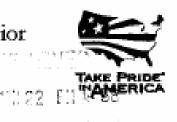
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U.S. GEOLOGICAL SURVEY Reston, VA 20192



ZI+ TYWG

In Reply Refer To: Mail Stop 409

JAN -8 2007

Timothy A. Wilkins Bracewell and Giuliani, LLP 111 Congress Avenue Suite 2300 Austin, TX 78701-4061

Subject: Complaint About the Quality of Information Disseminated to the Public

Dear Mr. Wilkins:

COP

This is our response to your Complaint about the Quality of Information Disseminated to the Public dated November 7, 2006, concerning the publication:

Mahler, B.J., Van Metre, P.C., and Wilson, J.T., 2004, Concentrations of Polycyclic Aromatic Hydrocarbons (PAHs) and Major and Trace Elements in Simulated Rainfall Runoff From Parking Lots, Austin, Texas, 2003: U.S. Geological Survey Open-File Report 2004-1208, version 2, 25 p.

The authors of Open-File Report 2004-1208 have responded to each of the 45 complaints you raised regarding the quality of the information in OFR 2004-1208. I find their response to be comprehensive to each of the questions raised. We have determined that a new version (Version 3) of the report will be released over the Internet as soon as possible. Where it is appropriate, the authors are making corrections or adding information to Version 3 for clarity. Their detailed response to each complaint is attached. We believe Version 3 addresses all the appropriate issues raised in the complaint.

Sincerely,

Robert m. Husch

Robert M. Hirsch Associate Director for Water Signing for the Director U.S. Geological Survey

CUS 200766-DO

- cc: AD-GIO Regional Hydrologist-CR Regional Director-CR Director-TX WSC
 - Director-TX WSC Chief, Office of Water Quality-MS-412 Chief Scientist for Hydrology-MS-436 Joye Durant-Office of the GIO

Memorandum

To:	Geographic Information Office, U.S. Geological Survey				
From:	Peter Van Metre and Barbara Mahler, USGS, Austin, Texas				
Date:	6/8/2007				
Re:	Response to "Complaint About Information Quality" regarding OFR 2004-1208				

This memorandum is written to reply to each of the 45 complaints under the USGS Guidelines for Ensuring the Quality of Information Disseminated to the Public under the U.S. Data Quality Act submitted by Bracewell & Giuliani LLP, Austin, Texas, on behalf of 10 companies in the sealcoat industry. Some changes are being made to the report in response to this complaint; a new version (version 3) will be released on the Internet as soon as practical.

Statement 1: Concentrations/runoff and scrapings from test plots

Response: The intent of the sentence was to say that for a given surface and sample type (e.g., scrapings from surfaces coated with asphalt-based sealcoat), concentrations from test plots were similar to those from in-use parking lots, as shown in figure 4. We agree that the sentence could be misinterpreted, and it will be removed.

Statement 2: Citation of a newspaper article

Response: The source of this background information will be cited as a City of Austin (COA) report (City of Austin, 2005).

Statement 3: Citation of a newspaper article

Response: See response to Statement 2. The source of this background information will be cited as a City of Austin (COA) report (City of Austin, 2005).

Statement 4: coal tar versus RT-12

Response: Industry product labels and Materials Safety Data Sheets (MSDS) routinely use the term "coal tar pitch" or "refined coal tar" rather than "RT-12" (for example, NEYRA industries "Jennite Coal Tar Emulsion" product (http://www.neyra.com/MSDSjenn.htm); NEYRA Industries "Tarconite" product (http://www.neyra.com/MSDStarc.htm); Star Industries "Star Seal Supreme" product (http://www.starseal.com/docs/pdf%20files/MSDS-SSS.pdf)). Industry web sites refer to "coal tar products" (for example, Tangent Rail Products, at http://www.tangentrail.com/tangent-p-s.html) and "coal tar emulsion" products (for example, Cooper Creek "Black Velvet Blacktop Sealer," at http://www.cooperscreekchemical.com/protective.html). The MacRae's Blue Book "Industrial Directory Yellow Pages" (www.macraesbluebook.com) located 61 companies when searched for "coal tar" (although not all provide sealcoat), and found none when searched for "RT-12." The term RT-12 is not widely used, even by industry, and does not have a CAS number. In contrast, coal tar and coal-tar pitch do have a CAS number: 65996-93-2. We conclude that our use of "coal-tar-emulsion-based" is accurate and consistent with industry naming conventions and see no utility in changing to the term RT-12. We will, however, add the word "refined" to "coal tar" in the introduction for clarity when the product is first described.

Statement 5: Reapplication 2–3 years

Response: The statement of reapplication every 2–3 years is based on product labels, applicator and product Web sites, and conversations with industry representatives during two industry briefings arranged by the City of Austin. For example, in a recent review of industry Web sites (November 22, 2006), we located the following:

Riegler Blacktop, Inc., recommends reapplying Brewer Cote coal-tar emulsion sealcoat every 2–3 years (<u>http://rieglerblacktop.com/commercialseal.html</u>);

Colorado Asphalt Services, Inc., recommends resealing every 2–3 years

(http://www.coloradoasphalt.com/FAQs.htm); Carolina Asphalt states that an "application should last 2–3 years" (http://www.carolinaasphalt.com/services.html); and Asphalt Sealcoat Manufacturers Association recommends resealing every 3–4 years (http://www.sealcoatmfg.org/whysealcoat.htm#When%20Should%20sealcoating%20be %20done).

These URLs will be added as references, and because not all companies recommend the 2–3-year reapplication schedule, we will reword the sentence to read, "Many companies recommend reapplication every 2–3 years." As the source of the information is the sealcoat product and applicator industry itself, we assume that it is reliable.

We are unaware of any independent, peer-reviewed studies of wear rates for different types of sealcoat and thus are not able to include any "relevant distinctions between anticipated longevity of different sealer products," per request. The statement was

included so that the reader would understand that sealcoat wears off, a point implicit in recommended reapplication rates.

None of the authors of the study recall making the statements in public presentations that were attributed to them in the Objection.

Statement 6: 660,000 gallons

Response: A check of the cited source Internet URL

(http://www.ci.austin.tx.us/watershed/bs_coaltar.htm) on December 11, 2006, indicates a link titled "City Presentation" from the cited URL to the presentation containing the annual-use estimate. However, whether the link is there or not is moot because the annual-use estimate now appears on page 2-1 of the COA report "PAHs in Austin, Texas Sediments and Coal-Tar Based Pavement Sealants" dated May 2005 (http://www.ci.austin.tx.us/watershed/downloads/coaltar_draft_pah_study.pdf). We will revise OFR 2004–1208 to reference this report because it is now posted on the Web. The information was provided by Bruce Lowry, of Wheeler Coatings Asphalt, LP., Round Rock, Tex. Mr. Lowry cited use of 650,000 gallons per year; an additional 10,000 gallons annually is sold off-the-shelf, a figure obtained by COA through polling of suppliers. As with other sources that are criticized in the Complaint as ". . . serves or intends to elevate unreliable information . . . ," we believe that this reference provides useful context information as part of the introduction.

Statement 7: coal tar PAH and fraction in sealcoat

Response: The statement is made to inform the reader that coal-tar sealcoat products contain high levels of PAH because (1) coal tar is predominantly PAH, and (2) sealcoat contains a large percentage of refined coal tar. The high PAH content of coal tar is supported by a large body of literature, one source of which is referenced in OFR 2004–1208. The percentage of refined coal tar in a sealcoat product is listed in the product MSDS; these numbers are consistent with tests performed by COA showing a mean of 8.4 percent "total" (sum of 16 parent) PAH in six coal-tar sealcoat products (note that the 16 parent PAHs make up only a fraction of true total PAH). In the interest of clarity, we will change the phrase "20- to 35-percent coal tar by weight" to "20- to 35-percent refined coal tar by weight," as this is the terminology we have found in the product MSDS and product descriptions (see Statement 4), including that provided by The Brewer Company, one of the Affected Parties

(<u>www.thebrewerco.com/pdf_Files/15000.pdf</u>, 15000 Series Pavement Sealers, Refined Coal Tar Pitch, 35 % by weight). We do not agree with the complaint and request for relief that a more detailed description of the "process of distillation and then emulsification . . ." is necessary.

Statement 8: COA product analyses

Response: The laboratory used by COA (DHL Analytical, Round Rock, Tex.) uses a certified U.S. Environmental Protection Agency method (EPA SW8270) for their PAH analyses; the subject data (PAH analyses of sealcoat products) are now included in a COA report

(http://www.ci.austin.tx.us/watershed/downloads/coaltar_draft_pah_study.pdf) (City of Austin, 2005). The reference will be changed to the COA report.

Statement 9: purpose

Response: The purpose of a study and the purpose of a report often are different, as is the case with this study and this report. Accordingly, USGS authors are instructed to include the purpose (or objective) of the *study* as distinct from the purpose of the *report*. Per common USGS report-writing practice, the purpose of the study is to be discussed in the introduction of a USGS report preceding the statement of purpose of the report. As a matter of USGS Water Resources Discipline policy

(http://water.usgs.gov/wid/FS_217-95/guide.html), all USGS-series reports are to include an explicit statement of the purpose (and scope) of the report, commonly in book reports under a second-order heading in the Introduction called "Purpose and Scope" The purpose of this report is to present the methods and data for the study, and thus addresses but one part of the broader-purpose study that includes interpretation of the data. The contents of OFR 2004–1208 are consistent with the stated purpose of the report. Because the form and content of the statements of purpose of the study and report are accurate and consistent with USGS policy and common USGS report-writing practice, no revisions were made.

Statement 10: application

Response: This objection refers to a brief, introductory statement included in the "Purpose and Scope" section of the report.

To remove any implication that "a single type of" coal-tar-emulsion sealer was applied to both test plots, the article "a" preceding "coal-tar-emulsion sealer" will be deleted from the subject sentence; and for parallel construction, the article "an" preceding "asphalt-emulsion sealer" also will be deleted.

Statement 11: frequency of test plot sampling, curing, and rainfall

Response: This objection refers to another statement in the "Purpose and Scope" section of the report that is intended to briefly summarize the approach of the study, and

thus by extension the scope of the material the reader will find in the report. This section of the report is not meant to provide a comprehensive description of methods. Regarding the issue of curing time, table 1 indicates that the test plots were sealed August 5 and 6, 2003, figure 2 and table 2 indicate that the first dissolved PAH samples were collected August 12, 2003, and figure 3 and table 3 indicate that the first particulate PAH samples were collected on August 21, 2003. The information in these figures and tables is sufficient for readers to determine that the time between application of the sealant and collection of the dissolved and particulate samples (the curing time) was 6 and 15 days, respectively. We do not agree that additional clarification in the text is warranted. The PCTC Guide Specification-PCTC01 specifies a minimum of 8 hours daylight curing time in good weather, and (in some cases) in excess of 24 hours in marginal weather conditions "before opening to traffic, and initially cure enough to drive over without damage to the sealcoat." If these curing times are sufficient to allow traffic to drive over the sealcoat without damage to the sealcoat, it is reasonable to believe a reader would conclude that curing times of 6 to 15 days are sufficient not to warrant "discussion of the curing time." We are unable to respond to the Affected Parties' request for a discussion of curing and PAH aging because their definition of "complete curing" is unknown, and the affects of curing and PAH aging are unknown.

Regarding the request for rainfall times and amounts and approximate temperatures, weather data are available to the public for numerous locations in Austin. National Weather Service rainfall data from Camp Mabry covering the project sampling period will be added to the report to indicate rainfall conditions in Austin prior to sampling.

Statement 12: scraping at most sites

Response: The objection refers to a statement in the "Purpose and Scope" section of the report which, as noted previously, is not intended to provide a comprehensive description of methods. Dates of scraping and washoff sampling and the surfaces that were sampled are provided in table 2.

The effect of rainfall history was not examined because PAHs are highly insoluble; thus we would not expect rainwater to extract (remove) these compounds from the adhered sealcoat surface. The scraping samples consist of the adhered sealcoat surface, thus rainfall is not expected to change the chemical composition of the scrapings.

The following text will be added to the report at the end of the last paragraph in the section "Sample-Collection Methods" to identify which three lots were excluded from the collection of scraping samples and explain their exclusion: "Scraping samples were not collected from the two unsealed concrete lots because it was assumed that the concrete contains almost no PAH (PAHs are hydrocarbons and thus are associated with fuels (coal, oil, wood) and combustion of fuels). One sample from an asphalt-emulsion sealed lot was not analyzed because the sample was corrupted." The technician who

collected the corrupted sample included a large amount of debris (leaves, grass cuttings, dirt, etc.) in the sample. This sample was not analyzed because the scrapings samples were designed to consist exclusively of adhered sealcoat.

Statement 13: dissolved PAH subset of sites

Response: PAHs are very hydrophobic compounds and their transport in streams is dominated by the particulate phase (suspended sediment) (Bradford and Horowitz, 1988; Foster and others, 2000; Ko and Baker, 2004). Therefore, the primary focus of the study was particle-phase transport from the parking lots. Nineteen samples were analyzed for dissolved-phase PAHs from a subset of sites, which included coal-taremulsion sealed, asphalt-emulsion sealed, and unsealed surfaces. The sites in the subset were selected on the basis of field convenience, while ensuring that at least one each of the three surface types was represented. Results for dissolved-phase samples are presented in table 3; the sites not sampled for dissolved-phase concentrations can be identified by their omission.

Statement 14: arranged for sealing

Response: The subject text in OFR 2004–1208 will be modified to present additional details per request. The text will read as follows:

The test plots were in the parking lot of Robert Mueller Municipal Airport (fig. 1). The airport was closed in 1999, and the parking lot has been in minimal use since then. Sometime before 1999, a coal-tar sealer was applied, which appeared to have worn off by the time of this study. Three of the test plots are 11- by 11-meter areas that were sealed during August 5–6, 2003 (table 1). Wheeler Coatings Asphalt, Inc., a commercial pavement-sealing company, on a voluntary basis, agreed to apply a refined coal-tar sealer (Tarconite, less than 34-percent coal tar by weight) to one site (TAR) and an asphalt sealer (Paveshield, less than 35-percent asphalt resin by weight) to one site (PAV). Wheeler personnel prepared the site for application by blowing off debris and priming oil and grease spots and applied the two commercial products according to manufacturers' specifications. Application of the coal-tar product also conformed to Federal Specifications R-P 335e Pitch, Coal Tar Emulsions and PCTC Guide Specification PCTC01. A retail refined coal-tar sealer (Henry Minuteman - Monsey, 33-percent coal tar by weight), a product available to homeowners for application to residential driveways, was applied to one site (MON) by City of Austin staff following the manufacturer's instructions. No sealer was applied to the control site (ASP). The test plots received virtually no vehicle traffic during the 2-month duration of the sampling.

Statement 15: choice of lots

Response: The relevent technical criteria used to select the sampling sites from the "universe of lots" are described in the paragraph cited. The only additional

"operational" criteria for sampling were (1) that information could be obtained regarding the date of application and type of sealcoat applied and (b) that permission to sample could be obtained. These operational criteria do not contribute information necessary to understand the study design and thus were not included in the text. We have assumed that the alleged contradiction refers to the phrase "represent a range of sealer ages." Data from tables 1 and 2 provide sufficient information to deduce that the six in-use, coal-tar-emulsion-sealed parking lots sampled reflect sealer ages ranging from approximately 2 to 50 months. Of the 12 samples (6 washoff and 6 scraping) from the six lots, six were collected from an approximately 2-month-old sealcoat surface, two from an approximately 6-month-old sealcoat surface, two from an approximately 35month-old sealcoat surface, and two from an approximately 50-month-old sealcoat surface. We do not believe that the aforementioned mix of sealer ages represented by our samples can be fairly characterized as "heavily overrepresented by very recently sealed lots with minimal curing time." We did not follow any "inclusion/exclusion criteria" related to sealer ages or any other parking lot characteristic. We do not believe that site-selection criteria or range of site ages warrants additional discussion in the report.

Statement 16: unreliable info on type and date of sealing

Response: The best information available was used. The information was cross-checked to the extent possible, for example, by checking the MSDS for the reported products, assuming that the MSDS are accurate on sealer contents. We do not believe any additional discussion is warranted.

Statement 17: 5 days no rain

Response: As noted in the response to Statement 11, rainfall data covering the project sampling period will be added to the report. We are not aware of any studies demonstrating the short- or long-term (6 months or more) effects of "curing and washoff of the removable material" alluded to by the Affected Parties. The attempt to collect particle-phase washoff samples on August 12, 2003, from the first lot sampled soon after a rain event produced insufficient mass of particles for analysis, so the sampling of particle-phase washoff was aborted and only dissolved PAHs were measured. This initial sampling attempt did not adhere to the 5-day limit because one objective of the test-plot sampling was to evaluate the effects of curing over the initial days and weeks after application, and an initial sampling within 1 week of application was part of the project plan.

Statement 18: dissolved only

Response: Please refer to our response to Statement 17. We attempted to collect particle samples on August 12, 2003, despite rainfall on the previous day. The plan called for the collection of the first sample from the test plots within 1 week of sealant application, and the sampling criteria called for a minimum of 5 days of no rain prior to sampling; the decision to attempt to collect a sample despite rainfall on the previous day was a judgment decision on the part of the Project Chief (Van Metre), who was required to balance the competing needs of the project plan and the sampling criteria. Van Metre concluded after the first sample (TAR) that they would not be able to collect enough material for particle analysis. This decision was based on his experience with suspended-sediment particle sampling; analysis of suspended-sediment concentration (SSC) is not done in the field, so the decision could not be made on the basis of SSC data. Dissolved samples, however, were collected on August 12, 2003, because sample volume for analysis was not a limitation. For one of those samples, MON, an SSC analysis was done (presented in table 5).

Other plots were not sampled, even if sufficient material might have been obtained, because the project plan specified sampling all four test plots on the same time interval (i.e., all sites to be sampled on the same day each time). Based on SSC concentrations measured at MON on August 12, 2003, the statement by the Affected Parties, "Had the same amount of water and area been sampled from these plots as was used on the TAR plot, sufficient material could have been collected on the filters to permit analysis for the other three plots" might be correct, at least for the MON site. However, as the SSC data was not available onsite, the decision not to continue collection of suspended sediments had to be made on the basis of professional judgment.

Regarding the Affected Parties' specific questions in paragraph 5, beginning with "In these circumstances, what basis...": As stated above, Van Metre concluded that there was insufficient sediment for analysis on the basis of visual inspection. Thus, no filters were processed and submitted to the laboratory for analysis from the remaining test plots. The decision to sample only dissolved concentrations on August 12, 2003, was made on the basis of the professional judgment of the Project Chief, and the reasoning (insufficient sediment for reliable particle analysis) is stated in the report. No changes were made to the report in response to Statement 18.

Statement 19: real rain sample at TAR

Response: Regarding the date, the text (p. 4, Sample-Collection Methods; paragraph 1) is incorrect and will be changed; the text was intended to refer to the September 26 sample, not August 26.

Regarding the issue of how samples were collected from the other test plots on September 26, deionized water was used as described in the "Sample-Collection Methods" section. It is implicit that the sample-collection methods were used except as noted otherwise in the text (TAR on September 26, 2003).

We agree that rainwater and deionized, distilled water are not chemically identical, and chemical differences between them might affect concentrations of PAHs in the dissolved phase. However, the three samples analyzed for dissolved PAH from TAR (a coal-tar-emulsion-sealed test plot for which one sample was collected using rainwater) each have a ΣPAH_{diss} that is about one-half that of the corresponding sample from MON, another coal-tar-emulsion-sealed test plot, suggesting that the chemical difference between the solvents did not play a major role in affecting ΣPAH_{diss} in this study. More importantly, however, the primary mechanism by which PAHs are transported from urban surfaces is via mobilization of the particulate phase, and mobilization and transport of fine particles is predominantly a physical process. We used the TAR September 26 sample because we wanted samples from all four test plots to be collected on the same day for each of the test-plot sampling events. Additionally, this was a research study investigating a previously unidentified contaminant source for which standard methods do not exist. Regarding the filtration method, we know of no reason why filtration should not work similarly with rainfall and deionized water. We developed the method for use in streams (Mahler and Van Metre, 2003a) and have used a similar method in a study of particle washoff from urban rooftops (Van Metre and Mahler, 2003), both of which are described in the peer-reviewed literature. The date will be corrected in the text, and the appropriate tables will be footnoted to remind the reader that the TAR September 26, 2003, sample was washed off with real rain, not simulated rain. No further changes were made to the report.

Statement 20:

Response: Mahler and Van Metre (2003a) is identified in the report as the source of filtration methods. Relevant details describing these methods as applied in this study are included in the "Sample-Collection Methods" section. No pre-filter is mentioned and none was used; a sentence will be added to that effect. As stated, 0.45-micron polytetrafluoroethylene (PTFE) filters were used. Regarding the question of glass-fiber filters (GFF) versus PTFE filters, we know of no peer-reviewed studies to support the statements of the Affected Parties and no reason why, or process by which, a PTFE filter might be "less efficient at trapping certain particulates and tend to selectively concentrate PAH-associated particulate" as proposed by the Affected Parties. Mahler and Van Metre (2003a) describes the technical advantages of PTFE filters over GFF filters. As noted in Mahler and Van Metre (2003a), PAH concentrations analyzed in sediment collected on PTFE filters tend to be higher than those analyzed in sediment collected on GFF filters, likely because when GFF filters are used, the filter must be

extracted along with the sediment, resulting in background interferences that affect quantification of the analyte. For this reason, the authors used PTFE filters for this and all other subsequent studies involving suspended-sediment analysis. Text to this effect will be added to the report. Additionally, Mahler and Van Metre (2003a) specified that the PTFE filter can be reused only for filtration of additional sediment from the same sample, not processed and reused for other samples, as implied in the Objection. Thus, in the report at issue, the methodology is clearly stated. PTFE filters were used consistently on all samples (for both sealed and unsealed lots) in the manner documented in peer-reviewed, published literature (Mahler and Van Metre, 2003a; Van Metre and Mahler, 2004). Given the purpose and scope of this report, it is not necessary or appropriate to discuss various methodologies that were not used.

Statement 21: SSC analyses

Response: The method reference for the suspended-sediment concentration (SSC) analysis will be added to the "Sample-Collection Methods" section (Guy, 1969). Additional text will be added providing the requested details of calculation of mean SSC concentrations.

The second paragraph of "Sample-Collection Methods" will be expanded into two paragraphs as follows:

Samples were pre-processed for analysis at the USGS laboratory in Austin. Water recovered during sampling was combined in a 50-L HDPE carboy equipped with a churn. Samples for measurement of suspended sediment concentration (SSC; Guy, 1969) were collected from the churn to allow quantification of the mass of sediment recovered in each sample. One SSC sample (250-milliliter [ml] plastic bottle) was collected at the start of processing. If more than 50 L of water was recovered, a second SSC sample was collected after the remaining water was added to the churn. For samples collected early in the study, this additional water was added to the churn after sufficient water had been removed by filtration to make room. This approach precludes calculation of a true volume-weighted mean, so for these samples the mean of the two SSC values was used to estimate total sediment recovered (table 5; "Mean of two samples" in column "Approach for calculating average concentration"). An adjustment was made in sample processing beginning with sample PAV collected September 26, 2003, and all samples processed thereafter [Note: sample TCO collected September 7, 2003, was incorrectly identified as a "volumeweighted mean" in version 2 of the report; this will be corrected in version 3.] If more than 50 L was recovered for processing, the first SSC sample was collected, and then the entire remaining volume of the first 50-L churn was filtered; then the remaining water was added and the second SSC sample was collected. For these samples, the mean SSC concentration is a volume-weighted mean of the two sample fractions (table 5; "Volumeweighted mean" in column "Approach for calculating average concentration"). The volume-weighted-mean concentrations accurately represent the overall mean even if concentrations in the two sample fractions are quite different. Where the mean of two samples was used and the SSC concentrations are relatively similar, the mean of the mean of the two samples should reasonably represent the total sample mean.

Samples were filtered through 0.45-micron pore size, polytetrafluoroethylene (PTFE) filters following the methods of Mahler and Van Metre (2003) [2003a in this response document]. No pre-filter was used. PTFE filters were chosen for use over glass-fiber filters, which must be extracted along with the sediment contained, producing an emulsion that interferes with quantification of the analyte (Mahler and Van Metre, 2003). A stainless steel plate filter holder was used for filtration of particulates for PAH analysis. [continuation of existing text]

Statement 22: Filters massaged...

Response: This method is described in Mahler and Van Metre (2003a), which is referenced in the report. Here is the description from the reference:

The PTFE filter must be sprayed with methanol before placing it on the filter holder to allow water to pass through the filter. Water is pumped through the filter until the filter clogs. The filter then is placed in a locking plastic bag, and a few milliliters of ultrapure or sample water is added. The filter is gently massaged until all the sediment has been removed and is in a slurry in the bottom of the bag. The filter can then be reused for filtration of additional sediment from the same sample, or a new filter can be used. The sediment slurry in the bottom of the bag is placed in a baked glass vial and chilled for shipment to the laboratory.

In the subject study, DI water was used as sample water, so DI water and sample water are the same. Regarding the question of sediment recovery, the filters are massaged until all visible sediment has been removed. Tests during development of the method in the late 1990s using spiked water slurries indicated nearly complete (more than 95 percent) recovery of sediment using PTFE filters; however, those results were not published. The method has been used and published in numerous studies, including studies for the U.S. EPA, the U.S. Air Force, and the U.S. Navy (for example, Van Metre and others, 2003a; Van Metre and Mahler, 2003; Van Metre and others, 2003b; Van Metre and Mahler, 2004]; Mahler and others, 2002; Mahler and Van Metre, 2003b; Mahler, 2003) without this particular question being raised. The description in the referenced paper (Mahler and Van Metre, 2003a) complies with OMB guidelines for transparency because the document contains sufficient detail for someone to replicate the analyses.

Statement 23: Surrogate compounds for PAH analyses

Response: Full quality-control data for each sample and batch of samples will be documented in tables in the revised report.

Statement 24: mass of sample extracted and dilutions

Response: Per this request we will add information regarding sample mass extracted, an identifier for dilution and the amount of the dilution if done, and the resulting reporting level for each sample. The following sentence will be added to the end of the "PAHs in the Particle Phase" section:

"Reporting level for each sample was calculated on the basis of sample mass, volume of extract, and the MRL.[minimum reporting level]"

Statement 25: duplicates vs. concentration

Response: Two sites were chosen for duplicate samples: a coal-tar sealed lot and an asphalt sealed lot. One of each type was chosen to reflect the different types of surfaces sampled. There was no concealed study design choice intended to promote bias in the results.

To avoid implying that the lots were chosen on the basis of prior knowledge of the expected PAH concentrations, the sentence referred to in this Objection will be revised to eliminate the phrase "one from a site with extremely elevated particulate PAH concentrations (greater than 4,000,000 μ g/kg total particulate PAH [Σ PAH_{part}])". Farther down in the same paragraph, the sentence

For one of the duplicate samples, Σ PAHpart differed by 8 percent (relative percent difference); for the second duplicate (sample with elevated concentrations), Σ PAHpart differed by 54 percent.

will be revised to read

For one of the duplicate samples, ΣPAH_{part} differed by 8 percent (relative percent difference). For the second duplicate, ΣPAH_{part} differed by 54 percent; this duplicate was from a site with extremely elevated particulate PAH concentrations (greater than 4,000,000 µg/kg total particulate PAH [ΣPAH_{part}])

Regarding additional detail requested on methodology requested, the revised text will read

Quality-control (QC) samples consist of environmental QC samples and internal laboratory QC samples (Table(s) X). Results of QC analyses are summarized below. Laboratory precision of the particulate PAH analysis was determined by analysis of two duplicate samples. Each duplicate was obtained by collecting and filtering a single sample using the same methods used for other samples. The resulting sediment slurry recovered was homogenized and split prior to extraction and analysis. Thus, the duplicates are designed to measure laboratory precision, not the repeatability of the field sample collection. For one of the duplicate samples, ΣPAH_{part} differed by 8 percent (relative percent difference); for the second duplicate (sample with elevated concentrations), ΣPAH_{part} differed by 54 percent; this duplicate sample was from a site with extremely elevated particulate PAH concentrations (greater than 4,000,000 µg/kg total particulate PAH [ΣPAH_{part}]). One equipment blank was analyzed for dissolved PAH. Three parent PAHs—fluoranthene, phenanthrene, and pyrene—were detected in the blank but at concentrations more than an order of magnitude less than the MRL. The concentrations in the blank were about one-half the concentrations in the environmental sample with the lowest concentrations (ZLK) and less than 1 percent of concentrations in the environmental sample with the highest concentrations (MON).

Statement 26: sum PAH and 2-methylnaphthalene

Response: The analytical method used by the USGS does not quantify 2methylnaphthalene individually but does quantify the mono-methylated naphthalene isomers, which include 2-methylnaphthalene. The latter compound, listed in table 2 of the report as "C-128 isomers, methylated Naphthalenes," was substituted for 2methylnaphthalene in the summation to obtain ΣPAH_{part} . The use of this compound rather than 2-methylnaphthalene could slightly overestimate ΣPAH_{part} because it might include other mono-methylated Naphthalene isomers in addition to 2methylnaphthalene, but the potential difference is very small. C-128 isomers, methylated Naphthalenes, were detected in only 11 of 40 analyses of particulate PAH (scraping, washoff samples, and duplicates) and the highest percentage of total PAH for which they accounted is 2.1 percent (the next highest percentage is 0.62 percent). We will add text to this effect at the end of the PAH methods section.

In the computation of ΣPAH_{part} , nondetections were assigned a value of zero, and estimated values were used at the value given. A statement to that effect will be added.

Statement 27: 8% versus 54% difference in duplicates

Response: A review of 42 duplicate samples of aquatic sediments analyzed by the USGS National Water Quality Assessment (NAWQA) Program in recent years using analytical methods similar to those used for this study (summarized in Van Metre and others, 2004) shows no relation between relative percent difference (RPD) and concentration (r-values between RPD and concentration for phenanthrene, fluoranthene, and benzo[*a*]pyrene were -0.074, -0.11, and -0.11, respectively). The highest concentration of fluoranthene among those 42 samples was 11,000 µg/kg (mean of duplicates), well below the mean fluoranthene concentration of 1,700,000 µg/kg in the duplicate sample from UNF. The median RPD of all (detected) PAH pairs in the 42 duplicate samples summarized in Van Metre and others (2004) was 11.9 percent (total number of individual PAH compared = 492). The larger difference in the duplicate sample from UNF was not considered to be sufficient information for us to conclude that error rates for samples with high concentrations are systematically greater. One alternate possibility is that a nonrepresentative split of this small-mass solid-phase sample was responsible for the large difference between duplicate analyses.

Statement 28: spike recovery rates

Response: As noted in our response to Statement 23, full quality-control data will be documented in tables in the revised report.

Statement 29: blank detections

Response: The sentence in the report following the sentences referred to in this Objection reads, "The detected concentrations ranged from 0.1 to 3.5 percent of the lowest concentration for that analyte in an environmental sample." This statement indicates that concentrations are very low relative to environmental concentrations and, therefore, are not considered to be an issue that warrants concern. Blank analysis results are now included in tables along with other quality-control data. Finally, analytes for which a blank detection occurred were used in the computation of total PAH, with no "blank correction" (subtraction of the concentrations (at 3.5 percent of less of the lowest measured concentration, as noted) are well below analytical precision.

Statement 30: E qualifier and request for more specifics.

Response: The E qualifier refers to an estimated value and was used to indicate one of several possible reasons for lower or unknown precision in reported values; this was standard practice in USGS data reports at the time OFR 2004–1208 was published. Per this request and other requests requesting more details on quality control (QC) (for example, dilution), we will provide additional detail in the text and table 2 of the report. The E qualifier will remain next to each estimated value as a general flag and a new Remarks column will be added for each constituent with these possible qualifiers:

Q = sample or batch of samples for which QC data could not be brought into control guidelines, I = interference issues from the sample matrix, n = peak identified and quantified below the Reporting Limit, m = constituent with highly variable QC data, S = constituent with no standard available, and d = dilution (table 2). The qualifiers Q, I, n, m, and S all lead to estimated values, those values that are detections but with lower or unknown precision.

Statement 31: use of PEC

Response: The comparison of particulate PAH concentrations (ΣPAH_{part}) recovered from simulated parking lot runoff to freshwater sediment quality guidelines (SQGs such as the probable effects concentration (PEC) is justified in our view because parking lots contribute directly or indirectly to runoff to urban streams, and particles washed from parking lots have the potential to be incorporated into freshwater sediments. We agree that dilution of parking lot particles occurs when they are introduced to streams and lakes; however, the amount of dilution (the "conversion factor" mentioned in the Objection) is unknown and likely highly variable. The comparisons to the PEC are made to provide some context for interpreting the relative strength of this potential source of sediment and PAHs to freshwater systems relative to screening levels for the receiving waters. If, for example, ΣPAH_{part} in particles in parking lot runoff were below any relevant SQGs for receiving water bodies, then we might reasonably conclude that their incorporation into sediments in those water bodies is unlikely to pose a risk to the benthic biota. We stand by our use of the PEC in the report, but will add text to the report to clarify our rationale for making these comparisons. The end of the first paragraph, column 1, page 6, will be revised to read as follows:

Concentrations of PAHs in particles in parking lot runoff are compared to the probable effect concentration (PEC), a screening-level benchmark. The PEC is the concentration of a contaminant in freshwater aquatic sediment above which adverse effects on sediment-dwelling organisms are expected to occur (MacDonald and others, 2000). The PEC for Σ PAH_{part} is 22,800 µg/kg. Although the particles in parking lot runoff do not in themselves constitute the aquatic sediments for which the PEC was developed, particles in parking lot runoff have the potential to be transported to streams and incorporated into aquatic sediments. Comparison of concentrations in particles associated with parking lot runoff to compared the PEC allows, for example, consideration of the amount of dilution needed for the PEC to be met. Concentrations of Σ PAH_{part} exceeded the PEC in all samples except the final sample collected at the control site.

Statement 32: more on PEC

Response: The probable effect concentration (PEC)—the concentration in freshwater sediments above which adverse effects on sediment-dwelling organisms are expected to occur (MacDonald and others, 2000)—was used as a screening-level benchmark. Explanatory language to this effect is in the report. The fact that PAH concentrations in all but one sample exceed this metric indicates that parking lot runoff, even from unsealed lots, is high in PAH relative to a metric developed for chemicals of concern (including PAH) in freshwater sediments; and particles in parking lot runoff that carry chemicals of concern have the potential to become incorporated into freshwater sediments. The factor by which the concentration in a sample exceeds the PEC, for example, indicates the amount of dilution necessary in the stream to reduce particle PAH concentrations to below the PEC.

Statement 33: average and units

Response: Regarding use of the word "average . . . concentrations," the text will be changed to read "mean . . . concentrations."

Per request, the standard deviation will be added to each mean value stated throughout the section "PAHs and Major and Trace Elements in Simulated Rainfall Runoff."

Regarding the question of units, the USGS National Water-Quality Laboratory routinely reports PAH in sediment analyses in μ g/kg (parts per billion), and our previous work as well as USGS NAWQA Program publications routinely use these units. Therefore, we used μ g/kg in this report. Because the very large magnitude of some of the concentrations in this particular data set renders them unwieldy, we changed to mg/kg (parts per million) in the article in *Environ. Sci. and Tech.* (Mahler and others, 2005). There is no reason that the choice of units would have an effect on rounding conventions (paragraph 4 of Objection) or on making the numbers "easier to differentiate and evaluate." The relative difference between 3,500,000 and 54,000 is the same as the relative difference between 3,500 and 54, and all four values reflect the same number of significant figures.

Regarding rounding conventions, we followed a rigid rounding convention in which we rounded all individual PAH concentrations to 2 significant figures, as presented in table 2. The rounding convention is documented in a primary reference on report-writing guidelines for USGS authors (Hansen, 1991). We chose 2 significant figures on the basis of our assessment of the level of analytical uncertainty (see comment 42 for more on rounding). The Σ PAH_{part} values shown in table 2 were computed by summing the unrounded individual PAH concentrations to avoid possible rounding errors when summing 13 individual constituents, then rounding the resulting sums. That is why there are small differences in some cases between Σ PAH_{part} in table 2 and the sum of the rounded values. This will be noted in the text.

Regarding the question of rounding large values (LBJ) to the nearest 100,000 compared to rounding small values to the nearest 100 (ZLK), rounding and analytical precision are relative, rather than at a constant level. This is particularly important when summing numbers of different magnitude. Quoting from Hansen (1991) referenced above: "If small numbers are added to (or subtracted from) large numbers of limited accuracy, the total should retain no more significant figures than are justified by the accuracy of the larger numbers. For example, in adding 356,000 (good to only three figures) and 1,420 (good also to three figures), the sum is 357,000, not 357,420. The figures that are dropped are within the limits of error of the larger number and are meaningless in the sum." Reporting the high concentrations down to units of 100s (LBJ, for example, at 8,969,800 µg/kg) would represent 5 significant figures, a level of precision that is not defensible. LBJ also provides an example of the effect of rounding of ΣPAH_{part} concentration after summation of unrounded individual PAH as opposed to before. As noted in the Objection, a value of 9,000,000 μ g/kg is presented in table 2, whereas the sum of rounded values is $8,917,000 \,\mu\text{g/kg}$, which presumably should round to 8,900,000, not to 9,000,000. The sum of unrounded PAH concentrations in this sample is 8,969,800 µg/kg, leading us by convention to round to 9,000,000.

Regarding the mean values for each type of surface sampled, these values were calculated from the rounded ΣPAH_{part} concentrations shown in table 2. Duplicate samples were averaged before calculating the mean to avoid "double counting" them. The mean value of ΣPAH_{part} from the six coal-tar-sealed, in-use lots is 3,453,333 µg/kg, hence, it was rounded to 3,500,000 µg/kg. Rounding procedures used to compute values for each table will be noted on the respective table in the revised report.

Statement 34: stats test at p=.1

coal tar and unsealed

Response: Text will be revised to provide the actual p-values rather than a threshold p-value (see response to Statement 35). The nonparametric Kruskal-Wallis test was used because the data are log-normally distributed.

Statement 35: small sample size for asphalt affects stats power

Response: The number of samples (sample size) for each type of lot was a matter of judgment by the authors. More coal-tar sealed lots were sampled than asphalt or unsealed lots because coal tar is known to contain a very high concentration of PAHs, and coal-tar sealant was the type most commonly used in the study area (Austin, Texas).

In response to the question about statistical comparisons of asphalt-sealed lots to other lots, we find that the statement in the original text that "Differences between concentrations from other groups were not significant in Kruskal-Wallis tests" is incorrect. The p-values indicating the strength of evidence for difference between each pair of surface types using Kruskal-Wallis and using the parametric t-test are shown in the table below:

Lot type (1)	Number of samples	Kruskal-Wallis p- value	t-test p-value
asphalt and coal tar	3 and 6	.07	.09
asphalt and unsealed	3 and 4	.03	.09

Summary of statistical test results for differences in $\Sigma \text{PAH}\textsc{part}$ by parking lot surface type

(1) asphalt = asphalt-based sealcoat; coal tar = coal-tar-based sealcoat; unsealed = unsealed asphalt or concrete

6 and 4

The two sentences in the report discussing the Kruskal-Wallis tests will be replaced by the following text:

.01

05

The concentration of ΣPAH_{part} in runoff samples from coal-tar-sealed parking lots was significantly greater than concentrations in samples from asphalt-sealed parking lots (p-value = .07) and unsealed parking lots (p-value = .01), and the concentration in samples

from asphalt-sealed parking lots was significantly greater than that from unsealed parking lots (p-value = .03).

Statement 36: runoff versus test plots similar

Response: Per Statement 1 response, we will remove this sentence. Per Relief Requested, some additional discussion of differences in results between test plots and in-use parking lots can be found in Mahler and others (2005).

Statement 37: comparison to PEC.

Response: Per response to Statements 31 and 32, we believe the comparison to sediment quality guidelines is a reasonable and valid way to provide context for concentrations of PAHs in parking lot runoff sediment.

Statement 38: dissolved concentrations

Response: See response to Statement 13 on the issue of sampling choices for dissolved analysis. As stated there, we focused on the particulate-phase analyses. Results for all dissolved-phase samples collected and analyzed are presented in table 3 of the report, and those sites not sampled can be identified easily. We do not believe the addition of a rationale to the text is necessary. The word "average" will be changed to "mean," and the standard deviation will be added to the sentence referred to in the Objection and elsewhere in the revised report.

Statement 39: similar dissolved by type.

Response: We stand by our characterization that concentrations of ΣPAH_{diss} are similar in runoff from coal-tar-sealed test plots and coal-tar-sealed in-use parking lots and also are similar in runoff from unsealed test plots and unsealed parking lots. We did not include "a thorough discussion of the implications of the approximate 4-fold increase in dissolved PAH concentrations" in samples from asphalt-sealed lots, as we do not know what those implications might be; and the primary purpose of this report, is ". . . to present sampling methods used for this study and the resulting chemical data." (page 2 of the report) Statement 40: scrapings compared with PEC

Response: Although we stand by our comparisons of particles in runoff to the PEC, we agree that comparison of scraping samples to the PEC is less logical, and therefore will remove this sentence and the following sentence. Again, we will acknowledge in the revised report that (a) we are using the PEC to provide a frame of reference or context for what differentiates concentrations that are high and potentially toxic from those that are below any benchmark threshold for toxicity; and (b) these sediments would likely be diluted upon reaching the stream—although the degree to which this would occur is unknown.

Statement 41: metals versus PEC

Response: Per our rationale for comparing particle PAH levels in runoff to PEC (Responses to Statements 31, 32, and 37), we believe that comparison of metals concentrations in particles in runoff to PECs is useful and valid, thus the statement is retained.

Statement 42: date of application recent

Response: We do not have information specifying the day of the month on which the lots were sealed. The first samples from in-use lots were collected on September 7, 2003 (table 2), not "around August 5" as mentioned in the Objection. Thus, a parking lot sealed in July 2003 would have undergone a minimum of 39 days and a maximum of 70 days of curing before sampling. The PCTC Guide Specification-PCTC01 specifies 8 hours drying time in "good daylight drying conditions before opening for traffic" to ". . . .cure enough to drive over without damage to the sealcoat" (24 hours or more in "marginal weather conditions"). Sample collection occurred well beyond the PCTC-specified curing time.

Statement 43: table 2 rounding

Response: Per respone to Statement 33, ΣPAH_{part} values were consistently rounded to two significant figures following summation of unrounded individual PAH concentrations. The decision to round to 2 digits was made on the basis of the precision of a very similar PAH method which had a median relative percent difference of 11.9 percent in 42 duplicate samples (total number of individual PAH compared = 492). Rounding to two significant figures can cause a change in value of as much as about 5 percent. To avoid possibly biasing summations of ΣPAH_{part} from cumulative rounding errors, unrounded individual PAH were used to calculate ΣPAH_{part} . This will be stated in the report. By convention, if the rounding decision was a tie, the value was rounded to the even digit (for example, 14,500 rounded to 14,000; 15,500 rounded to 16,000). The apparent discrepancy between the rounding of some values noted in the Objection is a result of summing rounded individual PAH concentrations instead of unrounded concentrations; unrounded concentrations should be used in all computations and the result then rounded. Regarding the specific examples presented in the Objection: For the unsealed test plot:

- Unrounded total = 407,650, rounded to 410,000
- Unrounded total = 14,500, rounded to 14,000

For asphalt-sealed in-use lots:

- Unrounded total = 830,170, rounded to 830,000
- Unrounded total = 769,580, rounded to 770,000

For coal-tar sealed in-use lots:

- Unrounded total = 8,969,800, rounded to 9,000,000
- Unrounded total = 516,990, rounded to 520,000

Our analysis of the effects of using rounded or unrounded individual PAH to calculate ΣPAH_{part} values indicates that the effect of the choice is negligible. The mean difference between ΣPAH_{part} values calculated with rounded individual PAH values (those listed in table 2) and ΣPAH_{part} values calculated with unrounded individual PAH values is 0.015 percent, and the maximum difference is 1.51 percent. In the sample noted above with an unrounded total (of unrounded individual PAH) of 830,170, the total of rounded individual PAH values is 830,360, or 0.70 percent greater. We contend our rounding approach is appropriate and defensible and make these comparisons to show that using any other rounding approach makes no significant difference in the outcome of the sampling.

Regarding the issue of "<, less than" and "E, estimated" in table 2 footnote, see response to Statement 30.

Regarding handling of nondetections, they were treated as zero values in summations of ΣPAH_{part} The practice of treating nondetections as one-half the detection limit has no statistical basis (Helsel, 2005). Regarding possible bias introduced by treating nondetections as zero values among samples with lower concentrations, any possible bias from this is small. The sum of the five compounds detected in 100 percent of the 27 runoff samples (duplicates included) accounts for a mean of 85 percent of ΣPAH_{part} (std. dev. = 5.6 percent); four other compounds were detected in 89 percent or more of samples. These high detection rates for compounds accounting for most of ΣPAH_{part} indicate that the treatment of nondetections in summations will not substantially alter the results. A sentence specifying the exclusion of nondetections and the inclusion of estimated values in the computation of ΣPAH_{part} will be added to the revised report text.

Statement 44: same as 43 but for dissolved.

Response: In the case of dissolved PAH, there is only one reason for an E qualifier in this set of data, that the detected value is below the MRL. We will adjust the footnote in the revised data table to add this information.

Statement 45: SSC and table 5.

Response: With the exception of the last comment, which pertains to footnote 4 and the incorrect date noted in footnote 2, the points raised in this Objection were addressed in our response to Statement 21. The date in footnote 2 was incorrect; it will be changed to 9/28/03 in the revised report. The indication that sample TCQ on 9/07/03 was a volume-weighted mean concentration also is incorrect. That sample was processed by the same approach used on other samples collected on 9/07/03 and 9/08/03. The table will be changed to indicate that this sample is a "Mean of two samples."

Regarding footnote 4: It is possible to estimate SSC using the mass of sediment recovered and the amount of water filtered. The Austin laboratory, however, does not do SSC analyses and is not approved by the USGS to do these analyses. Therefore, these data have not been published and are for internal information only. The decision to exclude two values of SSC for samples collected during processing was

made on the basis of professional judgment, supported in one case by the mass of sediment recovered during metals processing. One sample for which a decision was made to not use an SSC value was NWR collected 9/8/03. Two SSC samples had concentrations of 323 and 1,004 mg/L (table 5); the concentration estimated from the filtration done in processing the sample for analysis of metals was about 270 mg/L. The second SSC sample (1,004 mg/L) had the highest sand concentration (80 percent) of any sample collected during the study. The high sand content might explain the much larger total SSC concentration relative to the other sample and might suggest incomplete mixing when the sample was collected from the churn. We concluded that the sample likely was not representative, and thus it was not used. The other sample for which an SSC sample was not used was TAR on 9/26/03. Three SSC samples were collected during processing with concentrations of 16, 5, and 16 mg/L (table 5). This sample was collected from a test plot during a natural rainfall event rather than being collected while spraving with DI water. Six 9-L carboy containers were filled. The first SSC sample (16 mg/L) was collected from the first carboy filled in the field, and the second SSC sample (5 mg/L) was collected from the last (sixth) carboy filled in the field. The purpose in collecting these samples was to assess the extent of particle washoff during the course of a rain event. The third sample (16 mg/L) was collected from carboys 1-5, after they were combined in the 50-L churn. All sample processing (filtration) was done from carboys 1-5; therefore, the 16-mg/L concentration was deemed representative of the sample. A corrected footnote will be added to table 5 to clarify this situation, indicting

that this sample is of actual rainfall runoff and not simulated runoff and that sample 3 was from the water processed and, therefore, represents the mean concentration of SSC.

Regarding the Objection that these decisions "bias the subsequently computed PAH masses (Mahler and others, 2005) toward showing a difference between unpaved and coal-tar lots" the subject coal-tar lot (TAR) is a test plot, not an in-use lot, and therefore, was not used in the comparisons of concentrations or yields by parking lot type presented in Mahler and others (2005). The other sample (NWR) is an unsealed lot with low PAH concentrations. Including the higher-concentration sample that was ignored has the effect of roughly doubling the calculated sediment yield and, therefore, PAH yields. This change, however, does not change the rank of the PAH yield—it remains the highest yield from an unsealed lot and remains less than the yield from the lowest yield from a sealed lot (an asphalt-sealed lot). Inclusion of the higher concentration, therefore, would not alter the (nonparametric) statistical results of comparisons between types of lots, and exclusion of this SSC value does not introduce any bias into the results.

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