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# United States Department of the Interior

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## NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 2007.03

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Subject: Data reload for Hexahydrohexamethylcyclopentabenzopyran (HHCB) and Acetylhexamethyl-tetrahydronaphthalene (AHTN) by Gas Chromatography/Mass Spectrometry reported in NWQL schedules 1433, 4433, 5433, and custom lab codes 8033, 8043, 8050, 8066, 8068, and 8417

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Revision: none

### Purpose

The purpose of this technical memorandum is to alert USGS data users that results and method reports for two fragrance compounds, HHCB and AHTN, have been reversed. Additional information and clarification is provided in this memorandum to the previously released Rapi-Note 07-010.

### Background

Musk compounds HHCB and AHTN are used as fragrances in numerous personal care products, which may be discharged into domestic wastewaters. These two compounds are important as they serve as effective chemical markers or indicators of the occurrence and evaluation of wastewater contamination in environmental samples (Buerge and others, 2003). Laboratory analysis of the compounds is by gas chromatography / mass spectrometry (GC/MS) in extracts of the applicable matrices, as described in Zaugg, and others (2002, 2006) and Burkhardt, and others (2006). The two compounds have similar characteristics used in their identification (mass spectra, mass spectral quantitation ions, gas chromatographic retention times, and recoveries), and are analyzed together in standard mixtures.

### Effects on Data Users and the Data Base

Data for two fragrance compounds analyzed in Laboratory Schedules (LS) 1433 (filtered water), 4433 (unfiltered water), 5433 (solid material), custom lab code 8033 (unfiltered water) custom lab code 8043 (filtered water), custom lab code 8050 (sediments), custom lab code 8066 (tissues), custom lab code 8068 (POCIS), and custom lab code 8417 (tissues/sediments) have been incorrectly identified and reported for samples received at the NWQL on or prior to the following dates:

Method	Start Date	End Date	Data that will be reloaded in NWIS
Lab code 8043	01/01/2001	07/16/2001	No
Lab code 8050	06/27/2002	6/30/2006	No
Lab code 8066	06/04/2006	12/25/2006	No
Lab code 8068	04/25/2006	12/25/2006	No
Lab code 8417	01/01/2001	09/30/2006	No
Lab code 8033	01/01/2001	08/04/2006	Partial
Schedule 1433	07/16/2001	12/25/2006	Yes
Schedule 4433	08/01/2006	12/25/2006	Yes
Schedule 5433	06/01/2006	08/31/2006	Yes

The misidentification was discovered when blind samples received from the USGS Branch of Quality Systems (BQS) were analyzed by the NWQL as a part of routine quality assurance practices. The samples were spiked with one of the individual compounds rather than both compounds, and the misidentification was confirmed by analyzing the individual components from multiple sources by GC/MS. The misidentification occurred in the initial method validation when the retention time and associated data were switched which resulted in the validation data being assigned to the wrong compounds.

The NWQL will correct the misidentification as follows:

1. Data for both compounds will be switched if one or both of the compounds (see Table 1) had a detection in the sample.
2. No changes will be made for data where both compounds were reported as non-detections since the reporting levels are the same for both compounds.

**Table 1** Information summary for affected Laboratory Schedules/Codes.[NWIS (National Water Information System); CASRN (Chemical Abstract Service Registry Number); RL (reporting level); µg/L, micrograms per liter and µg/Kg, micrograms/kilogram RLType (Reporting Level Type); IRL, Interim Reporting Level]

Compound Name	NWQL Schedule/Custom Labcode	Matrix	NWIS Parameter Code / Old NWQL Method Code	NWIS 5-char method code	CASRN	RL	Units	RL Type
AHTN	1433	Filtered water	62065 / A	GCM37	21145-77-7	0.5	(µg/L)	IRL
HHCB	1433	Filtered water	62075 / A	GCM37	1222-05-5	0.5	(µg/L)	IRL
AHTN	4433/8033	Whole Water	62812 / Z	B0042	21145-77-7	0.2	(µg/L)	IRL
HHCB	4433/8033	Whole Water	62823 / Z	B0053	1222-05-5	0.2	(µg/L)	IRL
AHTN	5433	Sediment	63179 / A	S0225	21145-77-7	50	(µg/Kg)	IRL
HHCB	5433	Sediment	63209 / A	S0253	1222-05-5	50	(µg/Kg)	IRL

The changes to the data will occur in the NWQL database and only the data for the affected samples with detections of one or both compounds will be reloaded. This results in updating and reloading data for approximately 3,000 samples received at the NWQL within the dates specified above for NWIS parameter codes 62065, 62075, 62812, 62823, 63179, and 63209). A Rapi-Note will announce the date of the reload.

Data users need to be aware that this misidentification occurred during development of the methods and applies to all data sets generated prior to the end dates listed above. Data users with data reported via memo and spreadsheets prior to the end dates listed above for custom results should correct these results in their databases by switching the reported detections for Hexahydrohexamethylcyclopentabenzopyran (HHCB) and Acetyl-hexamethyl-tetrahydronaphthalene (AHTN).

## **Revising publications**

The publications for the analytical methods (Zaugg, and others, 2002, 2006, and Burkhardt, and others, 2006) have been reviewed for errors and revisions will be posted.

Data tables and/or data interpretation in previously published publications using results for these two compounds may need revision and/or errata sheets. Phrasing for updating errata is provided for author consideration:

“Quality assurance reviews during May 2007 at USGS NWQL detected an error by which the identifications of AHTN and HHCB were reversed. This error affected data published in this report. The results in this revised publication reflect correct data reporting for these two compounds.”

## **Corrective Action and Prevention**

The NWQL is implementing a process of varying the concentration of the third party check to ensure that identification of compounds is accurate and will analyze individual compound standards for significant changes in methodology in order to minimize this type of error in the future.

//signed//

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## **Keywords**

Acetyl-hexamethyl-tetrahydronaphthalene (AHTN), Hexahydrohexamethylcyclopentabenzopyran (HHCB), Laboratory Schedules 1433, 4433, and 5433 and Custom Lab Codes 8033, 8043, 8050, 8066, 8068, and 8147

## **References**

Zaugg, S.D., Smith, S.G., Schroeder, M.P., Barber, L.B., and Burkhardt, M.R., 2002, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory---Determination of wastewater compounds by polystyrene-divinylbenzene solid-phase extraction and capillary-column gas chromatography/mass spectrometry: U.S. Geological Survey Water-Resources Investigations Report 01-4186, 37 p.

Zaugg, S.D., Smith, S.G., and Schroeder, M.P., 2006, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory- Determination of wastewater compounds in whole water by continuous liquid-liquid extraction and capillary-column gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods, book 5, chap. B4, 30p.

Burkhardt, M.R., Zaugg, S.D., Smith, S.G., and ReVello, R.C., 2006, Determination of wastewater compounds in sediment and soil by pressurized solvent extraction, solid-phase extraction, and capillary-column gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods, book 5, chap B2, 33p.

Buerge, I. J., Buser, H., Muller, M. D., and Poiger, T., 2003, Behavior of the Polycyclic Musks HHCB and AHTN in Lakes, Two Potential Anthropogenic Markers for Domestic Wastewater in Surface Waters, *Environmental Science and Technology*, v. 37, no. 24, p. 5636-5644.