barry Whitehill, Paul Williams and Carl Hemming who helped collect samples, Kara Weller who helped dissect the specimens, and Randy Brown for preparation and reading of the otoliths. We also thank Philip Martin and Tim Obritsckewitsch for their assistance in producing Figure 1.

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TABLE OF CONTENTS

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INTRODUCTION

This study was conducted at four sites in interior Alaska, including Kanuti, Tetlin and Yukon Flats National Wildlife Refuges, and Fairbanks, Alaska. The purposes of Kanuti, Tetlin, and Yukon Flats National Wildlife Refuges include:

1. To conserve fish and wildlife populations and habitats in their natural diversity;

2. To fulfill the international treaty obligations of the United States with respect to fish and wildlife and their habitats;

3. To provide, in a manner consistent with purposes 1 and 2 above, the opportunity for continued subsistence uses (which included freshwater fish) by local residents; and

4. To ensure, to the maximum extent practicable and in a manner consistent with purpose 1 above, water quality and necessary water quantity within the refuge (USFWS 1987a,b,c).

In addition, the Tetlin and Yukon Flats Refuges include conservation of salmon as a refuge purpose. Tetlin Refuge also includes conservation of other fish species as a refuge purpose. This study was designed to benefit the above refuge purposes and Service trust resources.

Acceptably low contaminant concentrations are critical components of water quality and fish and wildlife habitat. Acceptable concentrations are those which will not adversely affect fish and wildlife populations or their habitats.

Chlorinated hydrocarbons, or organochlorines, are a particularly onerous group of compounds because they are lipophilic, persistent in the environment, bioaccumulate and biomagnify, and are neuroactive agents (Hoffman et al. 1995). Examples of organochlorine compounds are polychlorinated biphenyls (PCBs), dichloro-diphenyltrichloroethane (DDT) and its degradation products DDE and DDD, variations and degradation products of chlordane, and chlorinated benzenes such as hexachlorobenzene (HCB). With some exceptions, e.g., PCBs (Loganathan and Kannan 1994), concentrations of organochlorines in biota are generally declining (Schmitt et al. 1999) due to numerous prohibitions on their use and production.

Persistent organic pollutants such as PCBs and organochlorine pesticides such as DDT have been shown to reach subarctic and arctic areas by global atmospheric transport (Bidleman et al. 1989, Patton et al. 1989, Muir et al. 1990a, Wilson et al. 1995, Barrie et al. 1997, Ewald et al. 1998). Airborne concentrations of persistent organic pollutants in Arctic air are comparable to those in more populated and industrialized regions of North America and Europe (Fellin et al. 1996). Studies in Yukon and Northwest Territories, Canada have identified mercury, toxaphene and PCBs as potential concerns in freshwater fish, and cadmium, mercury, PCBs and radionuclides

as potential concerns in terrestrial animals and waterfowl (Jensen et al. 1997). Additionally, point sources, such as mining, landfills, and defense sites, all of which occur on or near Interior Alaska National Wildlife Refuges, may result in locally high contaminant concentrations.

The unique conditions of the arctic and subarctic affect not only deposition of organochlorine contaminants but also their subsequent transformations. For example, the presence of endosulfan in the Bering-Chukchi seas is compelling evidence that chemicals classified as "low persistence" in temperate climates are more recalcitrant in the Arctic (Bidleman 1996). Gregor et al. (1998) review the influence of physical and chemical processes on contaminant transport into and within the Arctic, while de March et al. (1998) provide a good overview of pollutant sources, chemical fate and bioaccumulation of persistent organic pollutants in Arctic food webs.

Ewald et al. (1998) have shown that migrating salmon transport pollutants into freshwater ecosystems. They found that Arctic grayling (*Thymallus arcticus*) from a salmon spawning lake had more than twice the concentrations of organic pollutants than grayling from a nearby salmon-free lake. Toxaphene concentrations in Lake Laberge, Yukon Territory, were as high as 2000 µg/kg wet weight in burbot liver and 350 µg/kg in lake trout (*Salvelinus namaycush*) muscle. Although high concentrations of toxaphene in biota of Lake Laberge have been partially attributed to food web differences in this lake compared to other lakes (Kidd et al. 1995a), this study demonstrates the presence of these compounds in the subarctic and the potential for their biomagnification in disturbed environments of the subarctic. Health Canada has issued public health advisories on consumption of some fish tissues from Lake Laberge (Eamer 1996).

Following are descriptions of three of the most common groups of organochlorine compounds. PCBs are a group of 209 synthetic halogenated aromatic hydrocarbons, although Aroclors, commercial mixtures measured in this study, may contain only 132 PCB congeners (Schulz et al. 1989). PCBs have been used as heat transfer agents in electrical transformers and capacitors, and in paints, lubricants, flame retardants, placticizers and waterproofing. PCBs have been shown to adversely affect survival, and cause reduced growth, liver damage, tumors, reduced survival of developing eggs, fry deformities, and reproductive failure in fish (Eisler 1986). The manufacture, processing, distribution, and use of PCBs, except in enclosed systems, was banned in the U.S. in 1979 (Eisler 1986). PCBs are widely distributed in the environment and releases from old stores and uses still occur. Residual PCBs remain a problem in some areas as evidenced by human consumption advisories in effect for the Great Lakes, Lake Champlain, the Hudson River, and elsewhere. Trace concentrations of the more persistent, more highly chlorinated PCBs have been detected in fish from almost every major river in the United States (Schmitt et al. 1999).

DDT is the best known and the first successful organochlorine pesticide. First synthesized in 1874, Paul Muller discovered its insecticidal activity in 1939 and subsequently received a Nobel Prize for his work. Early use in North America involved application of high concentrations of DDT to control Dutch elm disease. More than 90 species of birds, particularly American robins (*Turdus migratorius*), were found dead in neighborhoods sprayed with DDT (Hoffman et al.

1995). Nearly all deleterious effects of DDT are caused by the *para-para* (p,p') isomer of DDT and its degradation products p,p'-DDE and p,p'-DDD. Sublethal effects of DDT and its metabolites, primarily DDE, include eggshell thinning, embryotoxicity, and related adverse effects on avian reproductive success. All registrations of DDT in the U.S. were canceled by the Environmental Protection Agency in 1973. DDT and metabolites are widely distributed. In 1986, Schmitt et al. (1999) detected DDT or its metabolites in fish at all of their 107 sample sites across the United States.

Toxaphene is a broad spectrum pesticide composed of a mixture of up to 177 polychlorinated camphene compounds. Toxaphene replaced DDT as the insecticide of choice for cotton farming and many other uses (Schmitt et al. 1990), including soybeans and peanuts. It also has been used as a piscicide. Fortunately, it does not accumulate in birds (Eisler and Jacknow 1985). However, toxaphene is extremely toxic to freshwater and marine biota where it affects growth, learning and behavior, survival and reproduction, and causes backbone abnormalities (Eisler and Jacknow 1995, de Geus et al. 1999, Delorme et al. 1999). Toxaphene also has been found to be highly carcinogenic in rats and mice (de Geus et al. 1999). Toxaphene was banned for use in the United States by EPA in 1982, a practice that was followed by many other countries, although Argentina and Mexico still allow restricted use (de Geus et al. 1999). Concentrations of toxaphene in fish plateaued around 1980 after a period of steady increase during the 1970s. During 1980 and 1981, toxaphene was detected in fish at 88% of stations sampled throughout the United States (Schmitt et al. 1999). Toxaphene is frequently the most abundant pesticide detected in Arctic aquatic organisms (Wade et al. 1997).

This study was conducted by U.S. Fish and Wildlife Service biologists during 1998 and was designed as a preliminary survey to determine if further research should be done. Burbot liver was the tissue of choice for this study because organochlorine compounds are lipophilic, burbot use the liver as the main somatic fat reserve (Love 1980), and burbot consume high on the food chain and, therefore, are subject to biomagnification.

The objectives of this study were to:

1. Determine the concentrations of organochlorine compounds in burbot livers from Fairbanks, Alaska, and Kanuti, Tetlin and Yukon Flats National Wildlife refuges in Interior Alaska.

2. Compare concentrations of organochlorine compounds in burbot livers with regional data for organochlorines in burbot and concentrations shown to cause effects in fish.

3. Determine if further investigation of organochlorine contaminants in biota of Interior Alaska is warranted.

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METHODS AND MATERIALS

Sample Sites

Samples were collected at seven sites from four general locations. Two of the locations were in the Tanana River drainage (four sites on Tetlin Refuge and one site near Fairbanks), one location was on the Koyukuk River at Bettles (Kanuti), and one location was on the Yukon River at Beaver (Yukon Flats) (Figure 1). Sample site descriptions are as follows.

Site FA01, Tanana River at Fairbanks, T. 12 S, R. 2 W, Sec. 5, SE 1/4, Fairbanks Meridian (FM); 64°47'29" N, 147°57'30" W.

Site KA01, Koyukuk River at Bettles (Kanuti), T. 24 N, R.18 W, Sec. 8, NW 1/4, FM; 66°54'30" N, 151°41'30" W.

Site TE09, Tanana River at the mouth of Moose Creek, T. 14 N, R. 19 E, Sec. 9, NW 1/4, Copper River Meridian (CRM); 63°00'44" N, 141°48'57" W.

Site TE13, Tanana River at the mouth of the Kalutna River, T. 16 N, R. 16 E, Sec. 22, NE 1/4, CRM; 63°10'30" N, 142°24'00" W.

Site TE20, Tanana River at Mile Post 1303.3, T. 18 N, R. 14 E, Sec. 25, NW 1/4, CRM; 63°19' 00" N, 142°38'40" W.

Site TE21, Chisana River, at the mouth of Scottie Creek, T. 11 N, R. 22 E, Sec. 34, SE 1/4, CRM; 62°40'24" N, 141°15'30" W.

Site YF20, Yukon River at Beaver (Yukon Flats), T. 18 N, R. 2 E, Sec. 30, FM; 66°21' 30" N, 147°23' 30" W.

Collection Methods

Fish were collected at Fairbanks on 9/8-9/98, at Kanuti Refuge on 2/10/98, at Tetlin Refuge between 4/30/98 and 7/22/98, and at Yukon Flats Refuge on 8/1/98. Burbot were collected by angling. Each fish was labeled in the field and frozen upon returning from the field each day. Whole fish were stored in Fairbanks in an ultralow freezer (-40°C) until dissection. Whole fish samples were weighed to the nearest gram, and total lengths were measured to the nearest millimeter. Liver samples were dissected using hexane-cleaned stainless steel instruments at a Fish and Wildlife Service laboratory in Fairbanks. A new carbon steel scalpel blade was used for each specimen. Samples were transferred to precleaned 125-mL I-Chem glass bottles and frozen (-40°C) pending shipment to the analytical laboratory. Fish age was estimated by surface

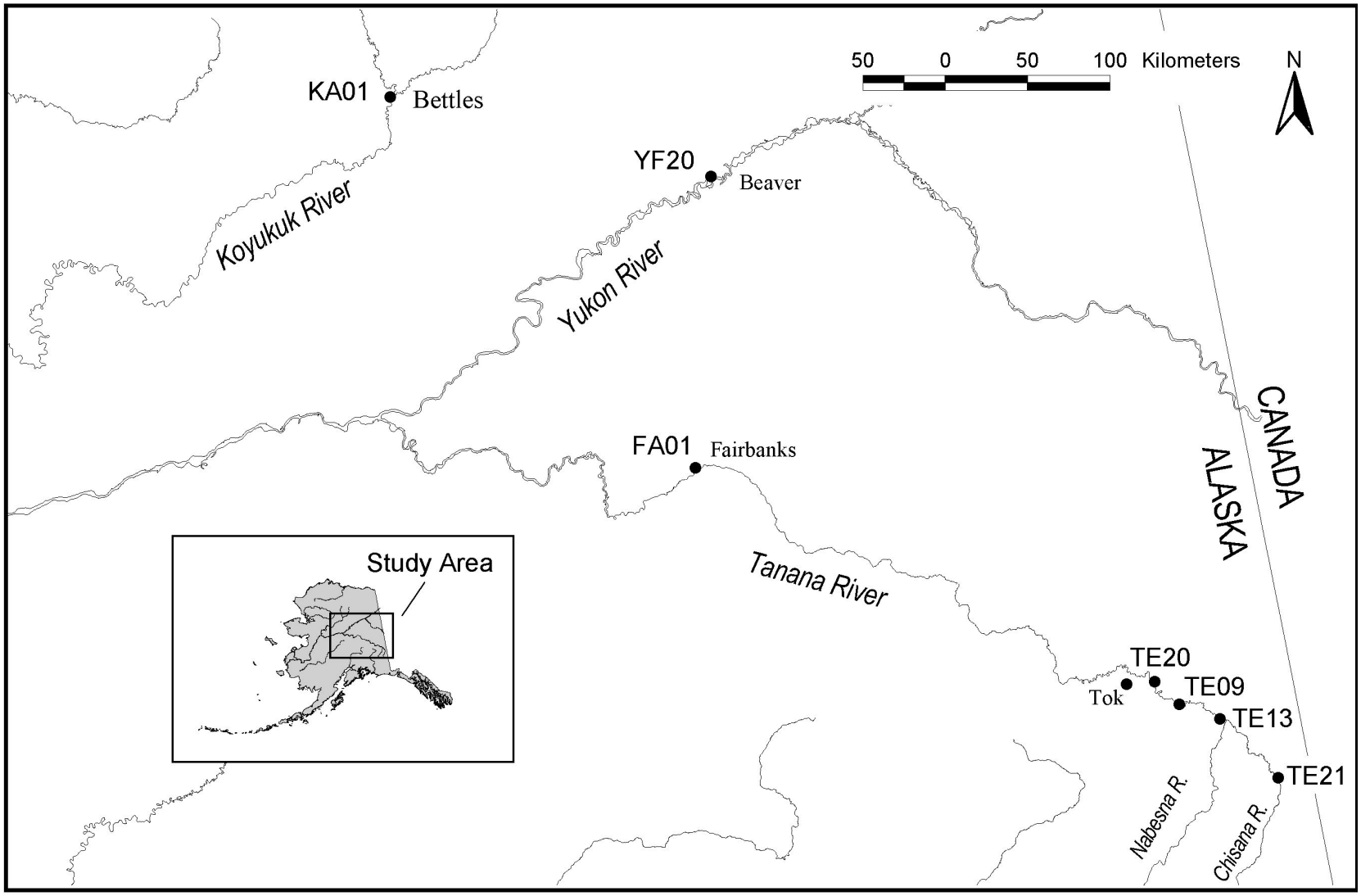

Figure 1. Locations of sample sites at Fairbanks, Alaska, and Kanuti, Tetlin and Yukon Flats National Wildlife Refuges, Alaska, 1998.

readings of otoliths according to Clinton and Beamish (1982).

Chemical Analyses

Samples were analyzed for organochlorine concentrations by AXYS Analytical Services, Sydney, B.C., Canada. Liver samples were homogenized using a Virtis blender and frozen. The homogenized samples were extracted by grinding the sample with anhydrous sodium sulphate, spiking with surrogate standards, and refluxing in a soxhlet apparatus for 16 to 20 hours. The cooled extract was concentrated by rotary evaporation. Concentrated extracts were loaded onto calibrated gel permeation columns (Biobeads SX-3) to remove high molecular weight interferences. The column was eluted with 1:1 dichloromethane and the second fraction collected. This fraction was concentrated by rotary evaporation, prior to additional chromatographic cleanup procedures. The extract was loaded onto a Florisil column (2.1% deactivated) which was eluted with hexane followed by 15:85 dichloromethane:hexane. This fraction contained chlorinated pesticides, toxaphene, and PCB congeners. The fraction was concentrated, an aliquot of recovery standard was added, and was then transferred to an autosampler vial in preparation for instrumental analysis. For analysis of non-ortho-substituted congeners, the fraction was first split and one-half subjected to additional cleanup on carbon/Celite to isolate the non-ortho substituted PCBs. The Florisil column was eluted with 1:1 dichloromethane:hexane and the eluate collected. This fraction contained polar chlorinated pesticides. The fraction was concentrated and an aliquot of recovery standard was added and then transferred to an autosampler vial in preparation for instrumental analysis.

Analysis of pesticides and PCBs (as Aroclors) was carried out using a Finnigan INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC A200S autosampler, and a DG10 data system running Incos 50 (Rev 11) software. The MS was operated at unit mass resolution in the Multiple Ion detection mode. Chromatographic separation was achieved with a DB-5 capillary column (60 m, 0.25 mm i.d., 0.10 µm film thickness). Analytes were quantified using the internal standard method of quantification, comparing the area of analyte peak to that of the corresponding surrogate standard and correcting for response factors.

Analysis of toxaphene used the same hardware and software as above. The MS was operated in the electron capture negative ionization mode. Chromatographic separation and quantification of analytes were achieved in the manner described above.

Analysis of the most polar chlorinated pesticides was accomplished using a Hewlett Packard 5890A gas chromatograph equipped with a 63Ni electron capture detector. Chromatographic separation was achieved using a DB-5 capillary column (60 m, 0.25 mm i.d., 0.10 µm film thickness). Quantification was achieved similar to the above method.

Percent moisture was determined by drying a weighed subsample for at least 16 hours at 105° C and then re-weighing. Percent moisture was calculated as the percent difference between the dry weight and the wet weight.

A percent lipid determination was carried out on extracts by drying two weighed subsamples of extract at 105°C for 30 minutes. The extracts were re-weighed and the percent lipid was calculated as the weight of lipid/wet sample weight.

Quality Assurance/Quality Control

Sample Collections

Sampling was conducted following a written study plan. Briefly, at the time of collection, samples collected and other pertinent data were recorded in a field notebook. Fish were rinsed with river water from the site of collection to minimize external contamination. Dissections were performed on semi-frozen fish to minimize sample contamination due to movement of fluids within the body cavity. Dissections were conducted using hexane-washed stainless steel and teflon dissection equipment on a clean, hexane-washed surface, with a new carbon steel blade for each sample. Tissues were placed in storage containers immediately after dissection and refrozen. Samples were shipped to the laboratory in coolers containing dry ice by overnight air courier.

A sample catalog was prepared prior to submittal of samples to the analytical laboratory. The catalog contained an identifier for the sample batch; study objectives; instructions to the laboratory on analyses requested; and a tabulated summary of all samples including a unique 10-digit identification number for each sample, species, tissue type, collection location, collection date, sample weight, and other variables.

Chemical Analyses

Laboratory quality assurance/quality control (QA/QC) procedures, screening criteria to accept or reject analytical data, screening results, and the basis for rejection of analytical data are described in Appendices B and C. In summary, duplicate (split) samples and spiked analyses were used to evaluate data quality. Table 1 identifies duplicate and spike analysis results, and method limits of detection (LODs). Data were considered acceptable for publication if they met the following criteria: the mean of all mean spike recoveries should be $\geq$ 85% and $\leq$ 115%; mean duplicate analysis relative percent differences (RPDs) should be $\leq$ 15%; and, no more than 3 individual analytes should be < 80% for mean spike recoveries or have > 20% mean RPDs. No analytes were rejected.

Concentrations reported for an analyte that are less than twice the LOD should be considered qualitative only. Values between 2 and 10 times the LOD should be considered semi-quantitative, i.e., liable to more variability than in the zone of quantitation, where measured values are greater than 10 times the LOD.

Table 1. Method limits of detection (LOD), duplicate analyses relative percent differences (RPD), and mean spike analytical results for burbot liver analyses. Values for some QA/QC variables were less than two times the LOD; these values are considered qualitative, were not used for QA/QC evaluations, and are indicated by blank cells.

Element	LOD (mg/kg)	Duplicates RPD (%)	Mean Spike (% recovery)
HCB	0.00021	6[a]	88
Aroclor 1242	0.00680		92
Aroclor 1248	0.00680		
Aroclor 1254	0.01600	22	95
Aroclor 1260	0.00580	0	97
∑PCBs[b]	0.01600		96
Alpha BHC	0.00200		88
Alpha chlordane	0.00046		94
Dieldrin	0.00026	0	71
Endrin	0.00068		87
Gamma BHC	0.00260		90
Gamma chlordane	0.00038		88
Heptachlor epoxide	0.00032		84
Mirex	0.00035		97
o,p'-DDD	0.00040		95
o,p'-DDE	0.00045		94
o,p'-DDT	0.00045		95
Oxychlordane	0.00490		97
p,p'-DDD	0.00052	0	88
p,p'-DDE	0.00092	2	90
p,p'-DDT	0.00075	0	88
Trans-nonachlor	0.00150		93
Toxaphene	0.00290		97

[a] Mean value, n = 2.
[b] The sum of PCBs-1242, 1248, 1254, and 1260.

Statistical Analyses

We compared contaminant concentrations in burbot liver among sites. Our goal was to use multivariate, parametric tests, primarily Analysis of Variance (ANOVA) with site as the factor and contaminant concentrations as the response variables. Multivariate tests reduce the increased experiment-wise error rate associated with numerous univariate tests, and may also discern patterns not evident in univariate data (Weis and Muir 1997, Sparks et al. 1999). Parametric tests are generally more powerful than non-parametric tests, but require that data sets have a high

proportion of detections. Non-parametric tests (primarily Kruskal-Wallis rank sum tests or ANOVAs on ranks) were dictated when any analyte had $\geq$ 50% of data below the LOD at any site, although these analyses were not amenable to multivariate data or use of covariates. Actual tests employed therefore depended upon the percent of data below the LOD by site (Table 2).

Analytes detected in no samples or only one sample were not statistically analyzed. If an analyte had $\geq$ 50% of data below the LOD at two sites, concentrations were compared among all four sites using ANOVA on ranks, where all data below the LOD were tied at the lowest rank (Helsel 1990). If an analyte had $\geq$ 50% of data below the LOD at only one site, we compared contaminant concentrations at the other three sites with the assumption that mean values from the dropped site, although unknown, were lower compared to sites with higher rates of detection. If an analyte had < 50% of data below the LOD at all four sites, concentrations were compared using a parametric ANOVA or Analysis of Covariance (ANCOVA).

For parametric tests, non-detect data were substituted with a random number between zero and the detection limit (Mazak et al. 1997), since a small number of such substitutions are unlikely to affect estimation, and data were log-transformed to achieve normality and stabilize variance. Non-parametric tests were performed on ranked data, with no transformations or substitutions. Analyses were done using SYSTAT 8.0, with $\alpha = 0.05$ unless stated otherwise. All post-hoc multiple comparisons were performed with the conservative Scheffe's method.

Biomagnifying compounds are lipophilic and tend to increase over an organism's lifetime, so we determined if percent lipid (Hebert and Keenleyside 1995, Kidd et al. 1998) and fish weight (as a surrogate for fish age) (Muir et al. 1990b) were correlated with contaminants, and therefore needed to be accounted for when comparing contaminant concentrations. If percent lipid and fish weight were significantly correlated with contaminant concentrations (Pearson r, $P \leq 0.05$), they were included as covariates in our ANOVAs (which then became ANCOVA). Site*lipid and site* weight interaction terms were included in ANCOVAs (Hebert and Keenleyside 1995). Covariates were not included when non-parametric tests were used.

Organochlorine contaminants are often correlated with each other in biota, and this property can be used to help reduce the actual number of response variables tested in multivariate tests. If the majority of contaminants were significantly correlated with each other (Pearson r, $P \leq 0.05$), we reduced them to Principal Components (PCs). Factor scores from the first PCs that accounted for > 80% of the variability in the data set were used as response variables for comparisons among sites. This option was available only for parametric, multivariate analyses.

Table 2: Summary of statistical analyses, based on the percent of samples with analyte concentrations less than the Limit of Detection, used to test for differences in analyte concentrations among sites.

Percent of non-detections	Statistical analysis
Analytes that were not detected in any or only one sample	Not analyzed.
Analytes that had $\geq$ 50% of data below the LOD at two or more sites	Univariate non-parametric ANOVA on ranks, Scheffe's post-hoc testing.
Analytes that had $\geq$ 50% of data below the LOD at only one site	Site dropped, then multivariate ANOVA on concentrations or factor scores from principal components analysis on remaining sites; lipid and weight covariates.
Analytes with < 50% of data below the LOD at all sites	Multivariate ANOVA on concentrations or factor scores from principal components; lipid and weight covariates.

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RESULTS

Twenty-nine burbot were collected, including 20 from the Tanana River (9 from below Fairbanks and 11 from Tetlin Refuge), 3 from the Yukon River (Yukon Flats Refuge), and 6 from the Koyukuk River (Kanuti Refuge). Burbot were collected from Fairbanks, Kanuti, and Yukon Flats during September, February, and August, respectively; and during April, May and July at Tetlin (Table 3). Fish from Kanuti, except one, were undergoing gametogenesis at the time of capture. Fish from other sites were not undergoing gametogenesis at the time of capture. Lipid concentrations of samples from Fairbanks and Yukon Flats were significantly greater than those of samples from Kanuti ($F_{3,25} = 8.5$, $P < 0.001$) (Table 3). Burbot sampled at Yukon Flats had significantly greater total length ($F_{3,24} = 19.5$, $P < 0.001$) and weight ($F_{3,25} = 42.4$, $P < 0.001$) than burbot from the other three sites (Table 3). Burbot age was not significantly different among Fairbanks, Kanuti, and Tetlin sample sites ($F = 1.5$, $P > 0.05$); Yukon Flats otolith ages were not determined. Lipid concentrations were significantly positively correlated with dieldrin ($n = 24$, $r = 0.44$, $P = 0.04$), HCB ($n = 24$, $r = 0.63$, $P < 0.001$), and heptachlor epoxide ($n = 24$, $r = 0.5$, $P = 0.002$) concentrations. Weight was significantly positively correlated with alpha-chlordane, dieldrin, HCB, heptachlor epoxide, mirex, toxaphene, and trans-nonachlor concentrations ($P = 0.006$ to 0.031).

Table 3. Mean and range (in parentheses) of lipid concentrations, and weight and length of sampled burbot for each site. Unlike letters indicate significant differences among sites for percent lipid ($F_{3,25} = 8.5$, $P < 0.001$).

Site	n	Collection Date	Lipid (%)		Weight (g)	Length (mm)
Fairbanks	9	9/8/98-9/9/98	40 (6-57)	A	745 (416-1012)	507 (440-560)
Kanuti	6	2/10/98	12 (4-32)	B	1149 (992-1416)	574 (546-635)
Tetlin	11	Various[a]	32 (4-48)	AB	1155 (536-2356)	532 (425-660)
Yukon Flats	3	8/1/98	57 (53-61)	A	3455 (3346-3600)	830 (790-870)[b]

[a] Dates of collection were: 4/30/98 - 3 fish, 5/1/98 - 1 fish, 5/8/98 - 2 fish, 5 /10/98 - 3 fish, and 7/22/98 - 2 fish.
[b] n = 2.

Median, range, standard deviation, and number of detections per total number of samples of organochlorine compounds in burbot liver are listed in Table 4. Aroclor 1248 was not detected at any site, endrin and gamma chlordane were detected in one sample each, and gamma-BHC was detected in seven samples. Concentrations of these analytes were not statistically analyzed.

All data are expressed as non-lipid-normalized, wet weight. The greatest concentrations

Table 4. Median, range and standard deviations of organochlorine concentrations, and number of detections per total number of samples in burbot livers from interior Alaska, 1998. Concentrations of median and range are mg/kg wet weight.

Site Statistic	alpha-BHC	alpha-chlordane	dieldrin	HCB	heptachlor epoxide	mirex	o,p'-DDD	o,p'-DDE	o,p'-DDT
Fairbanks									
Median		0.0063	0.0030	0.015	0.0014	0.0016	0.016	0.0011	0.018
Range	<0.002-0.0043	0.0028-0.015	0.0014-0.0056	0.0043-0.019	<0.00032-0.0025	0.0010-0.0055	0.0054-0.067	0.00079-0.0036	0.005-0.077
Std Dev		0.0042	0.0014	0.0052	0.0007	0.0014	0.0200	0.0010	0.025
n<LOD /total n[a]	5/9	0/9	0/9	0/9	1/9	0/9	0/9	0/9	0/9
Kanuti									
Median				0.0014		0.0004			
Range	<0.002	<0.00046	<0.00026-0.0028	0.0009-0.0037	<0.00032	<0.00035-0.0013	<0.00008-0.00046	<0.00045	<0.00045-0.0011
Std Dev				0.001		0.0004			
n<LOD /total n	6/6	6/6	5/6	0/6	6/6	1/6	5/6	6/6	3/6
Tetlin									
Median			0.0006	0.0024	0.0005	0.0004			
Range	<0.002-0.0052	<0.00046-0.0008	<0.00026-0.0012	0.0011-0.0071	<0.00032-0.0010	<0.00035-0.0007	<0.0004	<0.00045	<0.00045-0.0006
Std Dev			0.0003	0.0016	0.0003	0.0002			
n<LOD /total n	6/11	6/11	2/11	0/11	1/11	5/11	11/11	6/11	8/11
Yukon Flats									
Median	0.0026	0.016	0.008	0.031	0.0032	0.0046	0.0018	0.0006	0.0060
Range	0.0026-0.0027	0.0046-0.021	0.0033-0.014	0.023-0.045	0.0018-0.0049	0.0015-0.0074	0.00075-0.0056	<0.00045-0.0012	0.0019-0.0080
Std Dev	0.0001	0.008	0.005	0.011	0.0016	0.0030	0.0026	0.0005	0.0031
n<LOD /total n	0/3	0/3	0/3	0/3	0/3	0/3	0/3	1/3	0/3

Table 4 Cont.

Site Statistic	p,p'-DDD	p,p'-DDE	p,p'-DDT	Aroclor 1242	Aroclor 1254	Aroclor 1260	PCB-TOTAL	toxaphene	trans-nonachlor
Fairbanks									
Median	0.13	0.099	0.093	0.017	0.25	0.20	0.54	0.14	0.015
Range	0.076-0.440	0.0012-0.470	0.030-0.290	0.0081-0.024	0.140-0.580	0.092-0.840	0.27-1.40	0.024-0.20	0.0064-0.081
Std Dev	0.117	0.115	0.090	0.005	0.14	0.247	0.37	0.068	0.025
n<LOD/total n	0/9	0/9	0/9	0/9	0/9	0/9	0/9	0/9	0/9
Kanuti									
Median	0.0026	0.0064							
Range	<0.0052-0.0068	0.0012-0.0440	<0.00075-0.0068	<0.0068	<0.0062-0.076	<0.0058-0.0310	<0.016-0.110	<0.0029-0.018	<0.0015-0.012
Std Dev	0.0025	0.0165							
n<LOD/total n	2/6	0/6	3/6	6/6	3/6	3/6	3/6	3/6	4/6
Tetlin									
Median		0.0084	0.0010		0.0077	0.0081		0.012	0.0018
Range	<0.00052-0.00130	0.0014-0.0280	<0.00075-0.0014	<0.0068	<0.0062-0.0360	<0.0058-0.1500	<0.0016-0.1700	<0.0029-0.0270	<0.0015-0.0038
Std Dev		0.008	0.0004		0.0096	0.432		0.0083	0.001
n<LOD/total n	6/11	0/11	3/11	11/11	3/11	4/11	7/11	1/11	4/11
Yukon Flats									
Median	0.0250	0.140	0.0150	0.026	0.310	0.085	0.42	0.290	0.063
Range	0.0078-0.0460	0.041-0.290	0.0065-0.017	0.014-0.041	0.110-0.710	0.032-0.200	0.16-0.95	0.087-0.420	0.019-0.140
Std Dev	0.019	0.125	0.006	0.014	0.306	0.085	0.40	0.167	0.061
n<LOD/total n	0/3	0/3	0/3	0/3	0/3	0/3	0/3	0/3	0/3

[a] The number of samples which had values less than the limit of detection/the total number of samples.

among cyclodiene insecticides, which include alpha-chlordane, dieldrin, endrin, gamma-chlordane, heptachlor epoxide, and oxychlordane, were 0.021 mg/kg alpha-chlordane and 0.019 mg/kg oxychlordane at Yukon Flats Refuge. Only one detection of cyclodiene insecticides occurred at the Kanuti Refuge and, except for the sole detection of gamma-chlordane at Fairbanks, all of the greatest concentrations of individual cyclodiene insecticides were at Yukon Flats. Alpha-chlordane had the greatest concentration of any cyclodiene insecticide, with mean concentrations ranging from <0.00046 mg/kg at Kanuti and Tetlin refuges to 0.014 mg/kg at Yukon Flats Refuge.

Among DDT and its metabolites, the greatest concentrations measured were 0.47 mg/kg p,p'-DDE and 0.44 mg/kg p,p'-DDD, at Fairbanks. All of the greatest concentrations of individual DDT compounds and its metabolites were found in samples from Fairbanks. Mean concentrations of p,p'-DDE ranged from 0.011 mg/kg at Kanuti to 0.20 mg/kg at Fairbanks. The greatest concentrations among PCB Aroclors were 0.84 mg/kg Aroclor 1260 at Fairbanks and 0.71 mg/kg Aroclor 1254 at Yukon Flats. HCB and toxaphene were detected in concentrations up to 0.045 mg/kg and 0.42 mg/kg, respectively, both in samples from Yukon Flats. Mean concentrations of toxaphene ranged from 0.0057 mg/kg at Kanuti to 0.27 mg/kg at Yukon Flats. Individual sample data are listed in Appendix A.

At Kanuti, where both males and females were captured, three gravid female burbot had lesser concentrations of HCB, mirex, o,p'-DDT, p,p'-DDD, p,p'-DDE, p,p'-DDT, Aroclor 1254, Aroclor 1260, ∑PCB, toxaphene, and trans-nonachlor than the two males and a non-gravid female. Exceptions were that p,p'-DDE and trans-nonachlor were not detected in the non-gravid female and one male, respectively, similar to the gravid females.

Analytes that had ≥ 50% of data below the LOD in two or more sites included alpha-chlordane, o,p'-DDD, o,p'-DDE, o,p'-DDT, oxychlordane, PCB 1242, and total PCBs. These analytes were significantly lower at Kanuti and Tetlin than at Fairbanks and Yukon Flats ($P < 0.001$ to $= 0.021$) (Table 5). Additionally, concentrations of o,p'-DDD and o,p'-DDE were significantly greater at Fairbanks compared to Yukon Flats ($P = 0.014$ and $P = 0.026$, respectively) (Table 5). The analysis for o,p'-DDE was heavily influenced by an outlier at Yukon Flats, which when removed resulted in no significant difference between Fairbanks and Yukon Flats.

Analytes with ≥ 50% of data below the LOD at only one site included p,p'-DDD (at Tetlin), dieldrin, heptachlor epoxide, p,p'-DDT, Aroclor 1254, Aroclor 1260, toxaphene, and trans-nonachlor (at Kanuti). Significant differences occurred among sites in concentrations of p,p'-DDD ($F_{2,14} = 41.5$, $P < 0.001$) in the order Fairbanks > Yukon Flats > Kanuti (all $P < 0.019$) (Figure 2). Lipid and fish weight were not significantly correlated with p,p'-DDD (both $P > 0.05$).

Analytes with ≥ 50% of data below the LOD at Kanuti (dieldrin, heptachlor epoxide, p,p'-DDT, Aroclor 1254, Aroclor 1260, toxaphene, and trans-nonachlor) were reduced to the first

Table 5. Significant differences in concentrations of selected organochlorine pesticides in burbot livers among four sites in Alaska determined using ANOVAs with ranked data. Unlike letters indicate significant differences in rank sums (all $P < 0.05$).

Site	alpha-chlordane	o,p'-DDD	o,p'-DDE	o,p'-DDT	oxychlordane	PCB 1242	Total PCBs
Fairbanks	A	A	A	A	A	A	A
Kanuti	B	B	B	B	B	B	B
Tetlin	B	B	B	B	B	B	B
Yukon Flats	A	C	C	A	A	A	A

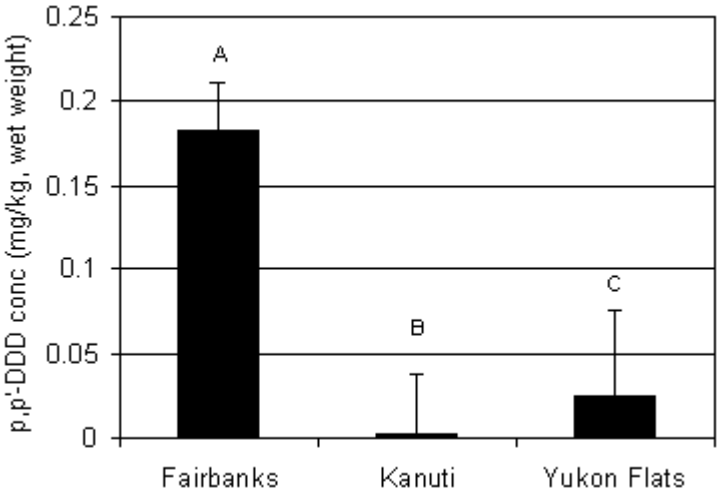

Figure 2. Means (+ standard error) for p,p'-DDD measured in burbot livers from three sites in interior Alaska, 1998. A fourth site, Tetlin, was measured but had $\geq$ 50% of data below the detection limit, so was not included in the analysis. Unlike superscripts indicate significant differences among sites (ANOVA with Scheffe post-hoc comparisons, all $P < 0.001$).

two PCs, which together accounted for 94% of the total variance (eigenvalues = 4.58 and 2.02, respectively). Component loadings indicated that dieldrin, heptachlor epoxide, Aroclor 1254, toxaphene and trans-nonachlor contributed to the first PC, while p,p'-DDT and Aroclor 1260 contributed to the second PC. Because the first and second PCs were orthogonal, the first PC was positively correlated with the contaminants that contributed to it, and the second PC was negatively correlated with contaminants that contributed to it. This means that higher contaminant concentrations were represented by higher factor scores from the first PC and

lower factor scores from the second PC. There were significant differences among sites for both PCs, indicated by significant multivariate statistics (Table 6). The first PC was significantly different among sites, in the order Yukon Flats > Fairbanks > Tetlin (Scheffe post-hoc comparisons, all $P < 0.001$). As a group, therefore, dieldrin, heptachlor epoxide, Aroclor 1254, toxaphene, and trans-nonachlor concentrations were higher at Yukon Flats than at Fairbanks and Tetlin, and higher at Fairbanks than at Tetlin (Figure 3). The second PC was greater at Tetlin compared to Fairbanks, but Yukon Flats was not significantly different from the other sites. Removal of an influential outlier resulted in significantly greater scores at Yukon Flats versus Fairbanks. As a group, therefore, p,p'-DDT and Aroclor 1260 concentrations were greater at Fairbanks compared to Tetlin and possibly compared to Yukon Flats, but there were no significant differences between concentrations at Tetlin and Yukon Flats (Figure 4).

The percent lipid in samples confounded this analysis. Lipid was a significant covariate, indicated by significant multivariate statistics (Table 6), but for the first PC only, reflecting the significant correlation of percent lipid with dieldrin and heptachlor epoxide. The site*lipid interaction was significant, indicated by significant multivariate statistics (Table 6), corroborating that percent lipid differed among sites (Table 3). Because the same contaminants were correlated with lipid, and because differences occurred among sites in percent lipid, differences among sites in contaminant concentrations could be due to differences in percent lipid, in addition to differences in lipid-adjusted contaminant concentrations.

HCB, mirex, and p,p'-DDE were detected in > 50% of samples at all four sites. There were significant differences among sites in contaminant concentrations, indicated by significant multivariate statistics (Table 7). All three contaminants were significantly different among sites, with concentrations at Fairbanks and Yukon Flats significantly greater than at Tetlin and Kanuti (Scheffe post-hoc comparisons, all $P < 0.001$) (Figure 4). Lipid was a significant covariate for HCB, indicated by significant multivariate test statistics (Table 7) reflecting HCB's significant correlation with percent lipid. The site*lipid factor was not significant (Table 7).

Table 6. Results of two-way multivariate ANOVA that tested whether organochlorine contaminant concentrations in burbot livers differed among three sample sites in interior Alaska. Factor scores from the first and second principal components (PCs) (derived from correlated contaminant concentrations) were significantly different among sites. Percent lipid was a significant covariate for the first PC, but not for the second, and the significant site*lipid interaction suggests differences in percent lipid among sites.

Factor, Covariate, or Interaction	Multivariate Statistics	Response Variables	Univariate Statistics
Site (Fairbanks, Tetlin, Yukon Flats)	Wilke's $\lambda = 0.046$ $F = 29.5$ $P < 0.001$	First PC factor scores (from dielrin, heptachlor epoxide, Aroclor 1254, toxaphene, and trans-nonachlor)	$F_{2, 17} = 84.9$, $P < 0.001$
		Second PC factor scores (from p,p-DDT and Aroclor 1260)	$F_{2, 17} = 7.9$, $P = 0.004$
Lipid	Wilke's $\lambda = 0.133$ $F = 52.3$ $P < 0.001$	First PC factor scores	$F_{1, 17} = 107.7$, $P < 0.001$
		Second PC factor scores	$F_{1, 17} = 0.26$, $P = 0.620$
Site*Lipid	Wilke's $\lambda = 0.094$ $F = 18.1$ $P < 0.001$	First PC factor scores	$F_{2, 17} = 53.2$, $P < 0.001$
		Second PC factor scores	$F_{2, 17} = 3.6$, $P = 0.050$

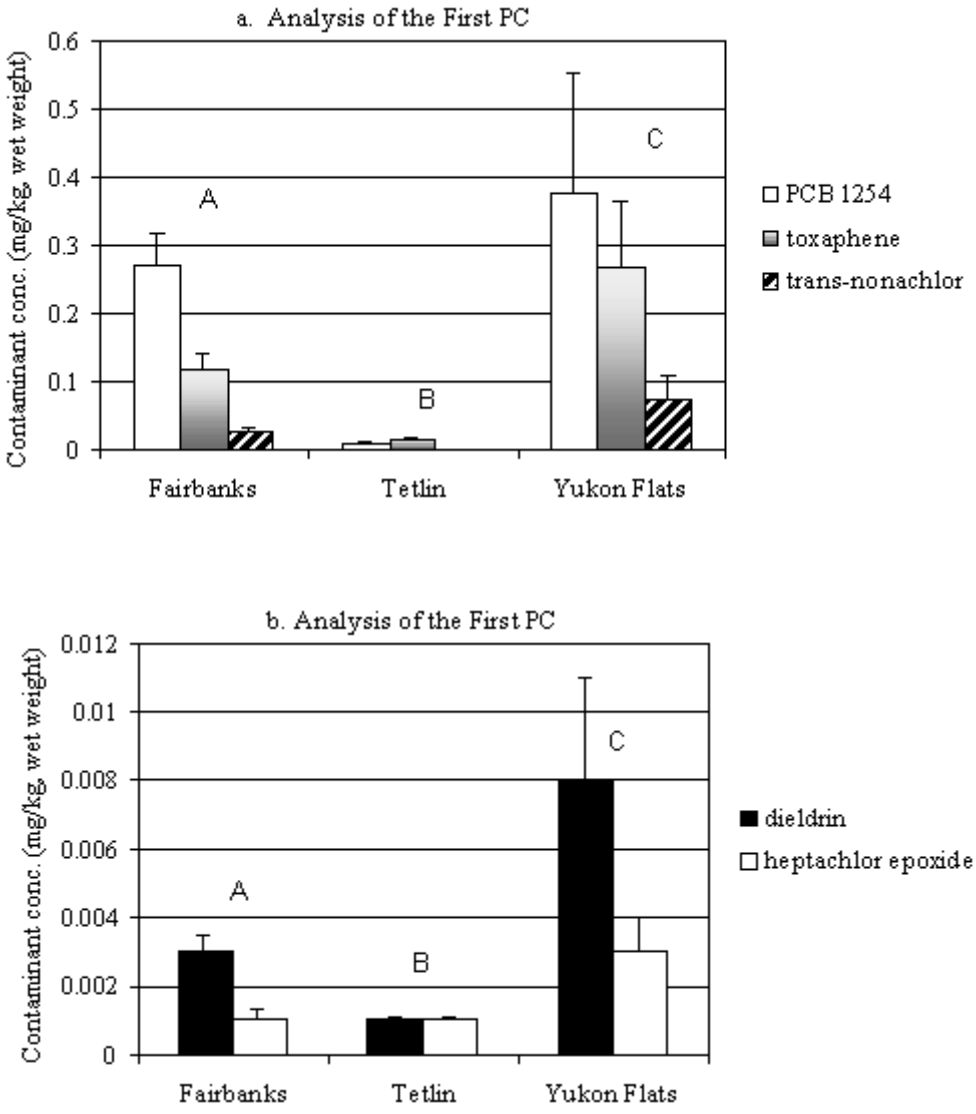

Figure. 3. Results of principal components (PC) analysis of organochlorine compounds in burbot *(Lota lota*) liver from Fairbanks, and Tetlin and Yukon Flats NWR, Alaska, 1998. Analytes shown were compared among sites after being reduced to the first (a and b) and second (c) PC. Means (+ standard error) are shown for comparison purposes. Unlike superscripts indicate significant differences among sites in PC factor scores (ANOVA with Scheffe post-hoc comparisons, all $P < 0.05$).

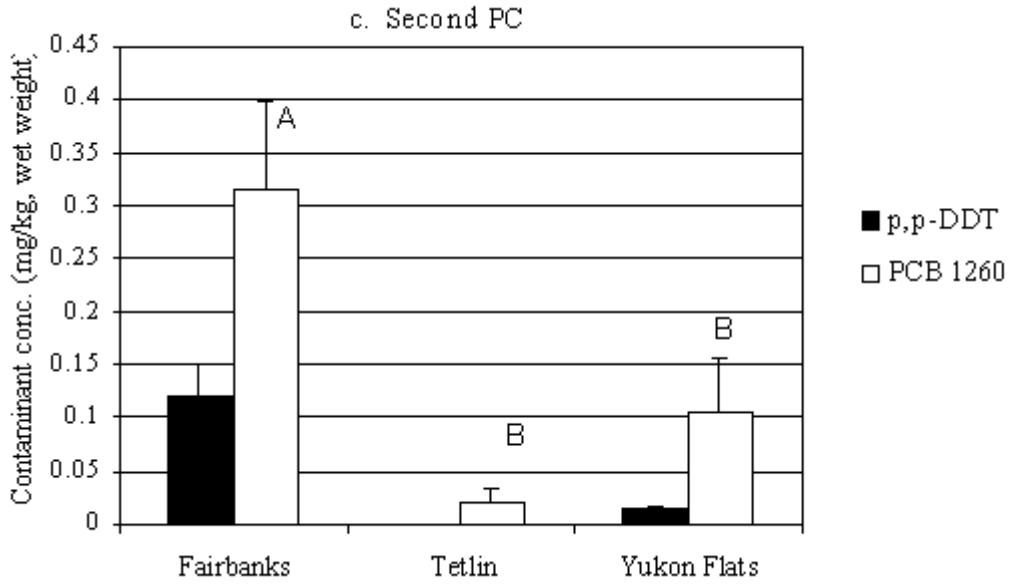

Figure 3. Continued. Results of principal components (PC) analysis of organochlorine compounds in burbot (*Lota lota*) liver from Fairbanks, and Tetlin and Yukon Flats NWR, Alaska, 1998. Analytes shown were compared among sites after being reduced to the first (a and b) and second (c) PC. Means (+ standard error) are shown for comparison purposes. Unlike superscripts indicate significant differences among sites in PC factor scores (ANOVA with Scheffe post-hoc comparisons, all $P < 0.05$).

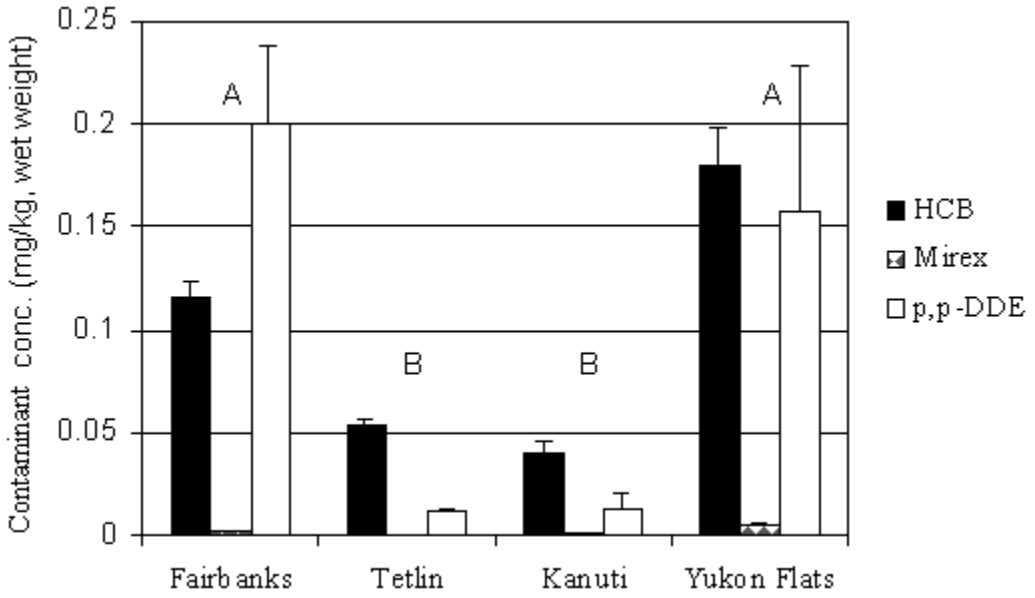

Figure 4. Means (+ standard error) for HCB, mirex, and p,p'-DDE measured in burbot livers from four sites in interior Alaska, 1998. Unlike superscripts indicate significant differences among sites for all three contaminants (MANCOVA, lipid as covariate, Scheffe post-hoc comparison, all P
< 0.012).

Table 7. Results of two-way multivariate ANOVA that tested whether organochlorine contaminant concentrations in burbot livers differed among four interior Alaska sample sites. Contaminant concentrations were significantly different among sites. Percent lipid was a significant covariate, but only for HCB.

Factor, Covariate, or Interaction	Multivariate Statistics	Response Variables	Univariate Statistics
Site (Fairbanks, Tetlin, Kanuti, Yukon Flats)	Wilke's $\lambda = 0.17$ $F = 6.5$ $P < 0.001$	Mirex HCB p,p'-DDE	$F = 13.8$, $P < 0.001$ $F = 22.1$, $P < 0.001$ $F = 16.5$, $P < 0.001$
Lipid	Wilke's $\lambda = 0.50$ $F = 7.4$ $P = 0.001$	Mirex HCB p,p'-DDE	$F = 3.3$, $P = 0.084$ $F = 8.7$, $P = 0.007$ $F = 0.041$, $P = 0.842$
Weight	Wilke's $\lambda = 0.93$ $F = 0.4$ $P = 0.769$	Mirex HCB p,p'-DDE	Not Applicable
Site*Lipid	Wilke's $\lambda = 0.63$ $F = 0.85$ $P = 0.574$	Mirex HCB p,p'-DDE	Not Applicable
Site*Weight	Wilke's $\lambda = 0.84$ $F = 0.31$ $P = 0.965$	Mirex HCB p,p'-DDE	Not Applicable

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DISCUSSION

Organochlorines are semi-volatile compounds. Semi-volatile compounds can cycle between gaseous and condensed phases in the environment, thus enabling them to be transported throughout the globe by air currents and recondense great distances from their point of release (Barrie et al. 1997). Long-range transport of organochlorines to Alaska and northern Canada and their subsequent incorporation into biota has been documented (Bidleman et al. 1989, Patton et al. 1989, Muir et al. 1990a, Wilson et al. 1995, Barrie et al. 1997, Ewald et al. 1998). The general direction of transport of these compounds is towards polar regions (Muir et al. 1995) because semi-volatile organic compounds that volatilize in warmer areas and condense in colder climatic zones fail to revolatilize in cold conditions and thus remain. Because it is caused by the temperature-driven differential volatility of these compounds, the phenomenon of "global fractionation/cold condensation" is compound-specific (Wania and Mackay 1993).

Most organochlorine contaminants have not been used in arctic and subarctic areas of North America. Notable exceptions to this are the pesticide DDT and the industrial chemicals PCBs, which have been used at many sites throughout northern Canada and Alaska. Organochlorine contaminants occur in Arctic and subarctic ecosystems due to one or more of three possible source routes: aerial deposition, transport in biota, and local use of the compounds. Once deposited, conditions in arctic areas enhance long-term accumulation of semi-volatile lipophilic organochlorine compounds in several ways: 1. low arctic temperatures lead to partitioning of organochlorine compounds to aerosols and rapid deposition, 2. low annual primary productivity provides a limited sink for contaminants, leading to increased concentrations in biota, 3. low ambient temperatures decrease organochlorine degradation rates, 4. fat-based trophic energy transfer in arctic food webs enhances the transfer of these lipophilic contaminants through the food chain, and 5. the slow growth rates and longevity of arctic freshwater fish increase the likelihood of higher contaminant burdens relative to similar species in temperate climates (Allen-Gil et al. 1997).

Ewald et al. (1998) reported that biotransport of PCBs and DDT in sockeye salmon (*Oncorhynchus nerka*) had a far greater influence on biota in an Alaskan lake than atmospheric input. They compared concentrations of organochlorines in Arctic grayling from two lakes, one impacted by sockeye salmon returning from the sea and one unimpacted by salmon, which received organochlorines only from atmospheric sources. Arctic grayling from the lake impacted by salmon had significantly greater concentrations of PCBs and DDT than those from the lake unimpacted by salmon. Organochlorines introduced by salmon are more readily available for bioaccumulation than those from atmospheric sources (Ewald et al. 1998). Migrating salmon, roe, and carcasses are fed upon directly by predators and thus pollutants are transferred to biota in a direct and efficient manner. Atmospherically deposited pollutants are subject to various abiotic processes prior to possible bioaccumulation (de March et al. 1998).

In this study, general patterns of greater organochlorine contamination at Yukon Flats and Fairbanks compared to Tetlin and Kanuti were consistent, with significant differences in concentrations for some analytes. However, clear differences in contaminant concentrations were confounded by differing lipid concentrations among sites, and a low (n = 3) sample size at Yukon Flats. Also, based on radio telemetry and tagging studies, the Alaska Department of Fish and Game has observed that some burbot move as far upstream on the Tanana River from Fairbanks as Northway (Tetlin) (Evenson 1993).

Concentrations of o,p'-DDT, o,p'-DDE, and p,p'-DDD were significantly greater at Fairbanks than at the other three sites. PCB concentrations were also greatest at Fairbanks although one sample from Yukon Flats had 0.95 mg/kg ∑PCBs, the second greatest concentration we measured. DDT and PCBs have been used at Ft. Wainwright Army Base and Eielson Air Force Base, near Fairbanks, and in and around the city of Fairbanks (R. Markey, Alaska Department of Environmental Conservation, pers. comm., USAF 1995, Harding Lawson Associates 1997). All of these sites are upstream of the capture sites and likely serve as local sources of these contaminants to the Tanana River. The liver sample of one fish from Yukon Flats was normal in color but fluid in consistency instead of firm as were all of the other livers sampled. This liver had the greatest concentrations, for all samples, of alpha chlordane, dieldrin, endrin, HCB, heptachlor epoxide, mirex, Aroclor 1242, Aroclor 1254, toxaphene, and trans-nonachlor. It was also the sample that had 0.95 mg/kg ∑PCB. This was a large, lipid-rich fish which can result in higher organochlorine concentrations, but reasons for the high concentrations in this fish are unknown. Health and Welfare Canada uses a concentration of 0.100 mg/kg as a fish consumption guideline for chlordane, dieldrin, and heptachlor epoxide (McCarthy et al. 1997). Concentrations of these compounds in burbot livers at Yukon Flats were more than an order of magnitude less than this guideline.

Inter-lake variability in concentrations of PCBs (Rasmussen et al. 1990), toxaphene (Kidd et al. 1995a) and other organochlorines (Kidd et al. 1995b, Kidd et al. 1998) has been shown to be due to the trophic level of the sampled organisms, i.e., biomagnification. Rasmussen et al. (1990) reported that concentrations of PCBs in lake trout increased about 3.5-fold and lipid content increased by a factor of 1.5 at each trophic level going up the food chain. Kidd et al. (1995b) measured the trophic level of aquatic organisms by measuring stable isotopes of nitrogen, $\delta^{15}N$. The lowest trophic level had an $\delta^{15}N$ of 1.3 - 5.0 parts per thousand, the next level had 5.8 - 9.9 parts per thousand, and top predators, lake trout and burbot, had 11.5 and 11.6 parts per thousand, respectively. They also noted that $\delta^{15}N$, i.e., trophic level, explained more of the variation in contaminant concentrations than percent lipid. An outstanding example of the length-of-food-chain phenomenon occurred at Lake Laberge, Yukon Territory. Organochlorine concentrations in burbot livers from Lake Laberge were greater than for all other studies listed in Table 8. Top predators, including burbot, feed at a higher trophic level at Lake Laberge than at other geographically similar lakes, which resulted in greater concentrations of o,p'-DDD, o,p'-DDE, o,p'-DDT, p,p'-DDD, p,p'-DDE and p,p'-DDT (∑ DDT), ∑ PCBs, and toxaphene (Kidd et al. 1995a). Contents of burbot stomachs from our study included fish, dragonfly nymphs (Order Odonata) (solely from Site TE20 on the Tetlin

Refuge), unidentifiable organic contents, and rocks. A high percentage of empty stomachs in sampled fish precludes our making conclusions regarding site-specific food habits. However, food chain effects could be a factor in the observed differences in organochlorine concentrations among sites.

Significant differences in lipid concentrations occurred among sites during this study; in increasing order, they were Kanuti< Tetlin< Fairbanks< Yukon Flats. These differences likely relate to time of capture although other factors may also be involved. The liver is the main somatic lipid reserve for burbot; burbot have no intestinal lipid in the mesentery and the lipid content of muscle is low. Burbot have low concentrations of organochlorines in muscle (Muir et al. 1997). Compared to the Salmonidae or Clupeidea, lipid deposition in the Gadidae, of which burbot is a member, is more strictly concentrated in the liver (Love 1980). Because their energy inputs and requirements vary throughout the year, lipid concentrations in burbot liver vary seasonally. Pulliainen and Korhonen (1990) conducted a study of 1052 burbot from Finland centering on various condition indices, including one for liver weight. They reported that livers weighed the most, i.e., contained the most lipid, during July for females and June for males. Liver weights dropped dramatically during the onset of gonadal development which began in September for males and October for females, and were the lowest for both sexes during October. Liver weights increased from October until another decrease during spawning in February and March. Tanana River burbot at Fairbanks spawn during the last week of January or the first week of February (Evenson 1993). Burbot from Kanuti were collected at or just after spawning; burbot from Tetlin were collected a month or two later; and fish from Fairbanks and Yukon Flats were collected in early September and early August, respectively. The pattern established by the date of collection and lipid content of our burbot liver samples coincides with the pattern described by Pulliainen and Korhonen (1990).

Relationships among organochlorine concentrations and the weight, length, age, and lipid concentrations of fish varied among studies. Weight, length, or age have been shown to be significant predictors of organochlorine concentrations in salmonids (Rasmussen et al. 1990) and burbot liver (Muir et al. 1990b). Kidd et al. (1998) reported that concentrations of lipid in burbot liver, and size and age of burbot were not correlated with organochlorine concentrations, and Kidd et al. (1993) reported that concentrations of toxaphene (liver log ∑CHB) were not significantly correlated with lipid, age, or length or weight of fish. Muir et al. (1990a, 1990b) also observed that toxaphene levels were not significantly correlated with age or weight of burbot. This lack of age/size effect has been observed for toxaphene and other more water-soluble organochlorines in lake trout, but not for PCBs and DDE (Muir et al. 1990a). At Schrader Lake, in arctic Alaska, samples from a population of lake trout and Arctic grayling showed no significant correlations between organochlorine concentrations and fish weight or length (Wilson et al. 1995). In our study, lipid and fish weight were significantly correlated with several contaminants. Kidd et al. (1998) also found significant differences among sample sites in organochlorine and lipid concentrations. However, similar to our study, when they used lipid as a covariate, it did not remove among-lake differences in

Table 8. Mean concentrations of organochlorine pesticides and PCBs in burbot (*Lota lota*), arctic grayling (*Thymallus arcticus*), and longnose sucker (*Catastomus catastomus*) from published studies in Alaska, Canada, Lapland, and this study from the Koyukuk, Tanana, and Yukon Rivers in interior Alaska. Units are mg/kg wet weight, except where noted.

Location	Species (matrix)	n	ΣDDT[a]	Toxaphene	Mirex	Dieldrin	ΣPCBs	Reference
Fairbanks, AK Chena River	Burbot (whole)	1[b]	0.16[c]	<0.01		<0.01	0.40[d]	Schmitt et al. 1990
	Grayling (whole)	8[b]	0.11-1.23[c]	<0.01	<0.01	<0.01	<0.01[e]-5.30[f]	Schmitt et al. 1990
	Longnose sucker (whole)	12[b]	0.03-1.16[c]	<0.01	<0.01	<0.01-0.01	<0.10[e]3.87[f]	Schmitt et al. 1990
Yukon, Canada Lake Laberge	Burbot (liver)	35	3.43	2.30		1.27	3.43	Palmer 1992, '93, '94
Ft. Smith, Northwest Territories, Canada Slave R. [g]	Burbot (liver)	10-11	0.021-0.077	0.15-0.88	<0.002-0.010	0.008-0.014	0.039-0.058[h], 0.51[i]	McCarthy et al. 1997
Northwest Territories, Canada Slave Lake	Burbot (liver)		0.027-0.051	0.24-0.76		0.005-0.010	0.075-0.14	Evans 1996
Northern Canada								
2 lakes	Burbot (liver)	5, 11	0.0004-0.040	0.0022-0.17			0.0019-0.057	Muir and Lockhart 1996
3 lakes	Burbot (liver)	5 - 29 2 - 8	0.014-0.039	0.041-0.37			0.027-0.11	Muir and Lockhart 1994, 1996
14 lakes/rivers	Burbot (liver)		0.021-0.27	0.054-1.53			0.050-0.58	Muir and Lockhart 1994
Lapland Lake Pahtajarvi	Burbot (liver)	4	0.025			<0.01		Palmer 1992, '93, '94
Alaska Interior Rivers	Burbot (liver)	3 - 11	0.013-0.55	0.0057-0.12	0.0005-0.0045	0.00016-0.0084	0.031-0.60[d]	This study

Table 8. Cont.

Location	Species (matrix)	n	ΣDDT[a]	Toxaphene	Mirex	Dieldrin	ΣPCBs	Reference
Lipid Weight (Ratio Method)								
Canada[j] 8 lakes	Burbot (liver)		0.051-1.49	0.81-2.34	0.0037-0.017	0.0071-0.071	0.30-1.94	Muir et al. 1990a
Alaska[j] Interior Rivers	Burbot (liver)	3 - 11	0.18-2.76	0.053-0.60	0.0024-0.014	0.0016-0.015	0.10-3.70[d]	This study

[a] Sum of o,p'-DDD, o,p'-DDE, o,p'-DDT, p,p'-DDD, p,p'-DDE, and p,p'-DDT.
[b] Composite of five fish per sample.
[c] Sum of p,p'-DDD, p,p'-DDE, and p,p'-DDT.
[d] Sum of Aroclors 1242, 1248, 1254, and 1260.
[e] Sum of Aroclors 1248, 1254, and 1260.
[f] Aroclor 1254 only.
[g] Median values.
[h] Sum of seven non-coplanar congeners.
[i] Sum of coplanar PCBs-77, 81, 105, and 169.
[j] Lipid-normalized (ratio method) concentrations; calculated in this study for comparative purposes only.

organochlorine concentrations.

Differences among sexes due to female transfer of lipophilic compounds to young in mammals has been documented (Wade et al. 1997). At Kanuti, three gravid female burbot had lesser concentrations of numerous organochlorines than two males and one non-gravid female. Although the sample size is very small, this may indicate that female burbot eliminate organochlorine compounds into gametic products and to a greater extent than males. Muir et al. (1990b) reported no significant difference in organochlorine concentrations in 68 male and female burbot livers. However, the date of collection and reproductive condition of their fish were not reported. Ewald et al. (1998) observed that as sockeye salmon migrated up the Copper River, Alaska towards their spawning lake, the lipid content of muscle decreased from 5.5% to 2.2% as lipids were reallocated from muscle to female gonads; comparable data were not provided for male fish. During that time, concentrations of DDT and PCBs in muscle lipid increased ≈550% and ≈370%, respectively, due to a decrease in the solving volume (Addison 1982), and concentrations of both compounds in roe lipids approximately doubled. Organochlorines in roe were then eliminated from the fish during spawning. Loss of organochlorines during reproductive activities also occurs in mammals. Male beluga whales (*Delphinapterus leucas*) from the north coast of Alaska have been shown to have higher concentrations of several organochlorines than females (Wade et al. 1997). Transplacental transfer to the fetus and through lactation to the nursing pups are the most probable causes of lower organochlorine concentrations in females. Female belugas have exhibited a decrease in concentrations with age likely due to continual reproductive success (Wade et al. 1997).

The lower concentrations of ∑DDT from our study are similar to those from five of the seven Canadian studies and Lapland (Table 8). ∑DDT concentrations in two lakes in northern Canada (Evans 1996, Muir and Lockhart 1996) are nearly two orders of magnitude less than ours. The greatest values of ∑DDT in our study, fish caught at Fairbanks, are greater than from all other studies listed in Table 8, except Lake Laberge. The greatest values of ∑DDT from other sites in our study are similar to those from the Canadian, except Lake Laberge, and Lapland sites. One study each on lake trout and brook trout (*Salvelinus fontinalis*) showed, in general, that DDE with whole body concentrations of 0.042 mg/kg and 2.68 mg/kg had no effect on survival or growth, respectively (Berlin et al. 1981, Wang and Simpson 1996). However, Berlin et al. (1981) reported reduced survival with whole body concentrations of DDE of 0.29 mg/kg. Whole body concentrations are generally lower than liver concentrations, so the results of these studies should be considered conservative when compared to liver data, as from our study.

The presence of PCBs in fish have been documented since the early 1970s (Bidleman et al. 1990). The range of ∑PCB concentrations from our study are similar to those from five of the seven Canadian studies cited in Table 8, with the range of concentrations from two lakes in northern Canada (Evans 1996, Muir and Lockhart 1996) generally lower than in our study (Table 8). However, mean ∑PCBs from Lake Laberge is more than twice as great as our

greatest value of 1.40 mg/kg in a burbot from Fairbanks. Mayer et al. (1977) reported a lethal body burden for rainbow trout (*Oncorhynchus mykiss*) for Aroclor 1254 of 640 mg/kg. Whole body residues of 0.4 mg/kg PCBs have been associated with reproductive toxicity in rainbow trout (EPA 1980). Liver histology of rainbow trout was not affected by Aroclor 1254 concentrations of 8 mg/kg PCBs (Lieb et al. 1974), but liver ultrastructure of rainbow trout was altered by Aroclor 1254 in concentrations as low as 2 mg/kg in liver (Hacking et al. 1977). Rainbow trout with 0.33 mg/kg whole body fresh weight of Aroclor 1254 incurred 10-28% prehatch mortality and numerous posthatch deformities (Eisler 1986). The greatest Aroclor 1254 value in burbot liver from our study was 0.71 mg/kg, but Aroclor 1254 concentrations were generally less than the effects concentrations cited. Threshold PCB concentrations at which adverse effects on growth and reproduction occur in laboratory studies, such as those cited, are generally greater than concentrations observed in natural systems (Eisler 1986).

Toxaphene concentrations in Canadian burbot were generally greater than in our study (Table 8), and one Canadian study (Muir et al. 1990a) reported that toxaphene was the major organochlorine detected in burbot liver. The presence of toxaphene in Arctic air, marine mammals, and fish suggest that it is a major organochlorine contaminant in northern latitudes (Muir et al. 1990b). Eisler and Jacknow (1985) stated that toxaphene residues of fish in excess of 0.4 to 0.6 mg/kg whole body may be hazardous to fish health and should be considered as presumptive evidence of significant environmental contamination. Because these are whole body analyses, these figures likely are not relevant to our data except as general guidance. Germany (de Geus et al. 1999) and Health and Welfare Canada (McCarthy et al. 1997) have established a maximum residue limit for toxaphene in fish for human consumption of 0.1 mg/kg.

Mirex concentrations from our study were almost identical to those reported by Muir et al. (1990a). Mirex concentrations were low; our greatest concentration was 0.0074 mg/kg in a burbot from Yukon Flats (Appendix A). Skea et al. (1981), in laboratory studies with brook trout, showed that whole body residues of 6.3 ppm fresh weight were not associated with adverse effects on growth or survival. Koenig (1977) reported that mixtures of DDT and mirex produced more than additive deleterious effects on fish survival and reproduction. Interaction effects of mirex with other anthropogenic contaminants are not well understood (Eisler 1985).

The National Contaminant Biomonitoring Program sampled fish in the Chena River, a tributary to the Tanana River, at Fairbanks from 1969 through 1986 (Schmitt et al. 1999). Each sample was composed of five whole body fish of the same species, including Arctic grayling and longnose sucker (*Catastomus catastomus*). These data are not directly comparable to ours because they are whole body samples and, unlike these species, burbot preferentially store lipids and, thus, lipophilic compounds such as organochlorines, in the liver. Other teleosts, such as Arctic grayling and longnose sucker, store lipid in muscle or in the coelomic cavity. This difference may result in interspecies comparisons involving burbot

to be less meaningful. However, the Chena River data are useful for documenting trends and as general indicators of contamination in the Chena River at Fairbanks. ∑DDT concentrations decreased from 1969 to1984 (Schmitt et al. 1999 and Pers. Comm. Christopher Schmitt USGS). After measurement of the greatest values in 1971 and 1972, concentrations declined approximately an order of magnitude by 1973 and remained low through 1986. A single burbot sample was collected in 1979 and its ∑DDT concentration (0.16 mg/kg) was similarly low. Aroclor 1254, the only PCB consistently measured, declined between the 1973 and 1977 sample periods and remained low thereafter. Similar to our study, Schmitt et al. (1999) reported Aroclor 1254 in greater concentrations than Aroclors 1242 or 1260. Concentrations of dieldrin, a degradation product of aldrin, decreased from 0.01 mg/kg to <0.01 mg/kg (LOD = 0.01 mg/kg) between 1971 and 1972 for Arctic grayling and longnose sucker, and remained below detection limits through 1986. Mirex and toxaphene concentrations were always less than their respective detection limits of 0.01 mg/kg or 0.005 mg/kg, and 0.1 mg/kg or 0.05 mg/kg, respectively.

In spite of these downward trends, however, livers of burbot collected at Fairbanks in our study showed organochlorine contamination. Further studies should be conducted to determine the extent of organochlorine contamination downstream of Fairbanks on the Tanana River using the Tetlin area as a control. Fish collected at Beaver on the Yukon River were also contaminated with organochlorines, but because of low sample size, this study should not be used to describe baseline conditions for Yukon River burbot. Further sampling of Yukon River burbot should be conducted to establish baseline conditions. In addition, organochlorine contamination should be determined at areas of known organochlorine use such as the U.S. Air Force facility at Galena, Alaska. In these studies, careful attention should be paid to confounding variables such as lipid percent, as affected by the reproductive state of the burbot, date of collection, and size and sex of fish sampled.

In conclusion, we measured organochlorine concentrations in livers of burbot collected from the Yukon, Koyukuk, and Tanana rivers in Alaska. In general, there were greater contaminant concentrations from sites below Fairbanks and Yukon Flats Refuge than at Tetlin and Kanuti refuges. Interpretation of these results was complicated by differing lipid concentrations in samples and differing fish weights among sites, and by a low sample size at Yukon Flats. However, conclusions regarding differences among sites, especially regarding Fairbanks, Tetlin and Kanuti, were robust across a variety of analyses. There were greater concentrations of DDT and its metabolites at Fairbanks, probably reflecting historical use of that pesticide within the city of Fairbanks and at nearby military bases. Concentrations of ∑DDT from Fairbanks are up to two orders of magnitude greater than in burbot from five studies in Canada. The range of ∑PCB concentrations from our study are similar to those from four of six Canadian studies (Table 8) and were generally less than laboratory-derived effects values. Toxaphene concentrations from our study were generally low. Further studies should help illuminate whether the concentrations we found at Fairbanks and Yukon Flats are of concern to fish and wildlife resources. Careful attention should be paid to confounding variables such as lipid percent, as affected by reproductive state and date of collection, and size and sex of

fish sampled.

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APPENDIX A: ORGANOCHLORINE CONCENTRATIONS IN BURBOT (*Lota Lota*) LIVERS COLLECTED FROM FAIRBANKS, ALASKA, AND KANUTI, TETLIN AND YUKON FLATS NATIONAL WILDLIFE REFUGES, 1998. ORGANOCHLORINE CONCENTRATIONS ARE MG/KG WW.

Site	Fish	Date	% Lipid	Weight (g)	Length (cm)	alpha-BHC	alpha-chlordane	dieldrin	endrin	gamma-BHC	gamma-chlordane	HCB	heptachlor epoxide
FA01	A	09/08/98	49	638	45.0	0.0043	0.0078	0.0038	<0.00068	<0.00260	0.0004	0.0150	0.0013
FA01	B	09/08/98	57	764	51.5	0.0041	0.0029	0.0021	<0.00068	0.0031	<0.00038	0.0110	0.0014
FA01	C	09/08/98	30	490	44.0	<0.002	0.0033	0.0030	<0.00068	0.0028	<0.00038	0.0078	0.0009
FA01	D	09/08/98	57	684	51.5	0.0040	0.0063	0.0056	<0.00068	<0.00260	<0.00038	0.0180	0.0025
FA01	E	09/08/98	6	1012	56.0	<0.002	0.0150	0.0014	<0.00068	<0.00260	<0.00038	0.0043	<0.00032
FA01	F	09/09/98	32	812	55.0	<0.002	0.0092	0.0039	<0.00068	<0.00260	<.00038	0.0180	0.0016
FA01	G	09/09/98	38	416	44.0	<.00200	0.0120	0.0052	<0.00068	<0.00260	<0.00038	0.0180	0.0020
FA01	H	09/09/98	47	934	53.0	<0.0020	0.0028	0.0020	<0.00068	<0.00260	<0.00038	0.0120	0.00037
FA01	I	09/09/98	43	956	56.5	0.002	0.0062	0.0030	<0.00068	<0.00260	<0.00038	0.0190	0.0012
KA01	A	02/10/98	32	992	54.6	<0.002	<0.00046	0.00028	<0.00068	<0.00260	<0.00038	0.0037	<0.00032
KA01	B	02/10/98	13	1046	56.8	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0019	<0.00032
KA01	C	02/10/98	8.6	1136	60.6	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0010	<0.00032
KA01	D	02/10/98	6.6	1168	55.8	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0015	<0.00032
KA01	E	02/10/98	9.6	1134	56.8	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0013	<0.00032
KA01	F	02/10/98	4.3	1416	63.5	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0009	<0.00032
TE09	A	04/30/98	30	1050	50.0	<0.002	<0.00046	0.00052	<0.00068	0.0031	<0.00038	0.0019	0.00032
TE09	B	04/30/98	29	1232	53.0	<0.002	<0.00046	<0.00026	<0.00068	0.0037	<0.00038	0.0011	<0.00032
TE09	C	04/30/98	48	2356	66.0	<0.002	0.00058	0.00047	<0.00068	<0.00260	<0.00038	0.0023	0.00036
TE09	D	05/01/98	47	1748	60.0	<0.002	0.00047	0.00056	<0.00068	<0.00260	<0.00038	0.0024	0.0004
TE13	A	05/08/98	4.4	1514	56.0	0.0025	<0.00046	0.0009	<0.00068	<0.00260	<0.00038	0.0031	0.00058
TE13	B	05/08/98	42	990	54.0	0.0021	<0.00046	0.0010	<0.00068	<0.00260	<0.00038	0.0034	0.00063
TE20	A	05/10/98	36	536	42.5	0.0021	0.00069	0.00088	<0.00068	0.0027	<0.00038	0.0034	0.00082
TE20	B	05/10/98	25	620	43.0	<0.002	<0.00046	0.0006	<0.00068	0.0026	<0.00038	0.0024	0.00051
TE20	C	05/10/98	14	874	54.0	<0.002	<0.00046	<0.00026	<0.00068	<0.00260	<0.00038	0.0016	0.00034
TE21	A	07/22/98	24	838	53.0	0.0025	0.00063	0.00086	<0.00068	<0.00260	<0.00038	0.0040	0.00079
TE21	B	07/22/98	48	946	53.5	0.0052	0.0008	0.0012	<0.00068	<0.00260	<0.00038	0.0071	0.0010
YF20	A	08/01/98	53	3420		0.0026	0.0210	0.0140	0.0012	<0.00260	<0.00038	0.0450	0.0049
YF20	B	08/01/98	61	3600	79.0	0.0026	0.0046	0.0033	<0.00068	<0.00260	<0.00038	0.0230	0.0018
YF20	C	08/01/98	58	3346	87.0	0.0027	0.0160	0.0080	<0.00068	0.0020	<0.00038	0.0310	0.0032

Appendix A, continued.

Site	Fish	mirex	o,p'-DDD	o,p'-DDE	o,p'-DDT	oxychlordane	p,p'-DDD	p,p'-DDE	p,p'-DDT	Aroclor 1242	Aroclor 1248	Aroclor 1254
FA01	A	0.0019	0.0220	0.0014	0.0190	<0.00490	0.210	0.140	0.093	0.017	<0.0068	0.180
FA01	B	0.0014	0.0300	0.00079	0.0052	0.0057	0.110	0.110	0.036	0.0081	<0.0068	0.140
FA01	C	0.0011	0.0380	0.0028	0.0580	<0.00490	0.290	0.230	0.200	0.012	<0.0068	0.280
FA01	D	0.0016	0.0054	0.00081	0.0082	0.0055	0.080	0.120	0.049	0.015	<0.0068	0.140
FA01	E	0.0055	0.0067	0.00097	0.0210	0.0160	0.190	0.470	0.190	0.0084	<0.0068	0.580
FA01	F	0.0021	0.0140	0.0011	0.0180	0.0080	0.130	0.220	0.140	0.017	<0.0068	0.250
FA01	G	0.0027	0.0088	0.0009	0.0085	0.0140	0.110	0.170	0.047	0.020	<0.0068	0.350
FA01	H	0.00096	0.0670	0.0036	0.0770	<0.00490	0.440	0.240	0.290	0.024	<0.0068	0.350
FA01	I	0.0016	0.0160	0.0012	0.0110	0.0061	0.076	0.099	0.042	0.017	<0.0068	0.160
KA01	A	0.00049	<0.0004	<0.00045	0.00047	<0.00490	<0.00052	0.011	0.0021	<0.0068	<0.0068	0.012
KA01	B	0.0013	<0.0004	<0.00045	0.0011	<0.00490	0.0068	0.044	0.0068	<0.0068	<0.0068	0.076
KA01	C	0.00037	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0012	<0.00075	<0.0068	<0.0068	<0.0062
KA01	D	0.0005	0.00046	<0.00045	0.00064	<0.00490	0.0053	0.016	0.0025	<0.0068	<0.0068	0.022
KA01	E	<0.00035	<0.0004	<0.00045	<0.00045	<0.00490	0.00057	0.0018	<0.00075	<0.0068	<0.0068	<0.0062
KA01	F	0.00038	<0.0004	<0.00045	<0.00045	<0.00490	0.00051	0.0016	<0.00075	<0.0068	<0.0068	<0.0062
TE09	A	<0.00035	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0079	<0.00075	<0.0068	<0.0068	0.004
TE09	B	<0.00035	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0014	<0.00075	<0.0068	<0.0068	<0.0062
TE09	C	<0.00035	<0.0004	<0.00045	<0.00045	<0.00490	0.00048	0.0098	0.00084	<0.0068	<0.0068	0.0077
TE09	D	<0.00035	<0.0004	<0.00045	0.00056	<0.00490	<0.00052	0.0084	0.0014	<0.0068	<0.0068	0.0093
TE13	A	0.0007	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0076	0.00057	<0.0068	<0.0068	0.0069
TE13	B	0.0004	<0.0004	<0.00045	<0.00045	<0.00490	0.00058	0.019	0.0011	<0.0068	<0.0068	0.011
TE20	A	0.00046	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0064	0.001	<0.0068	<0.0068	<0.0062
TE20	B	0.00037	<0.0004	<0.00045	<0.00045	<0.00490	0.0007	0.0099	0.0011	<0.0068	<0.0068	0.0078
TE20	C	<0.00035	<0.0004	<0.00045	<0.00045	<0.00490	<0.00052	0.0018	<0.00075	<0.0068	<0.0068	<0.0062
TE21	A	0.0007	<0.0004	<0.00045	0.00051	<0.00490	0.0013	0.028	0.0011	<0.0068	<0.0068	0.036
TE21	B	0.00042	<0.0004	<0.00045	0.00053	<0.00490	0.00091	0.019	0.0014	<0.0068	<0.0068	0.017
YF20	A	0.0074	0.0056	0.0012	0.0080	<0.00490	0.046	0.290	0.015	0.041	<0.0068	0.710
YF20	B	0.0015	0.00075	<0.00045	0.0019	0.0074	0.0078	0.041	0.0065	0.014	<0.0068	0.110
YF20	C	0.0046	0.0018	0.00063	0.0060	0.0190	0.025	0.140	0.017	0.026	<0.0068	0.310

Appendix A, continued.

Site	Fish	Aroclor 1260	PCB-TOTAL	toxaphene	trans-nonachlor
FA01	A	0.200	0.390	0.140	0.015
FA01	B	0.120	0.270	0.034	0.0095
FA01	C	0.440	0.730	0.053	0.0076
FA01	D	0.160	0.310	0.180	0.015
FA01	E	0.840	1.40	0.200	0.081
FA01	F	0.280	0.550	0.150	0.026
FA01	G	0.170	0.540	0.180	0.054
FA01	H	0.540	0.910	0.024	0.0064
FA01	I	0.092	0.270	0.093	0.017
KA01	A	0.011	0.023	0.0056	0.0013
KA01	B	0.031	0.110	0.018	0.012
KA01	C	<0.0058	<0.016	<0.0029	<0.0015
KA01	D	0.011	0.033	0.0052	<0.0015
KA01	E	<0.0058	<0.016	<0.0029	<0.0015
KA01	F	<0.0058	<0.016	<0.0029	<0.0015
TE09	A	<0.0058	<0.016	0.0085	<0.0015
TE09	B	<0.0058	<0.016	<0.0029	<0.0015
TE09	C	0.0068	<0.016	0.015	0.0019
TE09	D	<0.0058	<0.016	0.012	0.0015
TE13	A	0.0081	<0.016	0.0097	<0.0015
TE13	B	0.150	0.170	0.024	0.0021
TE20	A	0.0088	<0.016	0.023	0.0021
TE20	B	0.0096	0.017	0.012	0.0018
TE20	C	<0.0058	<0.016	0.0047	<0.0015
TE21	A	0.024	0.060	0.027	0.0038
TE21	B	0.014	0.033	0.020	0.0029
YF20	A	0.200	0.950	0.420	0.140
YF20	B	0.032	0.160	0.087	0.019
YF20	C	0.085	0.420	0.290	0.063

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APPENDIX B: DOCUMENTATION AND SAMPLE HANDLING

STUDY PROPOSALS

A study proposal was submitted prior to sampling. Study plans included objectives of the study, a discussion of the justification for the study including a review of related research, a methods section including discussion of collection and analysis procedures, and a cost proposal based on number and types of samples to be collected.

FIELD DOCUMENTATION

During the field portion of the study, sample documentation was recorded in a waterproof field notebook in permanent ink. The date and time of collections at each site were specified as well as the location of the exact collection site and any other pertinent information.

SAMPLE CATALOG

A sample catalog was prepared prior to submission of the samples to the laboratory. The catalog contained study objectives; background information including number of samples and dissection techniques; previous findings and concerns; methods of preservation and storage; instructions to the laboratory, including a description of the analyses requested together with the suggested analytical method; a list of data recipients; a cost estimate for the requested analyses; and a tabulated summary of information on each sample. This information included the sample identification, the date of collection, the type of tissue, the species, the sample location, sample weight or volume, and analyses requested for each particular sample. The catalogs were submitted to AXYS Analytical Services, Ltd., 2045 Mills Road, Sidney, BC, Canada V8L 3S8.

Catalogs were inspected by a Quality Assurance Officer at the Patuxent Analytical Control Facility. Upon approval, they were forwarded to the laboratory together with the listed samples. Laboratory data were received by the authors following review and approval by the Quality Assurance Officer.

CHAIN OF CUSTODY

No chain of custody forms accompanied these catalogs. Sampling was performed for baseline information, and was not anticipated to be used in legal proceedings.

SAMPLE STORAGE AND SHIPMENT

Liver samples were placed in coolers with blue ice and transported by boat for storage. Samples were shipped to the laboratory by air courier. Frozen samples were shipped with dry ice.

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APPENDIX C: QUALITY ASSURANCE/QUALITY CONTROL OF CHEMICAL ANALYSES

The U.S. Fish and Wildlife Service (Service) currently maintains contracts with several analytical laboratories, and also performs analytical work at the Patuxent Analytical Control Facility (PACF), Patuxent National Wildlife Research Center, Laurel, Maryland, to determine the inorganic and organic composition of samples.

Contract laboratories are selected by a PACF technical committee using a process involving the correct analysis of samples submitted to prospective laboratories by PACF, and a review of the laboratory, its procedures, facilities, experience, and personnel. A final step in selecting a laboratory is an on-site inspection by representatives of the evaluation committee. Continued round-robin testing and cross-checking of contract laboratories by PACF has been used to monitor performance and alert the Service's Quality Assurance Project Officer of systematic analytical problems with particular analytes. Approximately 5% of all sample catalogs submitted for analysis to contract laboratories are also reanalyzed by PACF. In addition to these QA-QC measures, precision, accuracy, and potential laboratory contamination of samples are evaluated through the analysis of specific quality control samples. Reports produced by contract laboratories are required to contain the following:

1. A brief description of the methods used in the analysis.

2. The analytical results.

3. Results of any QA-QC samples analyzed in conjunction with the reported catalog, including:
    a. Limits of detection for each sample
    b. Duplicate analysis
    c. Spiked sample analysis
    d. Procedural blank analysis

4. A description of any problems encountered in the analysis.

The laboratory may also be required to submit copies of all raw data collected during the analysis upon request. In addition to a brief description of the methods, we have typically requested that the laboratory provide a description of detailed methods, and the specific instrumentation used, including model numbers.

ACCEPTANCE PARAMETERS

The parameters which QA/QC data must meet to be acceptable are as follows:

When sample values are >LOD, use these parameters:

Mean Spike Recoveries should be $\geq$ 85%.

Mean duplicate analysis RPDs should be ≤ 15%.
No more than 3 individual analytes should be < 80% for mean spike recoveries or >20% for mean RPDs.

When sample values are <LOD, the following parameters are used:

Mean Spike Recoveries should be ≥ 70%.
Mean duplicate analysis RPDs should be ≤ 30%.
No more than 3 individual analytes should be <50% for mean spike recoveries or >50% for mean RPDs.

LIMITS OF DETECTION

The criterion "limit of detection" (LOD) has been variously defined and its determination is the subject of controversy (Greenberg et al. 1992). A general definition for LOD is that it is the lowest concentration level that can be distinguished statistically from a blank sample. That is, it is a reliable limit for an analyte, above which values are consistently detectable and distinguishable from instrument noise. Samples reported as being below the detection limit in a data set are reported as <X where X is the LOD.

Individual sample LOD's may also be reported by the laboratory, because the method LOD actually varies depending on the nature of the individual sample. The greatest LOD reported for any individual sample was adopted as the analyte LOD.

ANALYTICAL PRECISION

Precision refers to the degree of agreement among repeated measurements of a given sample. Precision varies with such factors as the homogeneity of the sample, sample volume, sample matrix, instrumental method, instrumental drift, chemical interferences, and the analyte concentration in the sample. Estimates of precision used for this study were made using duplicate analysis, where at least two subsamples of a homogenized sample are collected and analyzed by the contract laboratory. Precision is monitored by the contract laboratory using range ratio control charts for each analyte of each matrix (sediment, tissue). The measure selected for estimating precision is the relative percent difference (RPD):

$$RPD = ([D_1 - D_2]/[(D_1 + D_2)/2]) \times 100$$

where RPD is the relative percent difference, D_1 is the concentration as determined by the first analysis, and D_2 is the concentration as determined by the second analysis.

Acceptable precision is based not only on the absolute value of the RPD, but also on the relationship between the concentration of the analyte and the LOD for that analyte. For duplicate samples with analyte concentrations where both values are less than the LOD, no estimate of precision is made because this comparison is normally inappropriate (Greenberg et al. 1992). For sample concentrations less than twice the LOD, precision is expected to be low, because instrument performance typically declines as the LOD is approached. The 95%

confidence interval for these cases is assumed to be 2(LOD) (or up to 200% of the actual reported value of a single sample). Sample analyses identifying concentrations less than 2(LOD) are not rejected based on poor precision but are considered qualitative.

Average RPD's for each analyte are calculated separately. The LOD may vary according to sample, the LOD used is the highest LOD identified for each analyte in the sample data set. For concentrations of an analyte >2(LOD) and <10(LOD), results are expected to be semi-quantitative, and dependent on their relation to the LOD. In these samples, both precision and accuracy may be reduced. For measurements >10(LOD), analyses can be expected to be highly quantitative.

ANALYTICAL ACCURACY

Spiked Samples

In addition to precision, measurements of correctness of the analyses are needed to guarantee the quality of semi-quantitative (>2 LOD and <10 LOD) and quantitative (>10 LOD) data, and to estimate chemical interferences that may occur. One method used by Service contract laboratories to estimate accuracy and gauge interference is the use of spiked samples. This method consists of dividing a homogenized sample into two subsamples, analyzing one as the sample, spiking the other subsample with a known quantity of one or more analytes, and analyzing the resulting mixture. The difference between the two subsamples, after accounting for any differences in sample weight, is the spike recovery. This value is usually reported as a percentage of the amount added. Recovery rates greater than 100% may indicate that the instrument was incorrectly calibrated, subject to upward drift, or that contamination of the sample may have occurred. Recoveries of less than 100% could occur due to loss of the analyte during the sample procedure (e.g., loss of some analyte due to volatility), instrument drift downward, errors in the calibration procedure, or chemical interferences inherent in the matrix being analyzed.

Usually, the amount of spiking solution added to a sample is sufficient to result in a concentration of that analyte of more than twice the original concentration in the sample and >2(LOD). The Spike/Background ratio must be ≥ 1; the ideal ratio is 5-10.

LITERATURE CITED

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