

**NSWC CRANE
CRANE, INDIANA**

**QUALITY ASSURANCE PROJECT PLAN FOR FULL-SCALE OPERATIONS
AT THE BIOREMEDIATION FACILITY
REV. 2, March 12, 1998**

COMMENT- RESOLUTION

Comments on DRAFT PETN SOP Rev. 1, Dated 03/23/98

Comments by: Allen A. Debus, EPA Region 5. (3/30/98)

Comment 1: First, it is a bit alarming to see that the PETN SOP wasn't apparently written until after the U.S. EPA's request for this document was made. Has SWOK ever utilized this method? On what data sets were the PQLs stated in Table 1 based on? From the Agency's perspective, it is never a good thing to approve an analytical SOP for use if there is no method performance data supporting its use within the laboratory setting. I can't tell if that's the case here or not.

Response:

Southwest Laboratories did not perform analysis of PETN for this project prior to this request. The laboratory did perform this analysis approximately two years ago for a DOE site in which a modified 8330 method was utilized and a separate SOP was not required. This SOP was adapted from their previous explosive SOP and specifically deals with PETN analysis. PQL levels are based on MDL studies performed in the laboratory during the DOE project. The laboratory will analyze at least seven replicates of prepared standards as the current data sets for the PQLs in both soil and liquid analysis prior to starting any sample analysis.

Comment 2: I also am unsure how the PETN will be measured. (I reviewed a draft PETN analysis SOP for another project over a year ago, and that was an SOP for PETN specifically....no other analytes). There are at least three confusing references within the SOP (sections 5.8, 7.4.7, and 8.5) indicating that the PETN may be measured in combination with the other 8330 analytes. If that is the case, why has a separate SOP been written for this compound? (The term "primary analysis" mentioned in section 5.8 could be taken to mean either the analysis on the primary column, or possibly a combined analysis in which the analyte PETN is added to the 8330 analysis.

Response:

The SOP has been modified to clarify the procedures used to measure PETN. Basically, since the nitroaromatics and nitroamines are also being analyzed along with PETN for this project, only one extraction of the sample will be utilized, with two analytical runs. All samples will be spiked with the surrogate 3,4-DNT. Samples will first be analyzed for the nitroaromatics and nitroamines at 254 nm. The wavelength will then be adjusted to 200 nm and the samples re-analyzed for PETN. Surrogate recoveries obtained in the analysis of the nitroaromatics and nitroamines will be used to establish extraction efficiency and matrix effects. The analysis of the Laboratory Control Sample and matrix spikes will further monitor the preparation efficiency and matrix effects for PETN

Comment 3: Just an editorial statement, the outcome of this review will not constitute an audit or a general approval of the SWOK SOP outside of its application to this specific project.

Response:

Comment noted.

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Comment 4: In Section 5.5 or some other appropriate location, there should be references made to a QC check standard used to check the initial calibration. This standard should be prepared from a source independent of that used for initial calibration.

Response:

Comment noted. The use of a second source standard to check the initial calibration has been added to section 5.6 of the revised SOP.

Comment 5: In section 5.5.2, add the phrase, "...upper extent of the.." before the phrase "detector linear range".

Response:

Comment noted and incorporated into next revision of this SOP.

Comment 6: Section 5.6 contains a reference to a Table 2 which was not appended to my copy of the QAPP.

Response:

The reference to Table 2 was made in error and has been deleted from the updated SOP.

Comment 7: The term "V.F." appears in section 5.7 and in another place, but it is undefined.

Response:

The term V.F. has been replaced in the updated SOP with the term verification.

Comment 8: Section 5.8 proposes not to use the surrogate 3,4 DNT because it will be used in another analysis, Method 8330. I do not understand the logic behind this, and it would be a good idea to include a surrogate in the PETN analysis.

Response:

Since Southwest Laboratories is performing one extraction and dual analysis at separate wavelengths, it is the laboratory's rationale that any extraction inefficiencies or matrix effects on the surrogate will be demonstrated in the analysis of the nitroaromatics and nitroamines. If the surrogate recovery is not within control limits due to the conditions listed above, then the LCS and matrix spike data for PETN would be evaluated to confirm the causes.

Comment 9: At the end of the sentence in Section 6.1 a phrase should be added, "..., or those developed for site specific cases."

Response:

Comment noted and incorporated into next revision of this SOP.

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Comment 10: The word, "shook" should be changed to "shaken" in Section 7.1.1.

Response:

Comment noted and incorporated into next revision of this SOP.

Comment 11: The subject of Low Level Aqueous Sample Analysis seems to have been omitted from the SOP. Would an SPE device/procedure be used?

Response: Low level aqueous analysis of PETN is not required for this project and therefore was not addressed in the SOP. The laboratory has not performed this analysis in the past and the need has not been encountered.

Comment 12: Tom Jenkins of the CRREL now recommends only a 2 to 4 hour sonication period for method 8330 explosives (to avoid compound degradation). So I am wondering whether the 18 hour sonication period for PETN may be excessive. SWOK should determine the appropriateness of the proposed 18 hour period for PETN by contacting Mr. Jenkins.

Response: All analysis reported to date has been performed utilizing the 18 hour extraction for explosives as specified in method 8330. The laboratory will change this requirement upon receipt of written confirmation to deviate from the published method.

Comment 13: At the end of section 7.1.2.3, a phrase should be added, "...but not for longer than 40 days."

Response:

Comment noted and incorporated into next revision of this SOP.

Comment 14: The equation for %RSD on page 7.3 should have the denominator changed to read "mean Calibration factor".

Response:

Comment noted and incorporated into next revision of this SOP.

Comment 15: In the first line of section 7.4.3, the word "standards" should be changed to singular.

Response:

Comment noted and incorporated into next revision of this SOP.

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Comment 16: I am uncertain as to whether or not the PQLs indicated in Table 1 are really measurable reporting limits, or simply MDLs. How or where was this information derived from?

Response:

The water PQL was derived from a multiplier of the MDL and the low level calibration standard. The soil PQL is based on the low level calibration standard. New MDLs are being processed at this time for both soil and water matrices and will be completed before samples are analyzed.