

ATTACHMENT H

NEIC DATA

United States Environmental Protection Agency
Office of Enforcement and Compliance Assurance
Office of Criminal Enforcement, Forensics and Training

NEICSP0287S01

LABORATORY REPORT

Red River Pollutant Identification

Oklahoma

NEIC Project No.: SP0287

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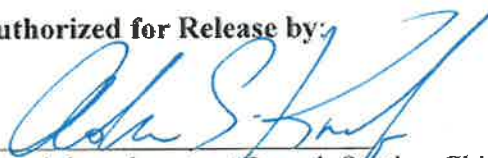


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and provides a clear indication of the end of this report.**

EXECUTIVE SUMMARY

INTRODUCTION

The U.S. Environmental Protection Agency (EPA) National Enforcement Investigations Center (NEIC) provided laboratory assistance to EPA's Region 6 Environmental Services Branch laboratory for the Red River Fish Kill project in Oklahoma. The EPA Region 6 laboratory requested NEIC's assistance in identifying a white substance that is appearing in water samples from the Red River in Oklahoma during the Regional laboratory's analyses for the project.

This report contains laboratory analytical results for white solid material recovered from two gel samples from stations 508672 and 508674 (hereafter referred to as samples 508672 and 508674), as documented on the chain of custody record (**Appendix A**). All NEIC analyses were conducted in accordance with the NEIC quality system by NEIC chemists Jon Beihoffer, Willis Collins, Linda Johnson, Richard Martinez, and Stephanie Volz from October 2011 through December 2011.

SUMMARY OF FINDINGS

- Magnesium hydroxide and sodium chloride crystalline phases are present in the dried white solid recovered from samples 508672 and 508674.
- No organic constituents were detected in the dried white solid recovered from samples 508672 or 508674 by nuclear magnetic resonances (NMR) spectrometry, infrared (IR) spectroscopy, or Raman spectroscopy.
- The elemental analysis results for the dried white solid recovered from sample 508672 were 14.7 percent (%) weight per weight (wt/wt) of magnesium, 1.2% wt/wt of sodium, 0.77% wt/wt of sulfur, 0.59% wt/wt of calcium, and 0.46% wt/wt of silicon.
- The elemental analysis results for the dried white solid recovered from sample 508674 were 21.9% wt/wt of magnesium, 3.1% wt/wt of sodium, 1.2% wt/wt of calcium, 1.0% wt/wt of sulfur, and 1.0% wt/wt of silicon.

LABORATORY ACTIVITIES

INTRODUCTION

On October 3, 2011, the NEIC laboratory received two gel and two water samples collected on August 26 and 30, 2011, as part of the EPA Region 6 Red River Pollutant Fish Kill project in Oklahoma. Laboratory sample delivery, receipt, and transfer are summarized in Table 1. A copy of the chain of custody record is attached in **Appendix A**. The samples were maintained under custody in accordance with the NEIC operating procedure *Evidence Management*, NEICPROC/00-059R3.

Table 1. LABORATORY SAMPLE DELIVERY, RECEIPT, AND TRANSFER
Red River Pollutant Identification
Oklahoma

Event	Date	Comments
Samples received	October 3, 2011	Delivered to NEIC by UPS (tracking No. 1Z A42 00W01 5075 3585) and received by Jon Beihoffer.
Samples unpacked and inspected	October 3, 2011	Received one blue-and-white cooler sealed with tape and custody seal. The tape and custody seal were intact. Jon Beihoffer unpacked and inspected the cooler. The cooler contained two gel and two water samples. The samples were received intact. No ice was present, but the samples were cold to the touch. The samples were stored in a locked small Pelican case in walk-in cooler A in room 1C-210.

ANALYTICAL PROCEDURES AND ANALYSTS

Table 2 lists the analytical procedures and analysts. The analytical procedures were used to determine the identity of the dried white solid material recovered from samples 508672 and 508674. The two water samples from stations 508670 and 508671 were not analyzed. All analyses were conducted by NEIC personnel in accordance with the NEIC quality system during October 2011 through December 2011. Data quality summaries for all laboratory measurements are maintained in the project file.

Table 2. ANALYTICAL PROCEDURES AND ANALYSTS
Red River Pollutant Identification
Oklahoma

Analytical Method/Procedures	Analyst
<i>Physical Description/Phase Separation</i> , NEICPROC/00-045R2	Jon Beihoffer
<i>Qualitative Identification by Infrared Spectroscopy</i> , NEICPROC/00-046R3	Stephanie Volz
<i>Raman Spectroscopy</i> , NEICPROC/09-001	Linda Johnson
<i>Analyses by Nuclear Magnetic Resonance Spectrometry</i> , NEICPROC/00-025R2	Jon Beihoffer
<i>X-Ray Diffraction for Qualitative Identification of Crystalline Phases</i> , NEICPROC/99-019R2	Richard Martinez
Elemental analysis by inductively coupled plasma-optical emission spectroscopy (ICP-OES), EPA SW-846 Method 6010C as guidance	Willis Collins

SAMPLE PREPARATION

The two gel samples were centrifuged at 3,000 revolutions per minute (rpm) for 5 minutes. After centrifuging, the gel from sample 508672 had two phases: a clear colorless nonviscous liquid on top and a white solid material on the bottom. The gel from sample 508674 had three phases: a clear colorless nonviscous liquid on top, a white solid material in the middle, and a clear, colorless, nonviscous liquid on the bottom. The liquid phases were transferred to glass vials, and the white solid materials were air-dried at room temperature for several days.

ANALYTICAL RESULTS

Table 3 contains the physical descriptions of the materials analyzed and summarizes the analytical results. Water samples from stations 508670 and 508671 were not analyzed because no organic constituents were detected in the dried white solid recovered from samples 508672 and 508674.

Elemental Analyses

A subsample of the dried white solid recovered from samples 508674 and 508672 was dissolved in 2 percent nitric acid solution and heated. Each subsample generated gas in the acid solution by bubbling, which is an indication of the presence of carbonates. The acid solutions were analyzed by inductively coupled plasma-optical emission spectroscopy (ICP-OES). The dried white solid recovered from sample 508672 contained 14.7% wt/wt of magnesium, 1.2% wt/wt of sodium, 0.77% wt/wt of sulfur, 0.59% wt/wt of calcium, and 0.46% wt/wt of silicon. The dried white solid recovered from sample 508674 contained 21.9% wt/wt of magnesium, 3.1% wt/wt of sodium, 1.2% wt/wt of calcium, 1.0% wt/wt of sulfur, and 1.0% wt/wt of silicon.

X-Ray Diffraction (XRD) Analyses

The diffraction pattern for the dried white solid recovered from samples 508672 and 508674 was consistent with a standard reference spectrum of magnesium hydroxide and had at least three matched lines with the International Centre for Diffraction Data diffraction pattern for magnesium hydroxide and sodium chloride. XRD analysis only detects constituents in the crystalline phase. Constituents that are amorphous are not detected by the analysis, which has a detection limit of 1 to 5 percent.

Infrared (IR) Analyses

The spectrum for the dried white solid recovered from sample 508672 was consistent with the spectrum of a standard of magnesium hydroxide and water. The spectrum for the dried white solid recovered from sample 508674 was consistent with the spectrum of a standard of magnesium hydroxide. The spectra were obtained using a diamond attenuated total reflectance (ATR) accessory. The detection limit for this analysis was 1 to 5 percent.

Nuclear Magnetic Resonance (NMR) Analyses

No constituent in a concentration of more than 100 parts per million (ppm) was extracted from subsamples of the dried white solids recovered from samples 508672 and 508674. Subsamples of the dried white solid recovered from sample 508672 were extracted in deuterated methanol and in deuterated toluene. No proton resonances were observed in these extractions. Subsamples of the dried white solid recovered from sample 508674 were extracted in deuterated acetone, in deuterated benzene, and in deuterated dimethyl sulfoxide. No proton resonances were observed in these extractions.

Raman Analyses

No useful information was obtained from the Raman analysis of a subsample of the dried white solid recovered from sample 508674. An interferogram was obtained for the sample, but the spectrum was noisy and did not contain any peaks.

Table 3. DESCRIPTIONS OF MATERIALS ANALYZED AND ANALYTICAL RESULTS
Red River Pollutant Identification
Oklahoma

Station No. Date Collected ¹	Station Location	Laboratory Physical Description	Analytical Results
508672 8/30/11	1109034-08	Dried white solid	Elemental results: 14.7% wt/wt of magnesium, 1.2% wt/wt of sodium, 0.77% wt/wt of sulfur, 0.59% wt/wt of calcium, and 0.46% wt/wt of silicon XRD results: magnesium hydroxide and sodium chloride crystalline phases are present. IR results: magnesium hydroxide is present, and no organic constituents detected. NMR results: no organic constituents detected in the extracts.
508674 8/26/11	1109024-10	Dried white solid	Elemental results: 21.9% wt/wt of magnesium, 3.1% wt/wt of sodium, 1.2% wt/wt of calcium, 1.0% wt/wt of sulfur, and 1.0% wt/wt of silicon XRD results: magnesium hydroxide and sodium chloride crystalline phases are present. IR results: magnesium hydroxide is preset, and no organic constituents detected. NMR results: no organic constituents detected in the extracts.
508670 8/26/11	1109024-04		Not analyzed
508671 8/30/11	1109034-05		Not analyzed

APPENDIX A

CHAIN OF CUSTODY RECORD
(1 page)

CHAIN OF CUSTODY RECORD

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