Prepared in cooperation with the U.S. Coast Guard

Determination of the Anionic Surfactant Di(Ethylhexyl) Sodium Sulfosuccinate in Water Samples Collected from Gulf of Mexico Coastal Waters Before and After Landfall of Oil from the Deepwater Horizon Oil Spill, May to October, 2010

By James L. Gray, Leslie K. Kanagy, Edward T. Furlong, Jeff W. McCoy, and Chris J. Kanagy

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Conversion Factors

SI to Inch/Pound

Multiply	By	To obtain
	Length	
micrometer (µm)	1000	millimeter (mm)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
meter (m)	1.094	yard (yd)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
liter (L)	2.113	pint (pt)
liter (L)	1.057	quart (qt)
liter (L)	0.2642	gallon (gal)
barrel (bbl)	0.0238	gallon (gal)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:
°F=(1.8×°C)+32
Temperature in degrees Fahrenheit (°F) may be converted to degrees Celsius (°C) as follows:
°C=(°F-32)/1.8

Abbreviated water-quality units used in this report: Chemical concentrations are given in metric units. Chemical concentration is given in milligrams per liter (mg/L) and micrograms per liter (µg/L). “Milligrams per liter” is a unit expressing the concentration of chemical constituents in solution as weight (milligrams) of solute per unit volume (liter) of water. “Micrograms per liter” is a unit expressing the concentration of chemical constituents in solution as weight (micrograms) of solute per unit volume (liter) of water. For concentrations less than 7,000 mg/L, the numerical value is the same as for concentrations in parts per million.

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Abstract

On April 22, 2010, the explosion on and subsequent sinking of the Deepwater Horizon oil drilling platform resulted in the release of crude oil into the Gulf of Mexico. At least 4.4 million barrels had been released into the Gulf of Mexico through July 15, 2010, 10 to 29 percent of which was chemically dispersed, primarily using two dispersant formulations. Initially, the dispersant Corexit 9527 was used, and when existing stocks of that formulation were exhausted, Corexit 9500 was used. Over 1.8 million gallons of the two dispersants were applied in the first 3 months after the spill.

This report presents the development of an analytical method to analyze one of the primary surfactant components of both Corexit formulations, di(ethylhexyl) sodium sulfosuccinate (DOSS), the preliminary results, and the associated quality assurance/quality control (QA/QC) from samples collected from various points on the Gulf Coast between Texas and Florida. Seventy water samples and 8 field QC samples were collected before the predicted landfall of oil (pre-landfall) on the Gulf Coast, and 51 water samples and 10 field QC samples after the oil made landfall (post-landfall). Samples were collected in Teflon® bottles and stored at -20ºC until analysis. Extraction of whole-water samples used sorption onto a polytetrafluoroethylene (PTFE) filter to isolate DOSS, with subsequent 50 percent methanol/water elution of the combined dissolved and particulate DOSS fractions. High-performance liquid chromatography/tandem mass spectrometry (LC/MS/MS) was used to identify and quantify DOSS by the isotope dilution method, using a custom-synthesized $^{13}C_4$-DOSS labeled standard. Because of the ubiquitous presence of DOSS in laboratory reagent water, a chromatographic column was installed in the LC/MS/MS between the system pumps and the sample injector that separated this ambient background DOSS contamination from the sample DOSS, minimizing one source of blank contamination.

Laboratory and field QA/QC for pre-landfall samples included laboratory reagent spike and blank samples, a total of 34 replicate analyses for the 78 environmental and field blank samples, and 11 randomly chosen laboratory matrix spike samples. Laboratory and field QA/QC for post-landfall samples included laboratory reagent spike and blank samples, a laboratory "in-bottle" duplicate for each sample, and analysis of 24 randomly chosen laboratory matrix spike samples. Average DOSS recovery of 89±9.5 percent in all native (non-$^{13}C_4$-DOSS) spikes was

observed, with a mean relative percent difference between sample duplicates of 36 percent. The reporting limit for this analysis was 0.25 micrograms per liter due to blank limitations; DOSS was not detected in any samples collected in October (after oil landfall at certain study sites) above that concentration. It was detected prior to oil landfall above 0.25 micrograms per liter in 3 samples, but none exceeded the Environmental Protection Agency aquatic life criteria of 40 micrograms per liter.

Introduction

On April 22, 2010, the Deepwater Horizon oil drilling platform sank following an explosion and oil began leaking from the Macondo-1 well into the Gulf of Mexico (GOM) at a depth of approximately 1,500 meters (5,000 feet). By July 15, when the well head was capped, at least 4.4 million barrels (185 million gallons) had been released into the GOM (Lehr and others, 2010; Rosenbauer and others, 2010). In comparison, the total volume of crude oil released to Prince William Sound by the 1989 Exxon Valdez oil spill was 250,000 barrels (Michel and others, 2005). In order to solubilize the oil and mitigate the formation of a surface oil slick, over 1.8 million gallons of the chemical dispersants Corexit 9500 and 9527, produced by Nalco Company (Naperville, Ill.), were applied to surface oil (approx. 1.1 million gallons) and to the underwater plume at the well head (approx. 0.77 million gallons) (Lehr and others, 2010). These amounts represent the largest volume of dispersants ever used to respond to an oil spill, and it is the first occasion where dispersants have been used at depth. The Federal Interagency Solutions Group estimated that as of July 14, 2010, 16 percent (potential range 10 to 29 percent) of the spilled oil was chemically dispersed in the environment (Lehr and others, 2010).

Oil dispersants are chemical mixtures of solvents and surfactants designed to reduce oil slicks and emulsions by increasing the apparent solubility or accommodation of oil in seawater. Initially, existing stocks of Corexit 9527 were used to treat the GOM oil spill, but they were rapidly exhausted. Most of the applied dispersant was Corexit 9500, which is of similar composition, except that it does not contain 2-butoxyethanol, a solvent. A list of components for each Corexit formulation is publicly available (U.S. Environmental Protection Agency, 2010a). The primary surfactant components of both formulations are di(ethylhexyl) sodium sulfosuccinate (DOSS; Chemical Abstracts Service Number 577-11-7)[1], Tween 80, Tween 85, and Span 80 (Place and others, 2010). It is uncertain whether Corexit dispersants would be toxic to aquatic life at expected environmental concentrations—a battery of *in vitro* mammalian cytotoxicity assays estimated that the LC50 (lethal concentration 50) for Corexit is approximately 100 parts per million (ppm) (Judson and others, 2010), whereas the U.S. Environmental Protection Agency (EPA) found LC50 in 48- and 96-hour toxicity tests between 2.6 and 6.6 ppm (U.S. Environmental Protection Agency, 2010b). The EPA has estimated an aquatic life benchmark concentration at 40 parts per billion (40 ppb) (U.S. Environmental Protection Agency, 2010c). It is worth noting that dispersants can enhance the bioavailability of toxic oil components through increased solubilization (McIntosh and others, 2010).

On September 24, 2010, the U.S. Coast Guard requested that the U.S. Geological Survey (USGS) undertake chemical analyses of water samples collected before and after the oil spill made landfall at different points along the Gulf Coast. Samples for this study were collected according to protocols outlined by Wilde and others (2010). Another report addressing the Coast

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[1] This report contains CAS Registry Numbers®, which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS client services.

Guard’s request showed that most of the coastal oil contamination (sediments and tarballs) correlated with Macondo-1 oil found in Louisiana, Mississippi, and Alabama, which is consistent with the spatial extent of the oil spill (Rosenbauer and others, 2010). To fulfill the request of the U.S. Coast Guard, the above-mentioned analytical method is applied to assess the presence of DOSS, before and after the oil spill made landfall, in near-shore GOM surface waters along the United States coast from Texas to Florida.

Purpose and Scope

This report presents data requested by the U.S. Coast Guard for DOSS concentrations in Gulf of Mexico surface water samples collected before Macondo-1 oil made landfall (pre-landfall samples) along the coast , and surface water samples collected after Macondo-1 oil made landfall (post-landfall samples). Samples were collected from coastal waters in Texas, Louisiana, Mississippi, Alabama, and Florida before and after landfall of oil; where possible, pre- and post-landfall samples were collected from the same site.

In this report, a novel analytical method for the determination of sub-ppb concentrations of DOSS in seawater samples is presented; DOSS is isolated from a 1.5-milliliter (mL) water sample on a 0.2-micrometer (µm) Teflon (polytetrafluoroethylene, PTFE) filter, eluted with a solution of 50 percent methanol in water, and analyzed by liquid chromatography with tandem mass spectrometry (LC/MS/MS). Isotope dilution internal standard procedures are used to quantify DOSS and correct for losses during isolation and analysis, using a custom-synthesized stable-isotope analog ($^{13}C_4$-DOSS), which is added to water samples prior to DOSS isolation.

It is worth noting that the USGS did not have a published analytical method for DOSS prior to the beginning of this project, and in the absence of validated sampling protocols, samples were collected and stored frozen in 1-liter (L) Teflon® bottles while a methodology for determining DOSS at predicted ambient environmental concentrations was developed and validated. Therefore, sufficient information documenting methodology and method performance has also been included to aid in the interpretation of results from this occurrence study.

Materials and Methods

Sample Collection and Preservation

Samples were collected between May and October of 2010 as part of the USGS response assessing potential effects of the oil spill, concurrent with the development of an analytical method for this study. Therefore, holding times for seawater samples and the effects of specific container types could not be determined prior to sample receipt and storage. Care was taken to handle samples in a way that losses during collection and storage were likely to be minimized. All pre-landfall samples were analyzed within 8 months of collection, and all post-landfall samples were analyzed within 8 months of collection.

Samples were collected in 1-L Teflon® containers according to the procedures of Wilde and others (2010), wrapped in aluminum foil to exclude light, and frozen in the field when possible. Samples were shipped overnight to the National Water Quality Laboratory (NWQL), transferred to a freezer at -20°C on receipt, and held at this temperature under chain-of-custody procedures until analysis.

As part of the USGS strategy for sample collection in response to the Macondo-1 oil release, field blanks, field laboratory blanks, and equipment blanks were defined and specified for this and other analyses (Wilde and others, 2010).

Sample Isolation and Instrumental Analysis

Samples were thawed overnight in the dark one day prior to preparation. Once samples were thawed, approximately 0.4 percent (4 mL/L) methanol was added to each sample container to facilitate dissolution of any DOSS that had become adsorbed to the Teflon containers. Preliminary experiments showed that the addition of methanol increased DOSS recovery. Samples were placed on a rotating mixer for 5 minutes to thoroughly mix water and particles, and replicate 1.5-mL aliquots were immediately collected: two aliquots for replicate ambient DOSS determinations, and a third aliquot for a select subset of samples to determine DOSS recovery from a laboratory matrix spike sample. Each aliquot was added to a separate methanol-rinsed polypropylene syringe body fitted with a 0.2-micron pore size PTFE filter enclosed in a 22-millimeter (mm) polypropylene housing (Pall Life Sciences, part number CORP-4642). Prior to sample addition, the filter was rinsed twice with 4 mL of methanol (MeOH). For samples analyzed after December 15, 2010 (primarily pre-landfall samples collected in May and June), each filter was rinsed with 2 mL of MeOH, 4 mL of 1-millimolar (mM) formic acid, 4 mL of isopropanol (IPA), 4 mL of 50:50 MeOH:water and finally 2 mL of MeOH. This improved cleaning procedure lowered background contamination and, in future studies, should result in a substantial reduction in the method detection limit (MDL). A 15-microliter (µL) aliquot of a 100-µg/L $^{13}C_4$-DOSS solution was added to the sample (final concentration 1 µg/L) as an isotope-dilution standard (IDS) prior to analyte isolation. For the matrix spike sample, the 1.5-mL aliquot also was amended to 5 µg/L with a solution of unlabelled DOSS to assess compound recovery. After addition of these solutions, each sample was passed through the PTFE filter. Aqueous DOSS and DOSS-containing particles were retained on the filter, then eluted with 1.5 mL of 50 percent methanol in water. The eluant was amended with a 25-µL aliquot of 2.4 µg/mL ibuprofen-d_3 in methanol, which was added as an injection internal standard used to measure recovery of the $^{13}C_4$-DOSS. Sample extracts were refrigerated at approximately 4°C until analysis.

Sample analysis was carried out using an Agilent 1200 Series high-performance liquid chromatograph (LC) coupled to a 6410 Series triple quadrupole tandem mass spectrometer (MS/MS) operated in multiple-reaction monitoring mode. During method development, ambient DOSS was detected and traced to contamination in the LC/MS/MS system, either as an adsorbed component in the system itself or as an unavoidable contaminant in ultrapure water. The LC/MS/MS was modified by placing an Agilent Zorbax Eclipse column (3.1-mm diameter × 50-mm length; 5-µm particle size) between the LC pumping system and the injector module. The addition of a second column resulted in chromatographic separation of the ambient DOSS contamination from the sample DOSS and permitted identification and quantification in environmental water samples without requiring correction for this instrument background. A 100-µL aliquot of the unconcentrated sample extract was injected on an Agilent Zorbax Eclipse column (4.6 mm × 50 mm; 1.8 µm). Initially, the mobile phase consisted of 20 percent methanol/80 percent 1-mM ammonium formate in water and a flow rate of 0.65 mL/min. A multistep gradient program was applied to reach a final condition of 100 percent methanol after 7 minutes of elution, with DOSS eluting in the pure organic phase with a retention time of approximately 12 minutes. The mass spectrometer was operated under negative electrospray ionization conditions to produce a detectable DOSS $(M\text{-}H)^-$ precursor ion. The retention time of each target compound, coupled with the observation and measurement of two unique precursor-to-product transitions, were required for acceptable qualitative identification of each compound. Isotope-dilution internal standard quantification of DOSS was used, based on the response of the $^{13}C_4$-DOSS added to each sample prior to isolation (Antignac and others, 2003).

Determination of the Anionic Surfactant Di(ethylhexyl) Sodium Sulfosuccinate

Quality Assurance/Quality Control

A total of 112 pre-landfall samples were analyzed, comprising 70 distinct field sites, 34 replicates, and 8 field blank samples. Similarly, a total of 61 different post-landfall samples were analyzed for this study. These included GOM water from 48 distinct field sites, 3 of which had duplicate samples collected, and 10 blank (field blank and field laboratory blank) samples. Each of these samples was analyzed in duplicate ("in-bottle" duplicates), and a subset was fortified with DOSS to 5 µg/L for recovery analysis. The results from laboratory and field blank analyses were discussed in the Method Detection Limit section. All samples and duplicates were fortified with 1 µg/L of the IDS, $^{13}C_4$-DOSS, and IDS recovery was calculated relative to response of d_3-ibuprofen-d_3 in the standards. Average recovery of the $^{13}C_4$-DOSS IDS was 88.8±13.8 percent (mean ± relative standard deviation, n = 239). In addition, 21 percent (n = 35) of the samples had an aliquot fortified to 5 µg/L of DOSS, quantified relative to the IDS. Average recovery in these samples was 89±9.5 percent. This demonstrates robust method performance across the range of salinity encountered in GOM coastal waters (2–3.5 percent). Finally, although some DOSS was lost to sorption to the inside of the Teflon bottles, when 0.4 percent methanol was added to sample containers the recovery was acceptable. Three 1-L Teflon® bottles were filled with water from Dauphin Island, Ala., that was collected prior to the oil plume's landfall and fortified to 50 µg/L with DOSS, frozen at -20ºC for one week, then processed as described above. The average recovery in these samples was 75 percent, indicating that our method of sample storage was effective.

Method Detection Limit

The MDL for the measurement of DOSS was calculated according to EPA procedures using a low-level standard (MDL = 0.043 µg/L, n = 7) (U.S. Environmental Protection Agency, 2005). However, there was persistent contamination from sources in both the laboratory and the field. The laboratory reagent water used for preparation of chromatographic eluant was determined to be contaminated with DOSS. This source of contamination was removed from the analysis by the addition of a second column placed in front of the injection system (see Sample Isolation and Instrumental Analysis Section). However, DOSS is a ubiquitous contaminant (Benjamin Place, Oregon State University, oral commun., November 2010) and there was additional contamination not associated with laboratory reagent water, so while the addition of a second column decreased blank contamination substantially, it did not eliminate it. Laboratory blank samples that were prepared as described above had an average concentration of 0.086±0.044 µg/L. Laboratory blank samples collected with modified protocols for pre-rinsing of filters had an average concentration of 0.041±0.014 µg/L. Because the concentration of DOSS in laboratory blank samples exceeded the calculated MDL, data for post-landfall samples were censored at a reporting level (RL) of approximately 3 times the average blank concentration (0.25 μg/L) to minimize the potential for false positive results. The pre-landfall samples, which had lower background DOSS concentrations, were also censored at the higher level (0.25 μg/L) to maintain consistency within this report. Results for blanks collected in the field were not censored so the possibility of additional contamination during sample collection and other field activities could be assessed. The average DOSS concentration in blanks collected in the field was 0.12±0.044 μg/L (n = 10) for samples analyzed prior to December 15, 2010, and 0.075±0.026 μg/L (n = 8) for samples analyzed after that date. Neither result was statistically different from the associated laboratory blanks (Student's T-test, significant difference at p-value less than 0.05; Helsel and Hirsch, 2002). It is imperative that future studies requiring trace analysis of surfactants use sampling methods informed by these field and laboratory blank findings, by incorporating cleaning solvents and rinse water that have been purified to remove low-level contamination.

Determination of DOSS in Coastal Surface Water, May–June 2010

Pre-landfall water samples were collected from 70 sites in Texas, Louisiana, Mississippi, Alabama, and Florida in May and June of 2010 (table 1). DOSS was observed above the RL (0.25 μg/L) at two sites in Texas and one site in Louisiana during this period, at concentrations ranging from 0.34 to 3.66 μg/L. These concentrations all were substantially less than the provisional EPA aquatic life benchmark of 20 μg/L (U.S. Environmental Protection Agency, 2010c). At present, there is no evidence (Rosenbauer and others, 2010) that the extent of the surface oil plume had reached these sites by their respective sampling dates; however, the observed DOSS concentrations may have resulted from preparation or bulk transport of the dispersant from staging areas to the Macondo-1 site or from other, as yet unidentified, sources. An inadvertent chain of custody breach occurred for 31 of the samples within the pre-landfall set, as is noted in table 1. As a result of this breach, the affected samples thawed and were stored at 4ºC. These samples were analyzed and are reported herein.

Determination of DOSS in Coastal Surface Water, October 2010

Post-landfall water samples were collected from a total of 48 sites in Texas, Louisiana, Mississippi, Alabama, and Florida in October of 2010 (table 2). In three samples, DOSS was qualitatively identified, but concentrations did not exceed the RL of 0.25 μg/L, and the absence of detectable DOSS after reanalysis suggests that any DOSS present in these samples likely resulted from laboratory or field contamination. The sampling locations encompassed the estimated areal extent of the Macondo-1 oil spill, as derived from wind, ocean currents, aerial photography, and satellite imagery (Norse and others, 2010). In a related study, there was evidence of Macondo-1 oil in tarballs and sediment samples from many of the same sites in Louisiana, Mississippi, and Alabama (Rosenbauer and others, 2010).

These samples were collected between October 4 and 15, 2010, which is at least 80 days after the well head had been capped and substantial use of Corexit 9500 had been discontinued. It is likely that DOSS concentrations in GOM water were attenuated both by dilution and degradation processes. Although the rate of degradation was not quantified, the observed loss of DOSS in samples that were exposed to fluorescent light at the NWQL over the course of 24 hours likely resulted from photolysis. Therefore, it is not surprising that DOSS was not detected in sunlit GOM water samples. Although these results indicate that DOSS was not detectable in coastal GOM water 80 days after most dispersant use had ceased, it could be present at concentrations less than the RL or in other areas not sampled as a part of this study. Over 43 percent of the Corexit that was used in response to this oil spill was applied at depth (Lehr and others, 2010). If photolysis is an important mechanism of DOSS transformation in the GOM, DOSS could be more persistent in subsurface plumes where little sunlight penetrates. In addition, Corexit is a complex mixture of surfactants and solvents, and DOSS is the only component that was determined in this study. The absence of DOSS from most coastal samples does not imply that effects resulting from dispersant application did not occur, as other surfactant components could be more persistent, and their presence, persistence, and potential effects have yet to be assessed.

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Table 1. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in pre-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality control data—Continued.

[ID, identifier; µg/L, microgram per liter; %, percent; <, less than; n/a, not applicable; LA, Louisiana; TX, Texas; MS, Mississippi; AL, Alabama; FL, Florida; BLM, U.S. Bureau of Land Management; R, river; DWH, Deepwater Horizon; NS, National Seashore; SP, State Park; NWR, National Wildlife Refuge]

Site name	Site ID	Latitude	Longitude	Sample date	DOSS concentration (µg/L)			Recovery $^{13}C_4$-DOSS (%)			Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	
East Sabine, LA	TX-46	29.7489	-93.6633	5/10/2010	[1] <0.25			108			
Texas Point, TX	TX-47	29.6825	-93.9564	5/10/2010	[1] <0.25			120			
Sabine Lake, TX	TX-48	29.9283	-93.8714	5/10/2010	<0.25			111			
High Island, TX	TX-49	29.5567	-94.3683	5/10/2010	<0.25			113			
East Bay, TX	TX-50	29.5747	-94.5558	5/10/2010	0.47			115			
Galveston Island, TX	TX-51	29.3042	-94.7694	5/10/2010	<0.25			111			91
Galveston Island, TX (field replicate)	TX-51	29.3042	-94.7694	5/10/2010	0.69			117			
Galveston Island, TX (field replicate)	TX-51	29.3042	-94.7694	5/10/2010	3.66			114			
Trinity Bay, TX	TX-52	29.7356	-94.8364	5/11/2010	<0.25			127			
Bolivar Peninsula, TX	TX-53	29.3883	-94.7192	5/11/2010	<0.25			127			
Galveston Bay, TX	TX-54	29.4936	-94.9111	5/11/2010	<0.25			116			
West Bay, TX	TX-55	29.2147	-94.9539	5/11/2010	<0.25			124			
San Luis Pass, TX	TX-56	29.0867	-95.1086	5/11/2010	<0.25			99			
West Bay, TX; field blank[3]	TX-55	29.2147	-94.9539	5/11/2010	0.04			100			
Jean Lafitte National Park, LA	LA-22	29.7422	-90.1419	5/14/2010	[1] <0.25			99			
Cypremont Point, LA	LA-23	29.7350	-91.8536	5/13/2010	[1] <0.25			112			
Cypremont Point, LA (field replicate)	LA-23	29.7350	-91.8536	5/13/2010	[1] <0.25			118			
Cypremont Point, LA (field replicate)	LA-23	29.7350	-91.8536	5/13/2010	[1] <0.25			120			
Lake Felicity, LA	LA-24	29.3461	-90.4292	5/18/2010	<0.25			95			
Rockerfeller Refuge Beach, LA	LA-25	29.6356	-92.7672	5/13/2010	<0.25			119			
Sister Lake, LA	LA-26	29.2519	-90.9217	5/17/2010	<0.25			115			
Point Chevreuli, LA	LA-28	29.5733	-91.5378	5/13/2010	<0.25			111			
Crooked Bayou, LA	LA-29	29.7233	-89.7236	5/18/2010	<0.25			91			
Mississippi R. Gulf Outlet, LA	LA-30	29.6856	-89.3958	5/7/2010	<0.25			121			
Grand Isle Beach, State Park, LA	LA-31	29.2603	-89.9503	5/10/2010	<0.25			109			110
Grand Isle Beach, State Park, LA (field replicate)	LA-31	29.2603	-89.9503	5/10/2010	<0.25			123			
Grand Isle Beach, State Park, LA (field replicate)	LA-31	29.2603	-89.9503	5/10/2010	<0.25			127			
Mississippi R. at Main Pass, LA	LA-32	29.3206	-89.1819	5/11/2010	<0.25			88			
Breton Sound, LA	LA-33	29.5883	-89.6119	5/7/2010	<0.25			114			
Mississippi Sound at Grand Pass, LA	LA-34	30.1519	-89.2458	5/7/2010	<0.25			119			
Mississippi R. at South Pass, LA	LA-35	28.9975	-89.1489	5/7/2010	<0.25			93			
Mississippi R. at Southwest Pass, LA	LA-36	28.9375	-89.3989	5/7/2010	0.34			110			
Grand Isle Beach at State Park, LA; field blank[3]	LA-31	29.2603	-89.9503	5/10/2010	0.03			106			

Table 1. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in pre-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality control data—Continued.

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Site name	Site ID	Latitude	Longitude	Sample date	DOSS concentration (µg/L)			Recovery $^{13}C_4$-DOSS (%)			Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	
South Cat Island Beach, MS	MS-37	30.2192	-89.0797	5/7/2010	<0.25			82			
West Ship Island Beach, MS	MS-38	30.2075	-88.0797	5/7/2010	<0.25			72			
East Ship Island Beach, MS	MS-39	30.2328	-88.8925	5/7/2010	<0.25			81			
West Horn Island Beach, MS	MS-40	30.2403	-88.7350	5/8/2010	<0.25			79			
East Horn Island Beach, MS	MS-41	30.2225	-88.5925	5/8/2010	<0.25			84			
Petit Bois Island Beach, MS	MS-42	30.2022	-88.4267	5/8/2010	<0.25			77			
Pass Christian Beach, MS	MS-43	30.3161	-89.2361	5/8/2010	<0.25			91			
Biloxi Beach, MS	MS-44	30.3933	-88.2361	5/8/2010	[1]<0.25			82			
Pascagoula Beach, MS	MS-45	30.3428	-88.5478	5/8/2010	<0.25			87			95
Pascagoula Beach, MS (field replicate)	MS-45	30.3428	-88.5478	5/8/2010	[1]<0.25			89			
Pascagoula Beach, MS (field replicate)	MS-45	30.3428	-88.5478	5/8/2010	[1]<0.25			87			
Pascagoula Beach, MS; field blank[3]	MS-45	30.3428	-88.5478	5/8/2010	0.09			74			
West Dauphin Island, AL	AL-1	30.2274	-88.3264	5/8/2010	<0.25			80			
Dauphin Island, AL	AL-2	30.2488	-88.1842	5/9/2010	<0.25			106			104
Dauphin Island, AL (field replicate)	AL-2	30.2488	-88.1842	5/9/2010	<0.25			103			
Dauphin Island, AL (field replicate)	AL-2	30.2488	-88.1842	5/9/2010	<0.25			122			
Dauphin Island, AL	AL-3	30.2469	-88.0778	5/9/2010	[1]<0.25			100			
Fort Morgan, AL	AL-4	30.2249	-88.0083	5/8/2010	<0.25			105			
Fort Morgan, AL	AL-5	30.2305	-87.9044	5/8/2010	<0.25			101			
Gulf Shores, AL	AL-6	30.2413	-87.7303	5/8/2010	<0.25			99			
Orange Beach, AL	AL-7	30.2691	-87.5816	5/8/2010	<0.25			105			
DWH Oil Spill BLM-1	AL-8, BLM-1	30.2316	-87.9377	5/24/2010	<0.25			85			
DWH Oil Spill BLM-2	AL-9, BLM-2	30.2288	-87.8672	5/24/2010	<0.25			85			
Fort Morgan, AL	AL-10, BLM-3	30.2283	-87.8311	5/24/2010	<0.25			82			
Dauphin Island, AL-Field Blank[3]	AL-3	30.2469	-88.0778	5/9/2010	0.10			91			
Gulf Island NS near Navarre, FL	FL-1	30.3624	-86.9702	5/11/2010	[1]<0.25			92			
Henderson Beach SP near Destin, FL	FL-2	30.3829	-86.4428	5/11/2010	[1]<0.25			102			
Grayton Beach SP near Seaside, FL	FL-3	30.3241	-86.1551	5/12/2010	<0.25			83			
St. Andrew's SP near Panama City, FL	FL-4	30.1247	-85.7360	5/12/2010	<0.25	[2]<0.25	[2]<0.25	97	93	90	91
St. Joe P. SP near Port St. Joe, FL	FL-5	29.7792	-85.4085	5/13/2010	<0.25			111			
St. George Island SP near E. Point, FL	FL-6	29.6979	-84.7678	5/13/2010	<0.25			107			
St. George Island SP near E. Point, FL (field replicate)	FL-6	29.6979	-84.7678	5/13/2010	<0.25			110			
St. George Island SP near E. Point, FL (field replicate)	FL-6	29.6979	-84.7678	5/13/2010	<0.25			102			

Table 1. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in pre-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality control data—Continued.

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Site name	Site ID	Latitude	Longitude	Sample date	DOSS concentration (µg/L)			Recovery $^{13}C_4$-DOSS (%)			Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	
St. Mark's NWR near St. Marks, FL	FL-7	30.0742	-84.1804	5/18/2010	[1]<0.25			119			
Piney Point at Cedar Key	FL-8	29.1278	-83.0534	5/18/2010	<0.25			114			
Piney Point at Cedar Key (field replicate)	FL-8	29.1278	-83.0534	5/18/2010	<0.25			122			89
Ft. Island Gulf Beach	FL-9	28.9072	-82.6908	5/19/2010	<0.25			110			
Ft. Island Gulf Beach (field replicate)	FL-9	28.9072	-82.6908	5/19/2010	<0.25			113			
Ft. Desota Park	FL-10	27.6244	-82.7383	5/17/2010	<0.25			114			
Ft. Desota Park (field replicate)	FL-10	27.6244	-82.7383	5/17/2010	[1]<0.25			112			
Captiva Island	FL-11	26.5256	-82.1942	5/20/2010	<0.25			115			117
Captiva Island (field replicate)	FL-11	26.5256	-82.1942	5/20/2010	<0.25			122			
Captiva Island (field replicate)	FL-11	26.5256	-82.1942	5/20/2010	[1]<0.25			106			
Tiger Tail Beach at Marco Island	FL-12	25.9361	-81.7346	5/21/2010	<0.25			114			
Tiger Tail Beach at Marco Island (field replicate)	FL-12	25.9361	-81.7346	5/21/2010	[1]<0.25			108			
Northwest Cape Sable Beach near Flamingo	FL-13	25.2248	-81.1700	5/22/2010	<0.25			94			
Northwest Cape Sable Beach near Flamingo (field replicate)	FL-13	25.2248	-81.1700	5/22/2010	[1]<0.25			100			
Dry Tortugas National Park	FL-14	24.6271	-82.8736	5/20/2010	<0.25			100			
Dry Tortugas National Park (field replicate)	FL-14	24.6271	-82.8736	5/20/2010	[1]<0.25			85			
Bill Bags Cape near Key	FL-15	25.6674	-80.1555	6/11/2010	[1]<0.25			61			
Bill Bags Cape near Key (field replicate)	FL-15	25.6674	-80.1555	6/11/2010	[1]<0.25			85			
John U. Lloyd State Park	FL-16	26.0817	-80.1094	5/26/2010	<0.25	[2]<0.25	[2]<0.25	74	71	75	90
John U. Lloyd State Park (field replicate)	FL-16	26.0817	-80.1094	5/26/2010	[1]<0.25			59			
John U. Lloyd State Park (field replicate)	FL-16	26.0817	-80.1094	5/26/2010	[1]<0.25			70			
John D. MacArthur State Park	FL-17	26.8226	-80.0381	5/27/2010	[1]<0.25			66			
John D. MacArthur State Park (field replicate)	FL-17	26.8226	-80.0381	5/27/2010	[1]<0.25			67			
Coco Plum Beach near Marathon	FL-18	24.7293	-81.1700	5/24/2010	<0.25	[2]<0.25		111	84		114
Coco Plum Beach near Marathon (field replicate)	FL-18	24.7293	-81.1700	5/24/2010	[1]<0.25			93			
BLM tract 1 near Jupiter Inlet	FL-19	26.9561	-80.0817	6/16/2010	<0.25			73			84
BLM tract 1 near Jupiter Inlet (field replicate)	FL-19	26.9561	-80.0817	6/16/2010	<0.25			83			
BLM tract 2 near Jupiter Inlet	FL-20	26.9561	-80.0819	6/16/2010	<0.25			76			
BLM tract 2 near Jupiter Inlet (field replicate)	FL-20	26.9561	-80.0819	6/16/2010	<0.25			74			
BLM tract 1 near Park Key	FL-21	24.6506	-81.5575	6/9/2010	<0.25			67			95
BLM tract 1 near Park Key (field replicate)	FL-21	24.6506	-81.5575	6/9/2010	<0.25			78			
BLM tract 1 near Park Key (field replicate)	FL-21	24.6506	-81.5575	6/9/2010	<0.25			79			
BLM tract 1 near Park Key (field replicate)	FL-21	24.6506	-81.5575	6/9/2010	<0.25			78			
BLM tract 2 near Sugarloaf Key	FL-22	24.6175	-81.5436	6/9/2010	<0.25			66			
BLM tract 2 near Sugarloaf Key (field replicate)	FL-22	24.6175	-81.5436	6/9/2010	<0.25			73			

Table 1. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in pre-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality control data.

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Site name	Site ID	Latitude	Longitude	Sample date	DOSS concentration (µg/L)			Recovery $^{13}C_4$-DOSS (%)			Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	
BLM tract 3 near Sugarloaf Key	FL-23	24.6167	-81.5397	6/9/2010	<0.25			71			
BLM tract 3 near Sugarloaf Key (field replicate)	FL-23	24.6167	-81.5397	6/9/2010	<0.25			71			
BLM tract at Edmont Key	FL-24	27.6014	-82.7636	6/14/2010	<0.25			78			
BLM tract at Edmont Key (field replicate)	FL-24	27.6014	-82.7636	6/14/2010	[1] <0.25			78			
Lathrop Bayou near Panama City, FL	FL-25	30.04083	-85.4328	6/10/2010	<0.25			73			
Lathrop Bayou near Panama City, FL (field replicate)	FL-25	30.04083	-85.4328	6/10/2010	<0.25			68			
Great White Heron NWR	FL-26	24.70083	-81.54861	7/7/2010	[1] <0.25			85			
Captiva Island; field blank[3]	FL-11	26.5256	-82.1942	5/20/2010	[1] 0.0894			111			
Captiva Island; field blank (field replicate)[3]	FL-11	26.5256	-82.1942	5/20/2010	[1] 0.0701			105			
John U. Lloyd State Park; field blank[3]	FL-16	26.0817	-80.1094	5/26/2010	[1] 0.0844			61			
John U. Lloyd State Park; field blank (field replicate)[3]	FL-16	26.0817	-80.1094	5/26/2010	[1] 0.0903			60			

[1] Chain-of-custody was inadvertantly broken on the sample. The sample was refrigerated but not kept frozen.

[2] In-bottle replicate

[3] See Wilde and others (2010) for terms and definitions regarding field blanks, field lab blanks, equipment blanks, and other quality control terms.

Table 2. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in post-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality-control data—Continued.

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Site name	Site ID	Latitude	Longitude	Sample date	Concentration DOSS (µg/L)		Recovery $^{13}C_4$-DOSS (%)		Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 1	Replicate 2	
East Sabine, LA	TX-46	29.7489	-93.6633	10/6/2010	< 0.25	< 0.25	78	71	
Texas Point, TX	TX-47	29.6825	-93.9564	10/6/2010	< 0.25	< 0.25	83	90	
High Island, TX	TX-49	29.5567	-94.3683	10/7/2010	< 0.25	< 0.25	89	91	76
Galveston Island, TX	TX-51	29.3042	-94.7694	10/13/2010	< 0.25	< 0.25	80	81	89
Galveston Island, TX	TX-51	29.3042	-94.7694	10/13/2010	< 0.25	< 0.25	80	84	84
Bolivar Peninsula, TX	TX-53	29.3883	-94.7192	10/7/2010	< 0.25	< 0.25	94	99	
West Bay, TX	TX-55	29.2147	-94.9539	10/14/2010	< 0.25	< 0.25	82	87	88
San Luis Pass, TX	TX-56	29.0867	-95.1086	10/5/2010	< 0.25	< 0.25	83	77	85
West Bay, blank[1]	Field blank	29.2147	-94.9539	10/14/2010	0.08	0.09	86	84	
Houston Lab at Shenandoah, TX[1]	Field lab blank	n/a	n/a	10/4/2010	0.03	0.11	76	82	
Houston Lab at Shenandoah, TX[1]	Field lab blank	n/a	n/a	10/4/2010	0.12	0.09	86	78	
Jean Lafitte National Park, LA	LA-22	29.7422	-90.1419	10/13/2010	< 0.25	< 0.25	87	78	
Jean Lafitte National Park, LA	LA-22	29.7422	-90.1419	10/13/2010	< 0.25	< 0.25	82	80	87
Cypremont Point, LA	LA-23	29.7350	-91.8536	10/5/2010	< 0.25	< 0.25	75	69	
Lake Felicity, LA	LA-24	29.3461	-90.4292	10/12/2010	< 0.25	< 0.25	89	88	
Rockerfeller Refuge Beach, LA	LA-25	29.6356	-92.7672	10/7/2010	< 0.25	< 0.25	88	80	
Sister Lake, LA	LA-26	29.2519	-90.9217	10/8/2010	< 0.25	< 0.25	70	80	92
Point Chevreuli, LA	LA-28	29.5733	-91.5378	10/5/2010	< 0.25	< 0.25	80	71	
Crooked Bayou, LA	LA-29	29.7233	-89.7236	10/13/2010	< 0.25	< 0.25	110	97	
Mississippi R. Gulf Outlet, LA	LA-30	29.6856	-89.3958	10/12/2010	< 0.25	< 0.25	93	103	90
Grand Isle Beach at State Park, LA	LA-31	29.2603	-89.9503	10/14/2010	< 0.25	< 0.25	95	87	
Grand Isle Beach at State Park, LA	LA-31	29.2603	-89.9503	10/15/2010	< 0.25	< 0.25	92	91	
Mississippi R. at Main Pass, LA	LA-32	29.3206	-89.1819	10/7/2010	< 0.25	< 0.25	66	64	86
Breton Sound, LA	LA-33	29.5883	-89.6119	10/13/2010	< 0.25	< 0.25	89	102	
Mississippi Sound at Grand Pass, LA	LA-34	30.1519	-89.2458	10/11/2010	< 0.25	< 0.25	58	69	84
Mississippi R. at South Pass, LA	LA-35	28.9975	-89.1489	10/7/2010	< 0.25	< 0.25	71	75	
Mississippi R. at SW Pass, LA	LA-36	28.9375	-89.3989	10/14/2010	< 0.25	< 0.25	88	92	

Table 2. Concentration of di(ethylhexyl) sodium sulfosuccinate (DOSS) in post-landfall coastal Gulf of Mexico water samples between Texas and Florida and associated quality-control data—Continued.

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Site name	Site ID	Latitude	Longitude	Sample date	Concentration DOSS (µg/L)		Recovery $^{13}C_4$-DOSS (%)		Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 1	Replicate 2	
Mississippi River at Main Pass, blank[1]	Field blank	n/a	n/a	10/6/2010	0.20	0.16	65	60	
South Cat Island Beach, MS	MS-37	30.2192	-89.0797	10/14/2010	< 0.25	< 0.25	85	84	83
West Ship Island Beach, MS	MS-38	30.2075	-88.0797	10/14/2010	< 0.25	< 0.25	101	91	91
East Ship Island Beach, MS	MS-39	30.2328	-88.8925	10/11/2010	< 0.25	< 0.25	60	63	
West Horn Island Beach, MS	MS-40	30.2403	-88.7350	10/12/2010	< 0.25	< 0.25	102	80	
East Horn Island Beach, MS	MS-41	30.2225	-88.5925	10/12/2010	< 0.25	< 0.25	84	84	76
Petit Bois Island Beach, MS	MS-42	30.2022	-88.4267	10/13/2010	< 0.25	< 0.25	82	85	84
Pass Christian Beach, MS	MS-43	30.3161	-89.2361	10/8/2010	< 0.25	< 0.25	56	64	
Biloxi Beach, MS	MS-44	30.3933	-88.2361	10/7/2010	< 0.25	< 0.25	56	69	91
Pascagoula Beach, MS	MS-45	30.3428	-88.5478	10/14/2010	< 0.25	< 0.25	99	92	83
Biloxi Beach, equipment blank[1]	Field blank	30.3933	-88.2361	10/6/2010	0.21	0.16	84	84	
Biloxi Beach, sediment equipment blank[1]	Field blank	30.3933	-88.2361	10/6/2010	0.13	0.10	55	62	
West Dauphin Island, AL	AL-1	30.2274	-88.3264	10/13/2010	< 0.25	< 0.25	85	83	
Dauphin Island, AL	AL-2	30.2488	-88.1842	10/7/2010	< 0.25	< 0.25	62	66	88
Dauphin Island, AL	AL-3	30.2469	-88.0778	10/6/2010	< 0.25	< 0.25	68	71	
Fort Morgan, AL	AL-4	30.2249	-88.0083	10/12/2010	< 0.25	< 0.25	91	105	
Fort Morgan, AL	AL-5	30.2305	-87.9044	10/13/2010	< 0.25	< 0.25	62	63	
Gulf Shores, AL	AL-6	30.2413	-87.7303	10/14/2010	< 0.25	< 0.25	85	86	
Orange Beach, AL	AL-7	30.2691	-87.5816	10/14/2010	< 0.25	< 0.25	80	83	91
DWH Oil Spill BLM-1	AL-8, BLM-1	30.2316	-87.9377	10/13/2010	< 0.25	< 0.25	89	83	91
DWH Oil Spill BLM-2	AL-9, BLM-2	30.2288	-87.8672	10/14/2010	< 0.25	< 0.25	88	90	79
Fort Morgan, AL	AL-10, BLM-3	30.2283	-87.8311	10/14/2010	< 0.25	< 0.25	90	86	
Blank[1]	Field lab blank	n/a	n/a	10/5/2010	0.10	0.08	91	95	
Blank[1]	Field lab blank	n/a	n/a	10/5/2010	0.15	0.13	99	99	

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Site name	Site ID	Latitude	Longitude	Sample date	Concentration DOSS (µg/L)		Recovery $^{13}C_4$-DOSS (%)		Matrix spike recovery (%)
					Replicate 1	Replicate 2	Replicate 1	Replicate 2	
Gulf Island NS near Navarre, FL	FL-1	30.3624	-86.9702	10/4/2010	< 0.25	< 0.25	76	71	
Henderson Beach SP near Destin, FL	FL-2	30.3829	-86.4428	10/5/2010	< 0.25	< 0.25	81	82	
Grayton Beach SP near Seaside, FL	FL-3	30.3241	-86.1551	10/5/2010	< 0.25	< 0.25	71	77	78
St. Andrew's SP near Panama City, FL	FL-4	30.1247	-85.7360	10/11/2010	< 0.25	< 0.25	88	76	90
St. Joe P. SP near Port St. Joe, FL	FL-5	29.7792	-85.4085	10/13/2010	< 0.25	< 0.25	77	86	
St. George Island SP near E. Point, FL	FL-6	29.6979	-84.7678	10/6/2010	< 0.25	< 0.25	102	120	
St. Mark's NWR near St. Marks, FL	FL-7	30.0742	-84.7678	10/7/2010	< 0.25	< 0.25	90	92	77
Lathrop Bayou near Panama City, FL	FL-25	30.0408	-85.4328	10/12/2010	< 0.25	< 0.25	84	89	85
Equipment blank[1]	Field blank	30.0742	-84.7678	10/8/2010	0.10	0.08	78	75	
Equipment blank[1]	Field blank	30.0742	-84.7678	10/8/2010	0.09	0.11	78	76	

[1] See Wilde and others (2010) for terms and definitions regarding field blanks, field lab blanks, equipment blanks, and other quality control terms.