Third National Health and Nutrition Examination Survey 1988–1994

Documentation, Codebook, and Frequencies

Dialkylphosphates

Laboratory
Surplus Sera

Survey Years:
1988 to 1994

SAS Export File:
SSNH3DAP.XPT

First Published: May 2009

NHANES III Data Documentation

Laboratory Assessment: Urinary Dialkylphosphates (Residual PTRRS urine)

Years of Coverage: 1988-1994 **First Published:** May 2009 **Last Revised:** N/A

Component Description

Measurement of dialkylphosphates in residual urine collected as a part of the Priority Toxicant Reference Range Study.

Eligible Sample

Participants ≥ 18 years of age from NHANES III (1988-1994). Not a representative sample of NHANES.

Description of Laboratory Methodology

Measurement of Urinary Dialkylphosphates Metabolites in Urine.
Urine samples were analyzed for DAP metabolites of OP pesticides using the methods of Bravo et al. (1). The 2002 method was used to analyze samples collected in NHANES III and NHANES 1999–2000. The 2004 method was used for subsequent analyses. Both methods were shown to agree using a Pearson correlation analysis ($r>0.97$, $p<0.001$ for all analytes); a Bland-Altman plot showed good agreement with no systematic bias between the two methods (mean percent difference <0.5% for all analytes). Briefly, 4 mL of urine were spiked with an isotopically-labeled internal standard mixture, then concentrated to dryness using an azeotropic codistillation with acetonitrile or lyophilization. The dried residue was dissolved in acetonitrile, and the DAPs were derivatized to their respective chloropropyl esters using 1-chloro-3-iodopropane and potassium carbonate. The solution containing the chloropropyl esters was concentrated and then analyzed using gas chromatography-positive chemical ionization-tandem mass spectrometry. The DAP metabolites were quantified using isotope-dilution calibration. The analytic limits of detection (LODs) were 0.5 ng/mL for dimethylphosphate (DMP), 0.18 ng/mL for dimethylthiophosphate (DMTP), 0.08 ng/mL for dimethyldithiophosphate (DMDTP), 0.2 ng/mL for diethylphosphate (DEP), 0.09 ng/mLfor diethylthiophosphate (DETP), and 0.05 ng/mLfor diethyldithiophosphate (DEDTP).

Laboratory Quality Control and Monitoring

The laboratory and method were certified according to the Clinical Laboratory Improvement Amendment (1988) guidelines (2).

Quality Control

Urine was collected from multiple (> 15) donors, combined, diluted with water (1:1 v/v) to reduce endogenous levels of the analytes of interest, and mixed overnight at 4°C. Our protocol for anonymous collection of urine was reviewed and approved by CDC's Institutional Review Board (IRB). The urine pool was pressure-filtered with a 0.2-µm filter capsule and divided into four pools. The first pool (QCL), the second pool (QCM), and the third pool (QCH) were spiked with the native standard stock solution to yield concentrations of 5 ng/ml, 15 ng/ml and 30 ng/ml, respectively. The fourth pool was not spiked. After being screened for possible endogenous analytes, the fourth pool was used as matrix material for calibration standards and blanks.

Just before each analytical run, calibration standards were prepared by diluting the working standard stock solutions in blank urine. The concentrations of the nine calibration standards ranged from 0.125 to 50 ng/ml for each of the analytes. To each run was added the nine calibration samples, two sets of three quality control samples (QCL, QCM, and QCH), and one blank urine sample; these were extracted and analyzed in parallel with the unknown samples.

All QC pools were characterized before use to determine the mean and 99th and 95th control limits by consecutively analyzing at least 20 samples from each QC pool. QC samples were analyzed in runs with 2 replicates in 10 runs over 10 days. After establishment of the control limits of the pools, individual QC samples contained within each analytical run were evaluated for validity by use of a multi-rule quality control (3).

Quality Assurance

A quantification and a confirmation product (MS/MS) ion are monitored for each analyte. In order to be quantified, the peaks must coelute with its isotopically labeled internal standard and possess both the quantification and confirmation ion in a specified ratio of abundance.

Blanks or negative controls were analyzed concurrently with the samples and were subtracted from the analytical data.

Data Processing and Editing

Data was received after all the laboratory testing was complete. The data were not edited.

Data Access: All data are publicly available.

Analytic Notes

There are 7 variables:

SEQN = RESPONDENT SEQUENCE NUMBER

DEDTP = DIETHYLDITHIOPHOSPHATE

DED = DIETHYLPHOSPHATE

DETP = DIETHYLTHIOPHOSPHATE

DMDTD = DIMETHYLDITHIOPHOSPHATE

DMD = DIMETHYLPHOSPHATE

DMTD = DIMETHYLTHIOPHOSPHATE

Concentrations are in ng/mL units.

References

1. Bravo R, Driskell WJ, Whitehead RD, Needham LL, Barr DB. 2002. Quantification of dialkyl phosphate metabolites of organophosphate pesticides in human urine using GC-MS/MS with isotope dilution method. J Anal Toxicol 26:245-252.

2. Clinical Laboratory Improvement Amendment. Public Law 100-578. 1988. 10-31-1988.

3. Caudill SP, Schleicher RL, Pirkle JL. 2008. Multi-rule quality control for the age-related eye disease study. Stat Med 27:4094-4106.

Locator Fields

Title: Urinary Dialkylphosphates (Residual PTRRS urine)

Contact Number: 1-866-441-NCHS

Years of Content: 1988-1994

First Published: May 2009

Last Revised: N/A

Access Constraints: None

Use Constraints: None

Geographic Coverage: National

Subject: Urinary Dialkylphosphates (Residual PTRRS urine)

Record Source: NHANES III

Survey Methodology: NHANES III is a stratified multistage probability sample of the civilian non-institutionalized population of the U.S.

Medium: NHANES Web site; SAS transport files

National Health and Nutrition Examination Survey
Codebook for Data Production (1988-1994)
(NHANES III)

NHANES III Dialkylphosphates (SSNH3DAP)
Person Level Data

May 2009

SEQN	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	Respondent sequence number
English Text: Respondent sequence number.	
English Instructions:	

SSDEDTP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIETHYLDITHIOPHOSPHATE
English Text: DIETHYLDITHIOPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.04 to 908.09	Range of Values	197	197	
.	Missing	0	197	

SSDEP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIETHYLPHOSPHATE
English Text: DIETHYLPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.14 to 60.96	Range of Values	184	184	
.	Missing	13	197	

SSDETP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIETHYLTHIOPHOSPHATE
English Text: DIETHYLTHIOPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.06 to 10127.27	Range of Values	196	196	
.	Missing	1	197	

SSDMDTP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIMETHYLDITHIOPHOSPHATE
English Text: DIMETHYLDITHIOPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.06 to 134.15	Range of Values	194	194	
.	Missing	3	197	

SSDMP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIMETHYLPHOSPHATE
English Text: DIMETHYLPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.35 to 130.03	Range of Values	192	192	
.	Missing	5	197	

SSDMTP	Target
	B(18 Yrs. to 150 Yrs.)
Hard Edits	**SAS Label**
	DIMETHYLTHIOPHOSPHATE
English Text: DIMETHYLTHIOPHOSPHATE	
English Instructions:	

Code or Value	Description	Count	Cumulative	Skip to Item
0.13 to 250.91	Range of Values	189	189	
.	Missing	8	197	