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## Documentation, Codebook, and Frequencies

Laboratory Component:

Urinary Organophosphate Insecticides: Diakyl Phosphate Metabolites

Survey Years: 2003 to 2004

SAS Export File: L26OPD\_C.XPT



First Release: July 2008 Last Revised: N/A

#### NHANES 2003–2004 Data Documentation Laboratory Assessment: Lab 26 Urinary Organophosphate Insecticides: Diakyl Phosphate Metabolites

First Published: July 2008

Last Revised: N/A

#### Component In 1999, about five billion pounds of pesticide active ingredients were Description used in the US, most of it for agricultural applications. The most recent registration data provided by the US EPA showed over 800 pesticidal active ingredients available in about 21,000 different formulations. Widespread use of the contemporary pesticides for agriculture and residential applications makes it virtually impossible for the average person to completely avoid exposure. Pesticide residues and their metabolites in human tissues and fluids can be indicative of pesticide exposure and the total body burden of these pesticides. Exposure to several pesticides was assessed by measuring urinary pesticide metabolites during NHANES 1999-2002. However, determination of the specific pesticide linked to the exposure can be inaccurate because some metabolites are common to multiple pesticides. Beginning in NHANES 2003-2004, specific pesticides in blood were also measured.

Little information is available concerning residential or household exposures to pesticides among the general population. Sufficient data do exist, however, from surveys or other focused research efforts to suggest that household exposure to certain common pesticides can be extensive and might be of significant public health concern. Pesticides of particular concern are: chlorpyrifos, 2,4-D, diazinon, permethrin, ortho-phenyl phenol, methyl parathion, and organophosphate pesticides.

# EligibleParticipants aged 6 years and older who met the subsampleSamplerequirements.

# Description of<br/>LaboratoryThis method was developed for quantifying the six DAP urinary metabolites<br/>of at least 28 organophosphorus pesticides using lyophilization and<br/>chemical derivatization followed by analysis using isotope-dilution gas<br/>chromatography-tandem mass spectrometry (GC-MS/MS). Urine samples<br/>were spiked with stable isotope analogues of the DAPs and the water was<br/>removed from the samples using a lyophilizer. The dried residue was

dissolved in acetonitrile and diethyl ether, and the DAPs were chemically derivatized to their respective chloropropyl phosphate esters. The chloropropyl phosphate esters were concentrated, and analyzed using GC–MS/MS. The limits of detection of the method were in the low mg/l (parts per billion) to mid pg/ml range (parts per trillion) with coefficients of variation of 7–14%. The use of stable isotope analogues as internal standards for each of these metabolites allows for sample-specific adjustment for recovery and thus permits a high degree of accuracy and precision. Use of this method with approximately 1100 urine samples collected from pregnant women and children indicate that the low limits of detection allow this method to be used in general population studies <sup>1</sup>.

#### Laboratory Quality Control and Monitoring

Urine specimens are processed, stored, and shipped to the Division of Laboratory Sciences, National Center for Environmental Health, Centers for Disease Control and Prevention for analysis.

Detailed specimen collection and processing instructions are discussed in the NHANES Laboratory/Medical Technologists Procedures Manual (LPM). Vials are stored under appropriate frozen (–20°C) conditions until they are shipped to National Center for Environmental Health for testing.

#### Mobile Examination Centers (MECs)

Laboratory team performance is monitored using several techniques. NCHS and contract consultants use a structured quality assurance evaluation during unscheduled visits to evaluate both the quality of the laboratory work and the quality-control procedures. Each laboratory staff person is observed for equipment operation, specimen collection and preparation; testing procedures and constructive feedback are given to each staff. Formal retraining sessions are conducted annually to ensure that required skill levels were maintained.

The NHANES QA/QC protocols meet the 1988 Clinical Laboratory Improvement Act mandates. Detailed QA/QC instructions are discussed in the NHANES LPM.

#### **Analytical Laboratories**

NHANES uses several methods to monitor the quality of the analyses performed by the laboratories. In the MEC, these methods include performing blind split samples collected on "dry run" sessions. In addition, contract laboratories randomly perform repeat testing on 2.0% of all specimens.

NCEH developed and distributed a quality control protocol for all the laboratories which outlined the Westgard rules used when running NHANES specimens. Any problems encountered during shipping or receipt of specimens, instrument calibration, reagents, and any special considerations are submitted to NCHS and Westat. Summary statistics for each control pool, QC graphs, are reviewed by NCHS for trends or shifts in the data. The laboratories are required to explain any identified areas of concern.

All QC procedures recommended by the manufacturers were followed. Reported results for all assays meet the Division of Laboratory Sciences' quality control and quality assurance performance criteria for accuracy and precision (similar to specifications outlined by Westgard (1981).

#### Analytic Subsample weights

Notes

Measures of urinary diakyl phosphate metabolites were measured in a one third subsample of persons 6 years and over. Special sample weights are required to analyze these data properly. Specific sample weights for this subsample are included in this data file and should be used when analyzing these data.

#### Variance estimation

The analysis of NHANES 2003-2004 laboratory data must be conducted with the key survey design and basic demographic variables. The NHANES 2003-2004 Demographic Data File contains demographic and sample design variables. The recommended procedure for variance estimation requires use of stratum and PSU variables (SDMVSTRA and SDMVPSU, respectively) in the demographic data file.

#### Links to NHANES Data Files

This laboratory data file can be linked to the other NHANES 2003-2004 data files using the unique survey participant identifier SEQN.

#### **Detection Limits**

The detection limits were constant for all of the analytes except for diethyldithiophosphate (URXOP6) in the data set. Diethyldithiophosphate (URXOP6) has two detection limits. Two variables are provided for each of these analytes. The variable named URD\_\_\_\_LC indicates whether the result was below the limit of detection. There are two values: "0" and "1"". "0" means that the result was at or above the limit of detection. "1" indicates that the result was below the limit of detection.

The other variable named URX\_\_\_\_ provides the analytic result for that analyte.

The detection limit divided by the square root of 2 is the value that is provided for results that are below the limit of detection.

Analyte	Item Id	Limit of Detection(LLOD)
Dimethylphosphate	URXOP1	0.5
Diethylphosphate	URXOP2	0.1
Dimethylthiophosphate	URXOP3	0.5
Diethylthiophosphate	URXOP4	0.2
Dimethyldithiophosphate	URXOP5	0.1
Diethyldithiophosphate	URXOP6	0.1

The lower limit of detections for the diakyl phosphate metabolites is

Please refer to the Analytic Guidelines for further details on the use of sample weights and other analytic issues.

References 1. Roberto Bravoa, Lisa M Caltabianoa, Gayanga Weerasekeraa, Ralph D Whiteheada, Carolina Fernandeza, Larry L Needhama, Asa Bradmanb and Dana B Barra. Journal of Exposure Analysis and Environmental Epidemiology (2004) 14, 249–259. doi:10.1038/sj.jea.7500322

#### **Locator Fields**

Title: Lab 26 Urinary Organophosphate Insecticides : Diakyl Phosphate Metabolites

Contact Number: 1-866-441-NCHS

Years of Content: 2003-2004

First Published: July 2008

Revised: N/A

Access Constraints: None

Use Constraints: None

Geographic Coverage: National

Subject: Urinary Organophosphate Insecticides : Diakyl Phosphate Metabolites

Record Source: NHANES 2003–2004

Survey Methodology: NHANES 2003-2004 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 (2003-2004)

### Urinary Organophosphate Insecticides: Diakyl Phosphate Metabolites (L26OPD\_C) Person Level Data

First Published: July 2008 Last Revised: N/A



SEQN	Target	
522	B(6 Yrs. to 150 Yrs.)	
Hard Edits	SAS Label	
	Respondent sequence number	
English Text: Respondent seque	ence number	
English Instructions:		

WTSC2YR		Ta	Target		
		B(6 Yrs. to 150 Yrs.)			
Hard Edits		SAS Label			
	Г	Two-year MEC weights of subsample C			
English Text: Two-year	MEC weights of subsample	С			
English Instructions:					
Code or Value	Description	Count	Cumulative	Skip to Item	
0 to 456851.11941	Range of Values	2612	2612		
	Missing	0	2612		

URXOP1		Ta	rget		
		B(6 Yrs. to 150 Yrs.)			
Hard Edits		SAS Label			
		Dimethylphosphate (ug/L)			
English Text: Dimethy	lphosphate	e (ug/L)			
<b>English Instructions:</b>					
Code or Value	Γ	Description	Count	Cumulative	Skip to Item
0.35 to 133.62	Rai	nge of Values	2494	2494	
· · ·		Missing	118	2612	

URDOP1LC		T	arget		
	<b>č</b>	B(6 Yrs. to 150 Yrs.) SAS Label			
Hard Edit	s				
		Dimethylphosphate comment code			
English Text: Dimet	hylphosphate comment code				
<b>English Instructions</b>	:				
Code or Value	Description	Count	Cumulative	Skip to Item	
0	At or above the detection limit	1507	1507		
1	Below lower detection limit	987	2494		
•	Missing	118	2612		

URXOP2		Target				
B(6 Yrs. to 150 Yrs.)       Hard Edits       SAS Label		B(6 Yrs. to 150 Yrs.)				
		Diethylphosphate (ug/L)				
English Text: Diethylph	osphate (ug/L)					
English Instructions:						
Code or Value	Description	Count	Cumulative	Skip to Item		
0.07 to 4481.79	Range of Values	2453	2453			
	Missing	159	2612			

URDOP2LC		Т	arget			
		B(6 Yrs. to 150 Yrs.) SAS Label				
Hard Edit	s					
		Diethylphosphate comment code				
English Text: Diethy	lphosphate comment code					
English Instructions	:					
Code or Value	Description	Count	Cumulative	Skip to Item		
0	At or above the detection limit	1507	1507			
1	Below lower detection limit	946	2453			
•	Missing	159	2612			

URXOP3		Target			
		B(6 Yrs. to 150 Yrs.)			
Hard Edits		SAS Label			
		Dimethylthiophosphate (ug/L)			
English Text: Dimethyl	thiophosphate (ug/L)				
English Instructions:					
Code or Value	Description	Count	Cumulative	Skip to Item	
0.35 to 1058.04	Range of Values	2494	2494		
	Missing	118	2612		

URDOP31	URDOP3LC		Ta	arget	
		B(6 Yrs. to 150 Yrs.)			
Hard Edit	s	SAS Label			
		Dimethylthiophosphate comment code			
English Text: Dimet	hylthiophosph	ate comment code			
English Instructions	:				
Code or Value	De	escription	Count	Cumulative	Skip to Item
0	At or above	the detection limit	2030	2030	
1	Below low	ver detection limit	464	2494	
•	]	Missing	118	2612	

URXOP4		Target       B(6 Yrs. to 150 Yrs.)			
Hard Edits SAS Label		5 Label			
		Diethylthiophosphate (ug/L)			
English Text: Diethylth	iophosphate (ug/L)				
English Instructions:					
Code or Value	Description	Count	Cumulative	Skip to Item	
0.14 to 83.15	Range of Values	2422	2422		
· .	Missing	190	2612		

URDOP41	URDOP4LC		arget		
	~	B(6 Yrs. to 150 Yrs.)			
Hard Edit	s	SAS Label			
	E	Diethylthiophosphate comment code			
English Text: Diethy	lthiophosphate comment code				
English Instructions	:				
Code or Value	Description	Count	Cumulative	Skip to Item	
0	At or above the detection limit	1604	1604		
1	Below lower detection limit	818	2422		
•	Missing	190	2612		

URXOP5		Target			
UMOI 5		B(6 Yrs. to 150 Yrs.)			
Hard Edits	Hard Edits SAS Label				
		Dimethyldithiophosphate (ug/L)			
<b>English Text:</b> Dimethyl	dithiophosphate (ug/L)				
Inglish Instructions:					
Code or Value	Description	Count	Cumulative	Skip to Item	
0.07 to 80.51	Range of Values	2458	2458		
	Missing	154	2612		

URDOP5LC		Target				
		B(6 Yrs. to 150 Yrs.)				
Hard Edits		SAS Label				
		Dimethyldithiophosphate comment code				
English Text: Dimet	hyldithiopho	sphate comment code				
<b>English Instructions</b>	:					
Code or Value	Ι	Description	Count	Cumulative	Skip to Item	
0	At or above	ve the detection limit	1357	1357		
1	Below lo	ower detection limit	1101	2458		
•		Missing	154	2612		

URXOP6		Target       B(6 Yrs. to 150 Yrs.)				
Hard Edits		SAS Label				
		Diethyldithiophosphate (ug/L)				
English Text: Diethyldi	thiophosphate (ug/L)					
English Instructions:						
Code or Value	Description	Count	Cumulative	Skip to Item		
0.07 to 12.67	Range of Values	2494	2494			
	Missing	118	2612			

URDOP6L	С	Target				
	č	B(6 Yrs. to 150 Yrs.)				
Hard Edit	s	SAS Label				
	Γ	Diethyldithiophosphate comment code				
English Text: Diethy	ldithiophosphate comment code					
<b>English Instructions</b>	:					
Code or Value	Description	Count	Cumulative	Skip to Item		
0	At or above the detection limit	777	777			
1	Below lower detection limit	1717	2494			
	Missing	118	2612			

URXUCR		Target       B(6 Yrs. to 150 Yrs.)				
Hard Edits		SAS Label				
		Creatinine, urine (mg/dL)				
English Text: Creatinin	e, urine (mg/dL)					
English Instructions:						
Code or Value	Description	Count	Cumulative	Skip to Item		
7 to 648	Range of Values	2530	2530			
•	Missing	82	2612			