Attachment 7

Standard Operating Procedure for Caribou Tissue Sampling Program

Arctic Caribou Contaminant Program
Collection and Tissue Analysis Protocol

Caribou are sampled for this program through local hunters. This may be arranged through the local Hunters and Trappers Organization or in collaboration with an existing program collecting samples for other purposes. The intent is always to solicit samples from animals that are being harvested for food and not to encourage any additional harvest. To this end, compensation for samples is carefully considered and is determined in collaboration with local communities, biologists and non-profit organizations (eg. Renewable Resource Councils).

Sample Collection
Ideally 20 caribou are sampled from each herd in the fall of the year (Sept – Oct). Although this is the ideal timing to compare to data from other caribou herds from across the Arctic, we recognize that it is not always possible, and we also recognize the value of obtaining samples from caribou when and where they are traditionally harvested for food. Ideally 10 bulls and 10 cows are harvested, but again, we recognize this is not always possible, sometimes due to harvest regulations and sometimes due to availability.

From each caribou, hunters collect one entire kidney, 500g of liver, 500g of muscle, a 1-inch square of hide with hair attached and the incisor bar (front teeth). All samples from one animal are stored together in one large ziplock bag, labelled with the date, location and sex of the caribou and then frozen for storage.

Sample Processing
In the laboratory, a biologist/technician processes the samples in a 'trace element clean' manner prior to analysis to ensure that a clean sample is being analyzed. Kidney fat and the kidney capsule are carefully removed from the kidney and the clean kidney is stored in a Whirl-pak. The outer layers of the muscle and liver sample are removed and clean inner subsamples are stored separately in Whirl-paks. All samples are frozen before being shipped to the analytical laboratory.

The two central incisors are carefully removed from the incisor bar (lower jaw) using a dental elevator, maintaining the integrity of the tooth roots. These are stored in a paper envelope before being used to age the caribou.

Sample Analysis
Entire kidneys will be Hair and homogenized kidneys will be analyzed for a suite of 36 elements by Inductively Coupled Plasma-Mass spectrometry (ICP-MS) (NLET 2002; USEPA 2007a). In brief, muscle (1 g) will be digested with nitric acid and hydrogen peroxide (8:1) in a high-pressure microwave oven at 200ºC for 15 minutes and the digest was analyzed by ICP-MS. Mercury will also be determined in kidneys and hair using a Direct Mercury Analyzer (DMA) using US EPA Method 7473 (US EPA 2007a). Certified biological reference materials for mercury and multi-element analysis (DOLT-2, DORM-2 and TORT-2 from National Research Council of Canada) will be analyzed for every 20 samples; recoveries should be within 10% of certified values.

Perfluoroalkyl substances (PFASs) will be determined in liver samples using analytical methods used for other Northern Contaminant Program projects (described in as described in Reiner et al. 2012 and Lescord et al. 2015). In brief, liver samples will be extracted with acetonitrile and the extract cleaned up on a carbon solid phase extraction column. PFASs (24 compounds) will be analyzed by LC-tandem mass spectrometry.

Polybrominated diphenyl ethers (PBDEs) (24 congeners including BDE 209 and related nonaBDEs found in technical "Deca" BDE) will be determined in liver samples. In brief, liver samples will be Soxhlet extracted with dichloromethane and lipid removed by gel permeation chromatography using USEPA Method 1699 (US EPA 2007b) with minor modifications. Extracts will be fractionated on silica gel

columns, reduced in volume and vialed for GC-MS analysis. PBDEs and other BFRs including hexabromocyclododecane (HBCD) will be analysed by low resolution GC-tandem mass spectrometry in negative ionization mode.

Liver, muscle and remaining kidney tissue will be archived for potential future analysis.

Caribou teeth will be aged using the cementum technique (Matson 1981).

Data Analysis

Data will be combined with the existing Arctic caribou dataset and assessed for temporal and geographic trends in contaminants of concern. Note that sex, age, season of collection, year of collection and/or herd may significantly affect tissue element concentrations.

References

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