Peer Review Summary Report for the External Peer Review of *Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response*

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I. INTRODUCTION

The EnDyna Team was tasked with selecting three scientific experts to evaluate the research report *Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected during the Deepwater Horizon Spill Response*. The objective of this letter-style peer review was for the Department of the Interior's (DOI) Bureau of Safety and Environmental Enforcement (BSEE) to receive written comments from individual experts on the scientific merit of the research report, appropriateness of the assumptions made, and quality and relevance of the data. In assembling these peer reviewers and coordinating the peer review, the EnDyna Team was charged with evaluating the qualifications of peer review candidates, conducting a thorough conflict of interest (COI) screening process, independently selecting the peer reviewers, distributing review materials, maintaining contact with the peer reviewers, and developing a final peer review report. This section describes the EnDyna Team's selection process for external peer reviewers of the BSEE report.

I.1 Identification of Experts

The experts that were selected for this peer review were identified by literature searches of scientific journals, professional societies, and scientific meetings, as well as searches of our internal peer review database. As a result of these searches, the EnDyna Team identified a total of 10 potential scientific experts with expertise in oil spill response, in-situ burning, and chemical analyses of hydrocarbons. Of the 10 experts contacted, the EnDyna Team received four positive responses expressing interest and availability to participate. The remaining six candidates were either not available during the peer review timeframe or did not respond to our invitation. Interested candidates provided their name, contact information, and curriculum vitae (CV) containing their education, employment history, area(s) of expertise, research activities, recent service on advisory committees, publications, and awards.

I.2 Conflict of Interest (COI) Screening Process

The Endyna team initiated COI screening on the four interested individuals to ensure that the experts had no COI or appearance of the lack of impartiality. The screening was conducted in accordance with BSEE Peer Review Process Manual (dated August 2014) and involved each expert completing a COI questionnaire to determine if they were involved with any other work and/or organizations that might create a real or perceived COI for the current task.

I.3 Selection of Candidates

In selecting the peer reviewers, the EnDyna Team evaluated each candidate's credentials to select the experts that, collectively, covered the areas of expertise needed for this peer review, had no real or apparent COI or appearance of the lack of impartiality, and were available to complete the peer review within the desired timeframe. After review and consideration of the available information described above, the EnDyna Team selected the three peer reviewers and provided a list to BSEE. The names, affiliations, and expertise of the three peer reviewers are provided below.

Selected Peer Reviewers

1. **Merv Fingas, PhD.**
   Spill Science (retired from Environment Canada)

   Dr. Fingas has more than 40 years of experience working on oil and chemicals spills. Prior to working as a private consultant at Spill Science, he was Chief of the Emergencies Science Division of Environment Canada for over 30 years, where he conducted studies in oil chemistry, spill dynamics and behavior, spill treating agents, oil spill remote sensing and detection, oil spill tracking and sampling, in-situ burning, and chemical counter-terrorism. Dr. Fingas has a Ph.D. in Environmental Sciences from McGill University.

2. **Christopher M. Reddy, Ph.D.**
   Woods Hole Oceanographic Institution

   Dr. Reddy is a Senior Scientist in the Department of Marine Chemistry and Geochemistry at Woods Hole Oceanographic Institution (WHOI). He also serves as the Director of the Coastal Ocean Institute at WHOI. His research interests include the effects of marine pollutants on the environment, particularly following major oil spills and he has developed advance sampling techniques to identify the source, transport, and ultimate fate of petroleum hydrocarbons in the coastal and open ocean. He also has a research interest in marine oil spill fingerprinting. Dr. Reddy has a Ph.D. in Chemical Oceanography from the University of Rhode Island.

3. **Alan M. Shiller, Ph.D.**
   University of Southern Mississippi

   Dr. Shiller is a Professor at the University of Southern Mississippi (USM). He also serves as the Director of the Center for Trace Analysis at USM, where he conducts research in the areas of geochemistry including oil spill effects on ocean chemistry, trace elements in natural waters, marine and estuarine chemistry, chemistry of rivers and weathering, and global carbon system. He recently studied both direct and indirect chemical effects associated with the *Deepwater Horizon* oil spill event. This included evaluating the presence of metals in the environment to fingerprint or identify the origin of a sample of oil. He has also helped identify and describe oil spills through investigations of polycyclic aromatic hydrocarbons. Dr. Shiller has Ph.D. in Oceanography from Scripps Institution of Oceanography, University of California.

II. BACKGROUND ON RESEARCH REPORT

BSEE has requested an external peer review of the final research report entitled, *Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected during the Deepwater Horizon Spill Response*, which was prepared by the National Oceanic and Atmospheric Administration (NOAA) and Louisiana State University (LSU). Part of BSEE's research is committed to ensuring that functional, safe, and environmentally responsible oil spill response methods are identified and used under appropriate conditions. In-situ burning is a practical oil spill response, and was used on an unprecedented scale during the response to the Macondo oil spill incident. Afterwards, tar balls were collected in various locations, including in deep sea trawls of the royal red shrimp fishery. As a result, BSEE contracted NOAA and LSU researchers to perform physical and chemical analyses of surface collected oil, field in-situ burn residues, deep water tar balls, and experimentally burned field collected oil. The results of these analyses are interpreted to characterize physical and chemical changes in oil during combustion and other weathering processes.

One note: researchers had to conduct this project with the limited samples that were available and didn't have any influence concerning the method of their collection. This retrospective study tried to meld the in-situ collections with lab-generated results, to the maximum extent possible.

III. CHARGE QUESTIONS

The purpose of this review was to obtain written comments from individual experts on the research report entitled, *Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected during the Deepwater Horizon Spill Response.* Each reviewer was charged with evaluating the report, providing their overall impressions of the scientific merit of the report, responding to six charge questions, and providing any other specific comments on the report. The six charge questions provided to the reviewers are included below.

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

    a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?
    b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

    a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.
    b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.
    c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

4. Were there any critical results or issues that were not discussed in the report?

5. Does this report present sufficient new data and knowledge, and are the findings useful?

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

IV. SUMMARY OF PEER REVIEWERS COMMENTS

IV.1 General Impressions

All reviewers generally thought the report was good and that it contained useful information. One reviewer even commended the authors for their efforts in avoiding ambiguity and highlighting the challenges encountered in this study. The reviewers also provided comments on specific issues and suggested potential improvements to the report.

Some general comments made included that the report could have been improved by estimating the extent of burning, identifying correlations in content to the extent of burning, and comparing to the Wang pyrogenic index. Another reviewer recommended adding an executive summary, providing a more pronounced summary/conclusions section, and including the recommendations as a separate section. This reviewer also questioned if different analytical approaches and usage of other compounds or bulk properties could have been used to identify burn residues. Another reviewer noted that there is a lack of supporting data in the report. The authors did provide figures and a table of summary statistics but the reviewer thought that it would be useful to include the quantitative data in an appendix.

IV.2 Responses to Charge Questions

1. ***Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?***

The reviewers, in general, agreed that the objectives of the study were clearly defined. However, one reviewer noted that the objectives could have been more specific and additional tasks could have been included to estimate the extent of burning, identify correlations in content to the extent of burning, compare to the Wang pyrogenic index, etc.

2. ***Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:***

One reviewer provided specific comments under this question and subquestions 2a and 2b, while the other two reviewers provided their responses under subquestions 2a and 2b. The reviewer noted that the gas chromatography analysis is appropriately designed and clearly described. However, the reviewer expressed concerns about the definition and usage of the asphaltene content. The reviewer also provided several comments regarding the asphaltenes, specifically, the use of the operationally defined method. The reviewer also suggest providing additional information on the precipitation and gravimetric analysis of asphaltenes and discussing if other methods exists for tracking asphaltene-like materials. Lastly, the reviewer emphasized that any changes in the samples be normalized to hopane as this is standard practice.

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

All reviewers generally found the methods appropriate and adequately described, but had some suggestions or concerns. One reviewer recommended that the methods be more specific, such as including that asphalt, resin, density, and viscosity all be measured. Another reviewer noted that beyond normalizing samples to hopane, the biggest issue with the study is the limited breadth of the target list. This does not mean that the study is defective, but it is limited. The reviewer suggests that in the Macondo well oil, the authors could account for total saturates and PAHs at 5 and 1%, respectively and that the authors should note that their results are based on tracking 5% of the mass. Another reviewer suggested adding references to the asphaltene extraction, the GC/MS characterization, and modified Fossil Fuel Pollution Index (FFPI). The reviewer also suggested describing the QA/QC procedures and criteria used to classify the samples ("Match", "Probable Match", etc.). In addition, the reviewer noted that it would be appropriate to have some discussion of the controlled burn methodology and useful to see a more explicit discussion of reproducibility of the results.

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

The reviewers, in general, agreed with the techniques used for forensic interpretation. However, one reviewer suggested adding information on the Wang pyrogenic index. Another reviewer noted that although the interpretive techniques were fine, little quantitative data were provided in the report. The authors did provide figures and a table of summary statistics but the reviewer felt that it was difficult to quantify the information in this format. This reviewer also noted that the criteria used to classify the samples ("Match", "Probable Match", etc.) were not provided and thus, could not be evaluated.

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

The reviewers provided varying responses to this charge question. One reviewer thought that some of the information was hard to believe, including the conclusions about the asphaltene and FFPI index. Another reviewer noted that the GC-MS analysis can only account for 5% of the mass, the asphaltene content may be incorporating weathering products, and that the results have not been normalized to hopane. This reviewer suggests acknowledging these issues in the study and addressing them if possible. Another reviewer was concerned that neither the QA/QC procedures nor basic data were provided in the report.

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

One reviewer noted that measuring the Wang pyrogenic index followed by estimating the completeness of combustion and then correlating this to the composition of resins, asphaltenes, etc. in the samples would have been better than the FFPI. Another reviewer recommended using the GC-MS data that are available to reexamine and expand on the PAH data and also noted that there is more available besides the FFPI. Another reviewer noted that the report provides some useful diagnostics to identify in-situ burned oil residue. However, the reviewer would have preferred to see more quantitative information on how the burning affected some of these indicators.

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

The reviewers generally agreed that the results were properly used to distinguish in-situ burn residues, but had some comments or questions. One reviewer noted that the study could have been strengthened by calculating the Wang pyrogenic index and estimating the degree of combustion. Another reviewer commented that the results were interesting and realistic, but suggested the authors explore how and why, and if there are examples in the combustion literature that would support these findings. Another reviewer noted that even though Table 3 presents a summary of the means used for distinguishing burn residues, the reviewer would have liked to have seen the actual data.

4. Were there any critical results or issues that were not discussed in the report?

No new issues were identified, but all reviewers referred the authors to their previous comments and recommendations. One reviewer reiterated that it would have been better to measure the Wang pyrogenic index, estimate the completeness of combustion, and then correlate the completeness of combustion to the composition of resins, asphaltenes, etc. in the samples. Another reviewer noted that the literature should be verified and another reviewer restated the lack of data presentation.

5. Does this report present sufficient new data and knowledge, and are the findings useful?

All reviewers agreed that the findings were useful. However, one reviewer noted that additional tables of specific results should have been presented to allow the readers to judge the results for themselves, which is similar to a previous comment made by another reviewer. Another reviewer commented that although this is a good start, there was not a concerted and dedicated effort to collect field samples so this study was limited. The reviewer recommends additional synthesis and distillation of data be conducted if time and budget permits. This reviewer also suggests that perhaps some or all of the samples could be analyzed with techniques that could add to the GC-MS results.

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

Two of the reviewers generally agreed that there are not many studies on this topic beyond those cited in this document. However, one reviewer noted that there are other studies on the *Deepwater Horizon* event that should be considered. Specifically, the reviewer mentioned that there is a vast collection of publications on the formation of PAHs from incomplete combustion of organic matter, which could be used for interpreting the PAH data. Another reviewer noted that there have been extensive studies on burn residue and pyrogenic index, which is only somewhat acknowledged in this study. A list of studies on these topics were provided by the reviewer and included in the Specific Observations section (Section V.3) for the authors to consider.

V. PEER REVIEWER COMMENTS BY CHARGE QUESTIONS

V.1 General Impressions

Merv Fingas

The report is quite good and a good scientific approach was used. The identification of the oil is good and does correctly identify residues that can be associated with the *Deepwater Horizon*. The identification is based on only a few parameters, however, should be adequate for the purpose.

The report could have been improved by estimating the extent of burning, identifying correlations in content to the extent of burning, and comparing to the Wang pyrogenic index. The extent of burning could have been estimated by using the Wang pyrogenic index.

It is unfortunate that 'original' samples of the oil were not available to act as starting samples, however, this is not the fault of the principal investigator. The presence of original samples and tests on their burns would have enabled a much greater extent of comparison and study.

Christopher M. Reddy

I enjoyed reading this document and learned several useful details. I also commend the authors for their efforts in avoiding ambiguity and highlighting the challenges they faced in this study. Overall, it captures the needs of the charge although there are numerous opportunities to improve it.

Briefly, I would recommend an executive summary and a more pronounced summary/conclusions. Please make the recommendations a separate section. Also, beyond better sampling, what could be done to provide greater insights into the identification of burn residues? That is, different analytical approaches and usage of other compounds or bulk properties.

Could faster turnaround times be provided so that burns can be tweaked with analytical data in near real time? Can responders who have a set list of observations/tasks record georeferenced data and images?

Collectively, more data with greater breadth in synthesis and interpretation should lead to more effective burns.

Alan M. Shiller

Overall this is an interesting report examining in-situ burn residue composition and seeking to determine if there exist diagnostic compositional indicators of burn residues. The report is somewhat constrained due to the limited samples that were available of in-situ burn residue as well as of MC252 oil. Also, only a few experiments were performed. The presentation is generally clear and sound. The objectives, methods, and results are fairly clear. My main comments relate to lack of supporting information. There are some places where further

references to the literature and more details about methods would be helpful. The main concern I have is the limited presentation of supporting data. There are a few graphical presentations of results and one supporting table of summary statistics. It would be useful, however, if all of the data were available in an appendix. There are also a few other minor additions that would be helpful and which I describe below. In summary, this is a generally good report and with some straight forward modifications this would far more useful.

V.2 Responses to Charge Questions

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

Merv Fingas

The objectives of the study are perhaps not as specific as they could have been. After the general charge to identify the physical and chemical changes in the oil, it might have been prudent to be more specific and add tasks such as estimate the extent of burning, identify correlations in content to the extent of burning, compare to the Wang pyrogenic index, etc.

Christopher M. Reddy

The authors have done an excellent job writing the necessary background and introduction to this report. Coupled with clear and comprehensive objectives, it is a well-organized document with an excellent description of the objectives.

Alan M. Shiller

The objectives of the project are straight forward and clearly laid out, mainly through the list of Tasks on page 10 of the report.

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

Merv Fingas

See specific comments under 2a and 2b.

Christopher M. Reddy

Yes and no. For the gas chromatography analysis, it was appropriately designed and clearly described.

I am concerned about the definition and usage of the asphaltene content. As there is no set structure (and there is an active and ongoing debate about it and many aspects of the asphaltenes), approaches to measure them in oils must be operationally defined. Due to ease and that it is chemically reasonable, the asphaltenes are often measured by the amount of material

that can be initially extracted in toluene or dichloromethane but then will precipitate in hexane or heptane. The asphaltenes are measured gravimetrically. This approach is almost entirely used during upstream and downstream and rarely for environmental analysis. I have several comments regarding the asphaltenes:

1) Using this operationally defined method, the authors may be measuring apples and oranges. As noted by the authors and others, the product that leaked from the Macondo well was light, sweet crude, which would presumably indicate trace amounts of asphaltenes. The authors measured less than 1%, a very reasonable result.
2) However, more recent studies on the *Deepwater Horizon* disaster by Aeppli et al. (2012; *Environmental Science and Technology*) and Lewan et al. (2014; *Organic Geochemistry*) have shown that polar, enriched in oxygen content, non-GC amenable materials insoluble in hexane are formed by weathering. Aeppli nicely showed this increase in asphaltene-like (but not asphaltenes, which are formed in reservoirs) relative to the conserved internal standard, hopane. So it's entirely possible that any changes in the operationally defined method may capture preferential enrichment of the native asphaltenes and also weathering processes that are forming another material also detected with this method. I recommend a revaluation of the results presented in this study.

3) What is the precision and accuracy of precipitation and gravimetric analysis of asphaltenes, especially at low levels? Please discuss.

4) What about other methods for tracking asphaltene-like materials? I would consider bulk oxygen and nitrogen, liquid chromatography, or spectrometric analysis as well as measuring the black or elemental carbon content.

5) This was mentioned previously, but it is absolutely paramount that any changes in the samples are normalized to hopane. This is standard practice and will likely shed greater light on the formation of the parent. Otherwise, I would consider this study incomplete.

Alan M. Shiller

See specific comments under 2a and 2b.

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

Merv Fingas

Yes, however again they might have been more specific such as specifying that the asphalt, resin, density, and viscosity all be measured.

Christopher M. Reddy

The authors are experts in the analysis of petroleum hydrocarbons and other organic chemicals. Their approach for using gas chromatography with mass spectral detection (GC-MS) is a very traditional, mature, and sound method. I would have done exactly what they would have done first. Beyond normalizing to hopane, the biggest issue at stake is the limited breadth of the target list. In the Macondo well oil, the authors could account for total saturates and PAHs at 5 and 1%, respectively, of the total mass. And with weathering and loss of the "front end," the amount of material that can be targeted with GC-MS gets smaller. This does not mean that this study is defective but it is limited.

While there are other approaches to measure oil residues, most are not mainstream. It would be unfair to expect the authors to use the most cutting edge instruments, but great if they could. The authors do need to note they are basing their results by tracking ~5% of the mass and future studies should consider other methods.

Alan M. Shiller

In general, the methods were adequately described. That said, there were some places that additional detail would be helpful. In particular, references to the asphaltene extraction, the GC/MS characterization, and modified FFPI would be useful. QA/QC procedures should also be described. The criteria for "Match", "Probable Match", etc. need to be described. On page 25, there is mention of a "simulated distillation of MC252 oil" that is not described in the methods.

It would also be appropriate to have some discussion of the controlled burn methodology. One wonders if there are some differences in what happens in open water where there is a large capacity of the water to dissipate heat at the bottom of the burning residue versus the experiments which were conducted in plastic pools with more limited heat dissipation ability. Additionally, the anecdotal communication presented on page 7, suggests that perhaps some burn components sink and some float. The controlled burn experiments would not be able to distinguish those components.

Finally, there only seemed to be a couple of controlled burn experiments (the exact number was unclear, possibly 3 replicates of surrogate oil and 3 replicates of emulsified MC252). A more explicit discussion of how reproducible the results were would be of use.

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

Merv Fingas

Yes, again there could have been more specific additions such as the Wang pyrogenic index - the identification of the oil was good and complete as carried out.

Christopher M. Reddy

See my previous comments.

Alan M. Shiller

In general, the interpretive techniques were fine. However, little data were actually presented in the report. There are a few example figures of before/after hydrocarbon profiles and one table of summary statistics (without n values). I find the figures to be illustrative examples, but it's hard to quantify what's going on by looking at them. In the end, one really just needs to trust them. Also, as noted above, no information was given on the criteria for "Match", "Probable Match", etc. So, I can't really evaluate the reasonableness of that.

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

Merv Fingas

Some of these are hard to believe – e.g., the conclusions about the asphaltene and FFPI index on page 18.

Christopher M. Reddy

See other comments but again GC-MS analysis can only account for 5% of the mass, the asphaltene content may be incorporating weathering products, and that the results have not been normalized to hopane. These issues need to be acknowledged (and addressed if possible).

Alan M. Shiller

Since QA/QC information was not provided, nor are the basic data available in the report, I can't really comment on this beyond the observation that what is presented in summary Table 3 seems okay.

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

Merv Fingas

Only partially – much better than the FFPI would have been to measure the Wang pyrogenic index. Then provide an estimate of the completeness of combustion and then try to correlate the completeness of combustion to the composition such as resins, asphaltenes, etc.

Christopher M. Reddy

See other comments, too. Using the GC-MS data that were available, I recommend that the authors reexamine and expand on the PAH data. Once normalized to hopane, what other ratios and calculations be used to examine these samples? There is much more available than the FFPI.

Alan M. Shiller

The report, as far as it goes, does give some useful diagnostics to identify in-situ burned oil residue. This was done mainly in the form of a summary of useful biomarker indicators. I would have preferred to see a more quantitative presentation of how the burning affected some of these other indicators. For instance, the literature abounds with pyrogenic/petrogenic diagnostic PAH ratios. I suspect their data could inform the interpretative utility/futility of some of these widely used ratios.

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

Merv Fingas

Yes, it seems to have worked. This could have been strengthened by calculating the Wang pyrogenic index and estimating the degree of combustion.

Christopher M. Reddy

The authors were successful in using select biomarkers to distinguish in-situ burn residues. This is interesting and certainly real, but how and why? Why does this signal appear and how might it be used to shed light on combustion processes? Are there examples in the combustion literature that would support these findings?

Alan M. Shiller

Table 3 presents the summary of their suggested means for distinguishing burn residues. It seems appropriate and I have no reason to doubt the authors. Nonetheless, I'd like to see the data.

4. Were there any critical results or issues that were not discussed in the report?

Merv Fingas

Looks good - however, it would have been better to measure the Wang pyrogenic index. Then provide an estimate of the completeness of combustion and then try to correlate the completeness of combustion to the composition such as resins, asphaltenes, etc.

Christopher M. Reddy

Beyond what has been mentioned, check the literature.

Alan M. Shiller

I've pretty much outlined my response to this in 2a, above as well as in my continuing comments about lack of data presentation.

5. Does this report present sufficient new data and knowledge, and are the findings useful?

Merv Fingas

Yes, certainly, note the possible strengthening of the data above. Further tables of specific results should have been presented to allow the readers and reviewers to judge the results for themselves.

Christopher M. Reddy

Yes. Overall, these findings are useful. The uniqueness of this study was access to actual field samples, pre- and post-burn, although there were only a few at quantities too small for comprehensive analysis.

As the authors have noted, there was not a concerted and dedicated effort to collect field samples so this study was limited. Along with the scarce metadata on the actual field samples, small mass of samples, and that the target list only captured a fraction of the chemical composition of the samples, this study is a good start. If time and budget permit, I recommend additional synthesis and distillation of data. And if there was enough material remaining, perhaps some or all of the samples could be analyzed with techniques that could add to the GC-MS results.

Alan M. Shiller

As far as it goes, this is a useful report. Addressing the issues raised above should be easy for the authors to do and would make this a far more useful contribution.

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

Merv Fingas

There have been extensive studies by some parties on burn residue and pyrogenic index, which is only somewhat acknowledged in this study.

Christopher M. Reddy

I am unaware of any other studies beyond those cited in this document on in-situ burns. Dr. Merv Fingas would be an ideal reviewer.

However, there are many other studies on the *Deepwater Horizon* disaster that should be considered. There is a vast collection of publications on the formation of PAHs from the incomplete combustion of organic matter, which will be invaluable for interpreting the PAH data.

Alan M. Shiller

There's not a lot that's been done on this subject, to my knowledge. The authors cite the appropriate literature.

V.3 Specific Observations

Merv Fingas

Page	Paragraph	Comment or Question
12	3	Two methods are given to measure asphaltene content – which one was used or were the results averaged?
18	1-2	The conclusion is that the doubling of asphaltenes after burning was not significant, however in the next paragraph a few percent of FFPI increase is said to be significant – neither result is credible.
31	Table 3	Needs explanations of the meaning of terms in the table.
32	Table 4	The density of other MC252 tarballs at 0.73 is unrealistically low – some problem with this number – did this tarball contain vegetative material?
32	2,3,4	Resins were not measured, nor were viscosity – both of these are important to judging burn extent.

The following are studies that could have enhanced this study.

<u>Studies of residue and burn efficiency correlations</u>

Fingas, M.F., Z. Wang, B. Fieldhouse, C.E. Brown, C. Yang, M. Landriault and D. Cooper, "In-situ Burning of Heavy Oils and Orimulsion: Analysis of Soot and Residue", in Proceedings of the Twenty-eighth Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp.333-348, 2005.

Fingas, M.F., B. Fieldhouse, C.E. Brown and L. Gamble, "In-Situ Burning of Heavy Oils and Orimulsion: Mid-Scale Burns", in Proceedings of the Twenty-Seventh Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp. 207-233, 2004.

<u>Pyrogenic Index</u>

Wang, Z., M.F. Fingas, M. Landraiult, L. Sigouin, and P. Lambert, "Distribution of PAHs in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 and 1997 Mobile Burn Study" in Diesel Fuels, editors: C. Song, C. Hsu and I. Mochida, pp. 237-253, 1999.

Wang, Z., M.F. Fingas, Y.Y. Shu, L. Sigouin, M. Landraiult, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study", Environmental Science and Technology, Vol. 33, pp. 3100-3109, 1999. (this is listed in references but doesn't appear to have been used)

Wang, Z., M.F. Fingas, M. Landraiult, L. Sigouin, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and

Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study", in Proceedings of The 1999 International Oil Spill Conference, American Petroleum Institute, Washington, D.C., pp. 1287-1292, 1999.

Wang, Z., M.F. Fingas, L. Sigouin, M. Landraiult, K. Li, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study", in Proceedings of the Twenty-First Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp. 673-703, 1998.

Wang, Z., M.F. Fingas, L. Sigouin, M. Landraiult and P. Lambert, "Distribution of PAHs in Burn Residue and Soot Samples and Differentiation of Pyrogenic PAHs from Petrogenic PAHs - the 1994 and 1997 Mobile Burn Study", in Proceedings of the Symposium on Chemistry of Diesel Fuels, Division of Petroleum Chemistry, 216th National Meeting, Boston, MA, pp. 607-610, 1998.

Christopher M. Reddy

Page	Paragraph	Comment or Question
Title		Since the response extended past the capping of the well, would it be informative to list the time period that the burns were conducted in the title?
Abstract	1st	Be more specific on the dates, distances, water depths—more information
Abstract and elsewhere		What is a tarball? How it is different than the other samples in this study. Recommend a table listing the terms and descriptions
Abstract and elsewhere		Remember to italicize *Deepwater Horizon*
Abstract and elsewhere		Remember to define acronyms first and only once.
Page 3		A significant fraction of the oil evaporated quickly. How did that affect the burns?
All pictures and elsewhere		Please add as much metadata as possible. Dates? Where? Time of the day? At what point during a burn, etc.?
All pictures with smoke		Does the smoke tell us anything about the burns?
Page 6		Use metric---"50 nm" slipped into the text

Page	Paragraph	Comment or Question
Page 6		Please comment more and show the data for the SIMDIS
Figure 5		Make font bigger and add more content to the caption
Figure 7		What are the temperatures of these burns? This relates to the formation of PAHs (see Max Blumer's classic papers and literature reviews on PAH formation).
Page 7		Can a box or table be constructed that lists the observations made in past burns? Tell the reader, for example, about the conclusions of Fingas et al. 1994.
Table 1 and elsewhere		Use IUPAC for PAHs---i.e., benz[a]pyrene
Page 16		Use other published results to compare results from this study on characterizing the Macondo well oil. Check the literature.
Figure 10a		Why the step down in the alkane content from 24 to 25? Does not seem smooth.
Figure 16		Expand and clarify figure caption.

Alan M. Shiller

Page	Paragraph	Comment or Question
3		Add references in paragraphs 2 and 3.
6		Add references to Figure 5 and 1st paragraph.
7		The note at the bottom about the post-burn sample should probably be in the methods.
10		Next to last paragraph: add references.
12		Add references to methods.
14	2	Discuss heat dissipation by water.
15	1	Discuss match criteria.
Results		Provide data.
25	4	Describe simulated distillation in methods.
Apx	T2	Give characteristic ratios.

VI. APPENDIX A: INDIVIDUAL REVIEWER COMMENTS

Review by:
Merv Fingas, Ph.D.

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: Merv Fingas
Affiliation: Spill Science
Date: Oct 22, 2014

I. GENERAL IMPRESSIONS

The report is quite good and a good scientific approach was used. The identification of the oil is good and does correctly identify residues that can be associated with the *Deepwater Horizon*. The identification is based on only a few parameters, however, should be adequate for the purpose.

The report could have been improved by estimating the extent of burning, identifying correlations in content to the extent of burning, and comparing to the Wang pyrogenic index. The extent of burning could have been estimated by using the Wang pyrogenic index.

It is unfortunate that 'original' samples of the oil were not available to act as starting samples, however, this is not the fault of the principal investigator. The presence of original samples and tests on their burns would have enabled a much greater extent of comparison and study.

II. RESPONSE TO CHARGE QUESTIONS

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

The objectives of the study are perhaps not as specific as they could have been. After the general charge to identify the physical and chemical changes in the oil, it might have been prudent to be more specific and add tasks such as estimate the extent of burning, identify correlations in content to the extent of burning, compare to the Wang pyrogenic index, etc.

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

Yes, however again they might have been more specific such as specifying that the asphalt, resin, density, and viscosity all be measured.

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

Yes, again there could have been more specific additions such as the Wang pyrogenic index - the identification of the oil was good and complete as carried out.

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

Some of these are hard to believe – e.g., the conclusions about the asphaltene and FFPI index on page 18.

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

Only partially – however, much better than the FFPI would have been to measure the Wang pyrogenic index. Then provide an estimate of the completeness of combustion and then try to correlate the completeness of combustion to the composition such as resins, asphaltenes, etc.

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

Yes, it seems to have worked. This could have been strengthened by calculating the Wang pyrogenic index and estimating the degree of combustion.

4. Were there any critical results or issues that were not discussed in the report?

Looks good – however, it would have been better to measure the Wang pyrogenic index. Then provide an estimate of the completeness of combustion and then try to correlate the completeness of combustion to the composition such as resins, asphaltenes, etc.

5. Does this report present sufficient new data and knowledge, and are the findings useful?

Yes, certainly, note the possible strengthening of the data above. Further tables of specific results should have been presented to allow the readers and reviewers to judge the results for themselves.

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

There have been extensive studies by some parties on burn residue and pyrogenic index, which is only somewhat acknowledged in this study.

III. SPECIFIC OBSERVATIONS

Page	Paragraph	Comment or Question
12	3	Two methods are given to measure asphaltene content – which one was used or were the results averaged?
18	1-2	The conclusion is that the doubling of asphaltenes after burning was not significant, however in the next paragraph a few percent of FFPI increase is said to be significant – neither result is credible.
31	Table 3	Needs explanations of the meaning of terms in the table.
32	Table 4	The density of other MC252 tarballs at 0.73 is unrealistically low – some problem with this number – did this tarball contain vegetative material?
32	2,3,4	Resins were not measured, nor was viscosity – both of these are important to judging burn extent.

The following are studies that could have enhanced this study.

Studies of residue and burn efficiency correlations

Fingas, M.F., Z. Wang, B. Fieldhouse, C.E. Brown, C. Yang, M. Landriault and D. Cooper, "In-situ Burning of Heavy Oils and Orimulsion: Analysis of Soot and Residue", in Proceedings of the Twenty-eighth Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp.333-348, 2005.

Fingas, M.F., B. Fieldhouse, C.E. Brown and L. Gamble, "In-Situ Burning of Heavy Oils and Orimulsion: Mid-Scale Burns", in Proceedings of the Twenty-Seventh Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp. 207-233, 2004.

Pyrogenic Index

Wang, Z., M.F. Fingas, M. Landraiult, L. Sigouin, and P. Lambert, "Distribution of PAHs in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 and 1997 Mobile Burn Study" in Diesel Fuels, editors: C. Song, C. Hsu and I. Mochida, pp. 237-253, 1999.

Wang, Z., M.F. Fingas, Y.Y. Shu, L. Sigouin, M. Landraiult, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study", Environmental Science and Technology, Vol. 33, pp. 3100-3109, 1999. (this is listed in references but doesn't appear to have been used)

Wang, Z., M.F. Fingas, M. Landraiult, L. Sigouin, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study",

in Proceedings of The 1999 International Oil Spill Conference, American Petroleum Institute, Washington, D.C., pp. 1287-1292, 1999.

Wang, Z., M.F. Fingas, L. Sigouin, M. Landraiult, K. Li, P. Lambert, R. Turpin, P. Campagna, and J. Mullin. "Quantitative Characterization of PAH in Burn Residue and Soot Samples and Differentiation of Pyrogenic and Petrogenic PAHs from PAHs - the 1994 Mobile Burn Study", in Proceedings of the Twenty-First Arctic and Marine Oil Spill Program Technical Seminar, Environment Canada, Ottawa, Ontario, pp. 673-703, 1998.

Wang, Z., M.F. Fingas, L. Sigouin, M. Landraiult and P. Lambert, "Distribution of PAHs in Burn Residue and Soot Samples and Differentiation of Pyrogenic PAHs from Petrogenic PAHs - the 1994 and 1997 Mobile Burn Study", in Proceedings of the Symposium on Chemistry of Diesel Fuels, Division of Petroleum Chemistry, 216th National Meeting, Boston, MA, pp. 607-610, 1998.

Review by:
Christopher M. Reddy, Ph.D.

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: Christopher Reddy
Affiliation: Woods Hole Oceanographic Institution
Date: November 4, 2014

I. GENERAL IMPRESSIONS

Please note that these questions lead to less than ideal means to review this document. I will do my best to answer each question but will expand where I see necessary.

I enjoyed reading this document and learned several useful details. I also commend the authors for their efforts in avoiding ambiguity and highlighting the challenges they faced in this study. Overall, it captures the needs of the charge although there are numerous opportunities to improve it.

Briefly, I would recommend an executive summary and a more pronounced summary/conclusions. Please make the recommendations a separate section. Also, beyond better sampling, what could be done to provide greater insights into the identification of burn residues? That is, different analytical approaches and usage of other compounds or bulk properties.

Could faster turnaround times be provided so that burns can be tweaked with analytical data in near real time? Can responders who have a set list of observations/tasks record georeferenced data and images?

Collectively, more data with greater breadth in synthesis and interpretation should lead to more effective burns.

II. RESPONSE TO CHARGE QUESTIONS

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

The authors have done an excellent job writing the necessary background and introduction to this report. Coupled with clear and comprehensive objectives, it is a well-organized document with an excellent description of the objectives.

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

Yes and no. For the gas chromatography analysis, it was appropriately designed and clearly described.

I am concerned about the definition and usage of the asphaltene content. As there is no set structure (and there is an active and ongoing debate about it and many aspects of the asphaltenes), approaches to measure them in oils must be operationally defined. Due to ease and that it is chemically reasonable, the asphaltenes are often measured by the amount of material that can be initially extracted in toluene or dichloromethane but then will precipitate in hexane or heptane. The asphaltenes are measured gravimetrically. This approach is almost entirely used during upstream and downstream and rarely for environmental analysis. I have several comments regarding the asphaltenes:

1) Using this operationally defined method, the authors may be measuring apples and oranges. As noted by the authors and others, the product that leaked from the Macondo well was a light, sweet crude, which would presumably indicate trace amounts of asphaltenes. The authors measured less than 1%, a very reasonable result.
2) However, more recent studies on the *Deepwater Horizon* disaster by Aeppli et al. (2012; *Environmental Science and Technology*) and Lewan et al. (2014; *Organic Geochemistry*) have shown that polar, enriched in oxygen content, non-GC amenable materials insoluble in hexane are formed by weathering. Aeppli nicely showed this increase in asphaltene-like (but not asphaltenes, which are formed in reservoirs) relative to the conserved internal standard, hopane. So it's entirely possible that any changes in the operationally defined method may capture preferential enrichment of the native asphaltenes and also weathering processes that are forming another material also detected with this method. I recommend a revaluation of the results presented in this study.

3) What is the precision and accuracy of precipitation and gravimetric analysis of asphaltenes, especially at low levels? Please discuss.

4) What about other methods for tracking asphaltene-like materials? I would consider bulk oxygen and nitrogen, liquid chromatography, or spectrometric analysis as well as measuring the black or elemental carbon content.

5) This was mentioned previously, but it is absolutely paramount that any changes in the samples are normalized to hopane. This is standard practice and will likely shed greater light on the formation of the parent. Otherwise, I would consider this study incomplete.

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

The authors are experts in the analysis of petroleum hydrocarbons and other organic chemicals. Their approach for using gas chromatography with mass spectral detection (GC-MS) is a very traditional, mature, and sound method. I would have done exactly what they would have done first. Beyond normalizing to hopane, the biggest issue at stake is the limited breadth of the target list. In the Macondo well oil, the authors could account for total saturates and PAHs at 5 and 1%, respectively, of the total mass. And with weathering and loss of the "front end," the amount of

material that can be targeted with GC-MS gets smaller. This does not mean that this study is defective but it is limited.

While there are other approaches to measure oil residues, most are not mainstream. It would be unfair to expect the authors to use the most cutting edge instruments, but great if they could. The authors do need to note they are basing their results by tracking ~5% of the mass and future studies should consider other methods.

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

See my previous comments.

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

See other comments but again GC-MS analysis can only account for 5% of the mass, the asphaltene content may be incorporating weathering products, and that the results have not been normalized to hopane. These issues need to be acknowledged (and addressed if possible).

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

See other comments, too. Using the GC-MS data that were available, I recommend that the authors reexamine and expand on the PAH data. Once normalized to hopane, what other ratios and calculations be used to examine these samples? There is much more available than the FFPI.

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

The authors were successful in using select biomarkers to distinguish in-situ burn residues. This is interesting and certainly real, but how and why? Why does this signal appear and how might it be used to shed light on combustion processes? Are there examples in the combustion literature that would support these findings?

4. Were there any critical results or issues that were not discussed in the report?

Beyond what has been mentioned, check the literature.

5. Does this report present sufficient new data and knowledge, and are the findings useful?

Yes. Overall, these findings are useful. The uniqueness of this study was access to actual field samples, pre- and post-burn, although there were only a few at quantities too small for comprehensive analysis.

As the authors have noted, there was not a concerted and dedicated effort to collect field samples so this study was limited. Along with the scarce metadata on the actual field samples, small mass of samples, and that the target list only captured a fraction of the chemical composition of the samples, this study is a good start. If time and budget permit, I recommend additional synthesis and distillation of data. And if there was enough material remaining, perhaps some or all of the samples could be analyzed with techniques that could add to the GC-MS results.

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

I am unaware of any other studies beyond those cited in this document on in-situ burns. Dr. Merv Fingas would be an ideal reviewer.

However, there are many other studies on the *Deepwater Horizon* disaster that should be considered. There is a vast collection of publications on the formation of PAHs from the incomplete combustion of organic matter, which will be invaluable for interpreting the PAH data.

III. SPECIFIC OBSERVATIONS

Page	Paragraph	Comment or Question
Title		Since the response extended past the capping of the well, would it be informative to list the time period that the burns were conducted in the title?
Abstract	1st	Be more specific on the dates, distances, water depths—more information
Abstract and elsewhere		What is a tarball? How it is different than the other samples in this study. Recommend a table listing the terms and descriptions
Abstract and elsewhere		Remember to italicize *Deepwater Horizon*
Abstract and elsewhere		Remember to define acronyms first and only once.
Page 3		A significant fraction of the oil evaporated quickly. How did that affect the burns?
All pictures		Please add as much metadata as possible. Dates? Where? Time of the day? At what point during a burn, etc?

Page	Paragraph	Comment or Question
and elsewhere		
All pictures with smoke		Does the smoke tell us anything about the burns?
Page 6		Use metric---"50 nm" slipped into the text.
Page 6		Please comment more and show the data for the SIMDIS.
Figure 5		Make font bigger and add more content to the caption.
Figure 7		What are the temperatures of these burns? This relates to the formation of PAHs (see Max Blumer's classic papers and literature reviews on PAH formation).
Page 7		Can a box or table be constructed that lists the observations made in past burns? Tell the reader, for example, about the conclusions of Fingas et al. 1994.
Table 1 and elsewhere		Use IUPAC for PAHs---i.e., benz[*a*]pyrene
Page 16		Use other published results to compare results from this study on characterizing the Macondo well oil. Check the literature.
Figure 10a		Why the step down in the alkane content from 24 to 25? Does not seem smooth.
Figure 16		Expand and clarify figure caption.

Review by:
Alan M. Shiller, Ph.D.

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: Alan M. Shiller
Affiliation: The University of Southern Mississippi
Date: 13 November 2014

I. GENERAL IMPRESSIONS

Overall this is an interesting report examining in-situ burn residue composition and seeking to determine if there exist diagnostic compositional indicators of burn residues. The report is somewhat constrained due to the limited samples that were available of in-situ burn residue as well as of MC252 oil. Also, only a few experiments were performed. The presentation is generally clear and sound. The objectives, methods, and results are fairly clear. My main comments relate to lack of supporting information. There are some places where further references to the literature and more details about methods would be helpful. The main concern I have is the limited presentation of supporting data. There are a few graphical presentations of results and one supporting table of summary statistics. It would be useful, however, if all of the data were available in an appendix. There are also a few other minor additions that would be helpful and which I describe below. In summary, this is a generally good report and with some straight forward modifications this would far more useful.

II. RESPONSE TO CHARGE QUESTIONS

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

The objectives of the project are straight forward and clearly laid out, mainly through the list of Tasks on page 10 of the report.

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

In general, the methods were adequately described. That said, there were some places that additional detail would be helpful. In particular, references to the asphaltene extraction, the GC/MS characterization, and modified FFPI would be useful. QA/QC procedures should also be described. The criteria for "Match", "Probable Match", etc. need to be described. On page 25, there is mention of a "simulated distillation of MC252 oil" that is not described in the methods.

It would also be appropriate to have some discussion of the controlled burn methodology. One wonders if there are some differences in what happens in open water where there is a large

capacity of the water to dissipate heat at the bottom of the burning residue versus the experiments which were conducted in plastic pools with more limited heat dissipation ability. Additionally, the anecdotal communication presented on page 7, suggests that perhaps some burn components sink and some float. The controlled burn experiments would not be able to distinguish those components.

Finally, there only seemed to be a couple of controlled burn experiments (the exact number was unclear, possibly 3 replicates of surrogate oil and 3 replicates of emulsified MC252). A more explicit discussion of how reproducible the results were would be of use.

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

In general, the interpretive techniques were fine. However, little data were actually presented in the report. There are a few example figures of before/after hydrocarbon profiles and one table of summary statistics (without n values). I find the figures to be illustrative examples, but it's hard to quantify what's going on by looking at them. In the end, one really just needs to trust them. Also, as noted above, no information was given on the criteria for "Match", "Probable Match", etc. So, I can't really evaluate the reasonableness of that.

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

Since QA/QC information was not provided, nor are the basic data available in the report, I can't really comment on this beyond the observation that what is presented in summary Table 3 seems okay.

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

The report, as far as it goes, does give some useful diagnostics to identify in-situ burned oil residue. This was done mainly in the form of a summary of useful biomarker indicators. I would have preferred to see a more quantitative presentation of how the burning affected some of these other indicators. For instance, the literature abounds with pyrogenic/petrogenic diagnostic PAH ratios. I suspect their data could inform the interpretative utility/futility of some of these widely used ratios.

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

Table 3 presents the summary of their suggested means for distinguishing burn residues. It seems appropriate and I have no reason to doubt the authors. Nonetheless, I'd like to see the data.

4. Were there any critical results or issues that were not discussed in the report?

I've pretty much outlined my response to this in 2a, above as well as in my continuing comments about lack of data presentation.

5. Does this report present sufficient new data and knowledge, and are the findings useful?

As far as it goes, this is a useful report. Addressing the issues raised above should be easy for the authors to do and would make this a far more useful contribution.

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

There's not a lot that's been done on this subject, to my knowledge. The authors cite the appropriate literature.

III. SPECIFIC OBSERVATIONS

Page	Paragraph	Comment or Question
3		Add references in paragraphs 2 and 3.
6		Add references to Figure 5 and 1st paragraph.
7		The note at the bottom about the post-burn sample should probably be in the methods.
10		Next to last paragraph: add references.
12		Add references to methods.
14	2	Discuss heat dissipation by water.
15	1	Discuss match criteria.
Results		Provide data.
25	4	Describe simulated distillation in methods.
Apx	T2	Give characteristic ratios.

VII. APPENDIX B: PEER REVIEW MATERIALS PACKAGE

Merv Fingas, Ph.D.

ENDYNA

October 15, 2014

Merv F. Fingas, Ph.D.
[redacted]

Dear Dr. Fingas:

Thank you for accepting our invitation to review the final research report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response." We are conducting this external letter peer review for the Department of the Interior's (DOI) Bureau of Safety and Environmental Enforcement (BSEE). Please find attached the Peer Review Materials Package, which is the official document that authorizes you to perform this work. In this package, we have included the following items:

1. Peer Review Charge Document
    - Project History and Objectives
    - Work Scope and Schedule
    - Location
    - Confidentiality Requirements
    - Disclaimer
    - Instructions for Preparing Written Comments and Logistics
    - Charge Questions
2. Peer Review Comment Template
3. Final Research Report: "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

We request that you **complete your written review of the document on or before November 14, 2014** and appreciate your sensitivity towards the deadline. Please email your comments in a MS Word attachment, using the template provided, to Betzy Colon at [redacted] and Danielle Murray at [redacted]. As noted in the confidentiality requirements below, your comments and the report should not be distributed or discussed with any outside party.

Once the three reviewers have completed and submitted their comments to the EnDyna Team, the comments will be provided directly to BSEE. Following completion and acceptance of your review, we will ask you to send us an invoice for your services.

Thank you again for participating in this peer review. Please do not hesitate to contact Betzy Colon ([redacted]) and Danielle Murray ([redacted]) if you have questions.

Sincerely,

Smita Siddhanti, Ph.D.
Program Manager

PEER REVIEW CHARGE DOCUMENT

ENDYNA PROJECT NUMBER:	DINP-001
TITLE:	Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"
PEER REVIEWER NAME:	Merv F. Fingas, Ph.D.
PERIOD OF PERFORMANCE:	Completion of written review on or before November 14, 2014

Project History and Objectives

BSEE has requested an external peer review of the final research report entitled "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response," which was prepared by the National Oceanic and Atmospheric Administration (NOAA) and Louisiana State University (LSU). Part of BSEE's research is committed to ensuring that functional, safe, and environmentally responsible oil spill response methods are identified and used under appropriate conditions. In-situ burning is a practical oil spill response, and was used on an unprecedented scale during the response to the Macondo oil spill incident. Afterwards, tar balls were collected in various locations, including in deep sea trawls of the royal red shrimp fishery. As a result, BSEE contracted NOAA and LSU researchers to perform physical and chemical analyses of surface collected oil, field in-situ burn residues, deep water tar balls, and experimentally burned field collected oil. The results of these analyses are interpreted to characterize physical and chemical changes in oil during combustion and other weathering processes.

One note: researchers had to conduct this project with the limited samples that were available and didn't have any influence concerning the method of their collection. This retrospective study tried to meld the in-situ collections with lab-generated results, to the maximum extent possible.

The objective of this letter-style peer review is for BSEE to receive written comments from individual experts on the scientific merit of the NOAA/LSU report, appropriateness of the assumptions made, and quality and relevance of the data.

Work Scope and Schedule

Your primary function as a peer reviewer is to review and provide written comments on the NOAA/LSU report. Specifically, you shall evaluate the report, provide general comments and overall impressions of the scientific merit of the report, respond to six charge questions, and provide other specific comments that you feel will improve the quality of the report. You are not requested to and should not provide input or advice on BSEE's policies and decisions. Your review is not page-limited, and you should take as much space as you feel is necessary to complete your review.

The EnDyna Team has selected you as part of a panel of three senior scientists with expertise/experience in oil spill response, in-situ burning, and chemical analyses of hydrocarbons to serve as peer reviewers. You have four weeks to complete your written review of the report. Once all three reviewers have completed and submitted their written comments to the EnDyna Team, the comments will be compiled into a comprehensive peer review report for distribution to BSEE. Your name and affiliation will be disclosed in the report, and the peer review report may be posted on BSEE's research webpage alongside the final research report.

LIST OF DELIVERABLES	
Receive Peer Review Materials Package	October 15, 2014
Complete and Submit Written Comments	November 14, 2014

Location
No travel is required as this is a letter-style peer review.

Confidentiality Requirements
As noted in the confidentiality agreement, the NOAA/LSU report and your comments should not be distributed or discussed with any outside party, including members of the public, DOI, or other Federal agencies. If you are contacted in person or in writing regarding the NOAA/LSU report under review by anyone other than EnDyna Team, you should immediately inform EnDyna.

Disclaimer
In accordance with the Office of Management and Budget's (OMB) *Final Information Quality Bulletin for Peer Review*, "this information is distributed solely for the purpose of pre-dissemination peer review under applicable information quality guidelines. It has not been formally disseminated by the Agency. It does not represent and should not be construed to represent any agency determination or policy."

In addition, the reviewers shall conduct their review of the report in a manner that complies with the guidelines and policies included in OMB's *Final Information Quality Bulletin for Peer Review*, Executive Order 12866, and DOI's Information Quality Standards, Department Manual, and Peer Review Process Manual.

Instructions for Preparing Written Comments and Logistics
In order to ensure that all charge questions are answered completely and each reviewer submits their comments in a consistent format, a peer review comment template containing the three major sections below is attached. Please use this template to prepare your written comments. Once you have completed the template, please email your comments in MS Word, to Ms. Betzy Colon at [redacted] and copy Ms. Danielle Murray at [redacted]. Comments are due on or before **November 14, 2014.**

I. General Impressions - Provide overall impressions (approximately 1/2 page in length) addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.

II. Response to Charge Questions - Provide narrative responses to the six charge questions. The charge questions are listed below.

III. Specific Observations - Provide specific observations or comments on the report mentioning page and paragraph.

Charge Questions:

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

    a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

    b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

    a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

    b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

    c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

4. Were there any critical results or issues that were not discussed in the report?

5. Does this report present sufficient new data and knowledge, and are the findings useful?

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

PEER REVIEW COMMENT TEMPLATE

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: _____
Affiliation: _____
Date: _____

I. GENERAL IMPRESSIONS

Provide overall impressions (approximately 1/2 page in length) addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.

Comments:

II. RESPONSE TO CHARGE QUESTIONS

Provide narrative responses to each of the six charge questions below.

1. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

Comments:

2. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

Comments:

b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

Comments:

3. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

Comments:

b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

Comments:

c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

Comments:

4. Were there any critical results or issues that were not discussed in the report?

Comments:

5. Does this report present sufficient new data and knowledge, and are the findings useful?

Comments:

6. What related research has been conducted, and do those results support the findings/conclusions of this study?

Comments:

III. SPECIFIC OBSERVATIONS

Provide specific observations or comments on the report mentioning page and paragraph (expand table if needed).

Page	Paragraph	Comment or Question

Christopher M. Reddy, Ph.D.

ENDYNA

October 15, 2014

Christopher M. Reddy, Ph.D.
[redacted]

Dear Dr. Reddy:

Thank you for accepting our invitation to review the final research report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected during the Deepwater Horizon Spill Response." We are conducting this external letter peer review for the Department of the Interior's (DOI) Bureau of Safety and Environmental Enforcement (BSEE). Please find attached the Peer Review Materials Package, which is the official document that authorizes you to perform this work. In this package, we have included the following items:

4. Peer Review Charge Document
    - Project History and Objectives
    - Work Scope and Schedule
    - Location
    - Confidentiality Requirements
    - Disclaimer
    - Instructions for Preparing Written Comments and Logistics
    - Charge Questions
5. Peer Review Comment Template
6. Final Research Report: "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

We request that you **complete your written review of the document on or before November 14, 2014** and appreciate your sensitivity towards the deadline. Please email your comments in a MS Word attachment, using the template provided, to Betzy Colon at [redacted] and Danielle Murray at [redacted]. As noted in the confidentiality requirements below, your comments and the report should not be distributed or discussed with any outside party.

Once the three reviewers have completed and submitted their comments to the EnDyna Team, the comments will be provided directly to BSEE. Following completion and acceptance of your review, we will ask you to send us an invoice for your services.

Thank you again for participating in this peer review. Please do not hesitate to contact Betzy Colon ([redacted]) or Danielle Murray ([redacted]) if you have questions.

Sincerely,

Smita Siddhanti, Ph.D.
Program Manager

PEER REVIEW CHARGE DOCUMENT

ENDYNA PROJECT NUMBER:	DINP-001
TITLE:	Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"
PEER REVIEWER NAME:	Christopher M. Reddy, Ph.D.
PERIOD OF PERFORMANCE:	Completion of written review on or before November 14, 2014

Project History and Objectives

BSEE has requested an external peer review of the final research report entitled "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response," which was prepared by the National Oceanic and Atmospheric Administration (NOAA) and Louisiana State University (LSU). Part of BSEE's research is committed to ensuring that functional, safe, and environmentally responsible oil spill response methods are identified and used under appropriate conditions. In-situ burning is a practical oil spill response, and was used on an unprecedented scale during the response to the Macondo oil spill incident. Afterwards, tar balls were collected in various locations, including in deep sea trawls of the royal red shrimp fishery. As a result, BSEE contracted NOAA and LSU researchers to perform physical and chemical analyses of surface collected oil, field in-situ burn residues, deep water tar balls, and experimentally burned field collected oil. The results of these analyses are interpreted to characterize physical and chemical changes in oil during combustion and other weathering processes.

One note: researchers had to conduct this project with the limited samples that were available and didn't have any influence concerning the method of their collection. This retrospective study tried to meld the in-situ collections with lab-generated results, to the maximum extent possible.

The objective of this letter-style peer review is for BSEE to receive written comments from individual experts on the scientific merit of the NOAA/LSU report, appropriateness of the assumptions made, and quality and relevance of the data.

Work Scope and Schedule

Your primary function as a peer reviewer is to review and provide written comments on the NOAA/LSU report. Specifically, you shall evaluate the report, provide general comments and overall impressions of the scientific merit of the report, respond to six charge questions, and provide other specific comments that you feel will improve the quality of the report. You are not requested to and should not provide input or advice on BSEE's policies and decisions. Your review is not page-limited, and you should take as much space as you feel is necessary to complete your review.

The EnDyna Team has selected you as part of a panel of three senior scientists with expertise/experience in oil spill response, in-situ burning, and chemical analyses of hydrocarbons to serve as peer reviewers. You have four weeks to complete your written review of the report. Once all three reviewers have completed and submitted their written comments to the EnDyna Team, the comments will be compiled into a comprehensive peer review report for distribution to BSEE. Your name and affiliation will be disclosed in the report, and the peer review report may be posted on BSEE's research webpage alongside the final research report.

LIST OF DELIVERABLES	
Receive Peer Review Materials Package	October 15, 2014
Complete and Submit Written Comments	November 14, 2014

Location
No travel is required as this is a letter-style peer review.

Confidentiality Requirements
As noted in the confidentiality agreement, the NOAA/LSU report and your comments should not be distributed or discussed with any outside party, including members of the public, DOI, or other Federal agencies. If you are contacted in person or in writing regarding the NOAA/LSU report under review by anyone other than EnDyna Team, you should immediately inform EnDyna.

Disclaimer
In accordance with the Office of Management and Budget's (OMB) *Final Information Quality Bulletin for Peer Review*, "this information is distributed solely for the purpose of pre-dissemination peer review under applicable information quality guidelines. It has not been formally disseminated by the Agency. It does not represent and should not be construed to represent any agency determination or policy."

In addition, the reviewers shall conduct their review of the report in a manner that complies with the guidelines and policies included in OMB's *Final Information Quality Bulletin for Peer Review*, Executive Order 12866, and DOI's Information Quality Standards, Department Manual, and Peer Review Process Manual.

Instructions for Preparing Written Comments and Logistics
In order to ensure that all charge questions are answered completely and each reviewer submits their comments in a consistent format, a peer review comment template containing the three major sections below is attached. Please use this template to prepare your written comments. Once you have completed the template, please email your comments in MS Word, to Ms. Betzy Colon at [redacted] and copy Ms. Danielle Murray at [redacted]. Comments are due on or before **November 14, 2014.**

I. General Impressions - Provide overall impressions (approximately 1/2 page in length) addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.

II. Response to Charge Questions - Provide narrative responses to the six charge questions. The charge questions are listed below.

III. Specific Observations - Provide specific observations or comments on the report mentioning page and paragraph.

Charge Questions:

7. Are the objectives of the study clearly defined? If not, what are your recommendations for improving the description of objectives?

8. Was the methodology used to define the physical and chemical characteristics of the residue appropriately designed and clearly described? Additionally and specifically:

    a. Please comment on the methods designed for Tasks 1-6. Were they appropriate, well described and properly implemented?

    b. Were the qualitative and quantitative techniques used for forensic interpretations appropriate and properly used?

9. Are the conclusions drawn supported by the laboratory results and analysis of each task? Additionally and Specifically:

    a. Please comment on the sampling and statistical validity of laboratory analysis of samples used to interpret the findings.

    b. Please comment on the physical and chemical characteristics identified in the study (i.e., density, percent asphaltenes, normal alkane profile, pyrogenic PAHs, FFPI) and if these can point to in-situ burning as a significant driver of weathering and fate.

    c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

10. Were there any critical results or issues that were not discussed in the report?

11. Does this report present sufficient new data and knowledge, and are the findings useful?

12. What related research has been conducted, and do those results support the findings/conclusions of this study?

PEER REVIEW COMMENT TEMPLATE

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: _____
Affiliation: _____
Date: _____

I. GENERAL IMPRESSIONS

Provide overall impressions (approximately 1/2 page in length) addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.

Comments:

II. RESPONSE TO CHARGE QUESTIONS

Provide narrative responses to each of the six charge questions below.

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Comments:

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Comments:

III. SPECIFIC OBSERVATIONS

Provide specific observations or comments on the report mentioning page and paragraph (expand table if needed).

Page	Paragraph	Comment or Question

Alan M. Shiller, Ph.D.

ENDYNA

October 15, 2014

Alan M. Shiller, Ph.D.
[redacted]

Dear Dr. Shiller:

Thank you for accepting our invitation to review the final research report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected during the Deepwater Horizon Spill Response." We are conducting this external letter peer review for the Department of the Interior's (DOI) Bureau of Safety and Environmental Enforcement (BSEE). Please find attached the Peer Review Materials Package, which is the official document that authorizes you to perform this work. In this package, we have included the following items:

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Smita Siddhanti, Ph.D.
Program Manager

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Charge Questions:

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    c. Were the results properly used to distinguish in-situ burn residues from other oil residues?

16. Were there any critical results or issues that were not discussed in the report?

17. Does this report present sufficient new data and knowledge, and are the findings useful?

18. What related research has been conducted, and do those results support the findings/conclusions of this study?

PEER REVIEW COMMENT TEMPLATE

Peer Review of the Final Report "Comparison of Physical and Chemical Characteristics of In-Situ Burn Residue and Other Environmental Oil Samples Collected During the Deepwater Horizon Spill Response"

Name: _____
Affiliation: _____
Date: _____

I. GENERAL IMPRESSIONS

Provide overall impressions (approximately 1/2 page in length) addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.

Comments:

II. RESPONSE TO CHARGE QUESTIONS

Provide narrative responses to each of the six charge questions below.

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Comments:

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III. SPECIFIC OBSERVATIONS

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Page	Paragraph	Comment or Question