



## OSS Oil in Anhydrous Ammonia Validation Summary

This document serves as a summary of the verification data collected in the development of the OSS Oil in Anhydrous Ammonia test method. An overview of this data can be found below. Validation of this method occurred in the OSS facility in Bangor, ME as well as various CF facilities. It should be noted that the calibration, detection range, and repeatability limits of this method are subject to change as development of the sampling procedures continues. Reproducibility and bias will be defined as laboratory comparability data is collected from additional end-user facilities. The procedures used to determine the precision data and method limits is detailed in this report.

Method Data Summary		
MDL	Detection Range	Repeatability at 3.5 mg/L
0.2 mg/L	0.2 - 20 mg/L	9%

Table 1: Summary of the limits determined in method development

Detection Limit:

*Method Detection Limit (MDL)* – Seven replicates of anhydrous ammonia at the residual tank concentration were analyzed by the OSS Oil in Anhydrous Ammonia detection method. The MDL was calculated by multiplying the standard deviation of the seven replicate analyses by the Student's t value for (n – 1) degrees of freedom, where n equals the number of replicates. The Student's t value for seven replicates is 3.143. See Table 1. The study laboratory determined the MDL.

Method: Oil in Anhydrous Ammonia						
Fortification Level: residual tank concentration - ~0.2 mg/L						
Measured Result mg/L as Oil						
Replicate #1	Replicate #2	Replicate #3	Replicate #4	Replicate #5	Replicate #6	Replicate #7
0.17	0.14	0.31	0.27	0.24	0.21	0.31
Average of Seven Replicates: 0.24						
Standard Deviation of Seven Replicates: 0.067						
MDL (Student's t 3.143 x Standard Deviation) = 0.21						

Table 2: Method Detection Limit Study Data



Repeatability:

A simple repeatability study was conducted at a concentration of 3.5 mg/L. Oil concentration was tested eight times from a single sample. Results can be found below.

Rep.	Recovery (mg/L)
1	3.5
2	3.4
3	3.7
4	3.5
5	3.5
6	3.5
7	3.8
8	3.4
Average	3.5
St. Dev.	0.15

Table 3: Raw data from the repeatability study.

Repeatability limit ( $r$ ) - Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the " $r$ " value for that material; " $r$ " is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

Material	Average	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	$s_r$	$r$
Oil in Anhydrous Ammonia	3.5	1.5	9%

Reproducibility and bias of this method will be determined as additional data is collected.



Calibration data and method accuracy:

Figure 1 is a representation of all spike data performed to this date via the OSS Oil in Anhydrous Ammonia method. The data is linear with an  $R^2$  value of 0.972.

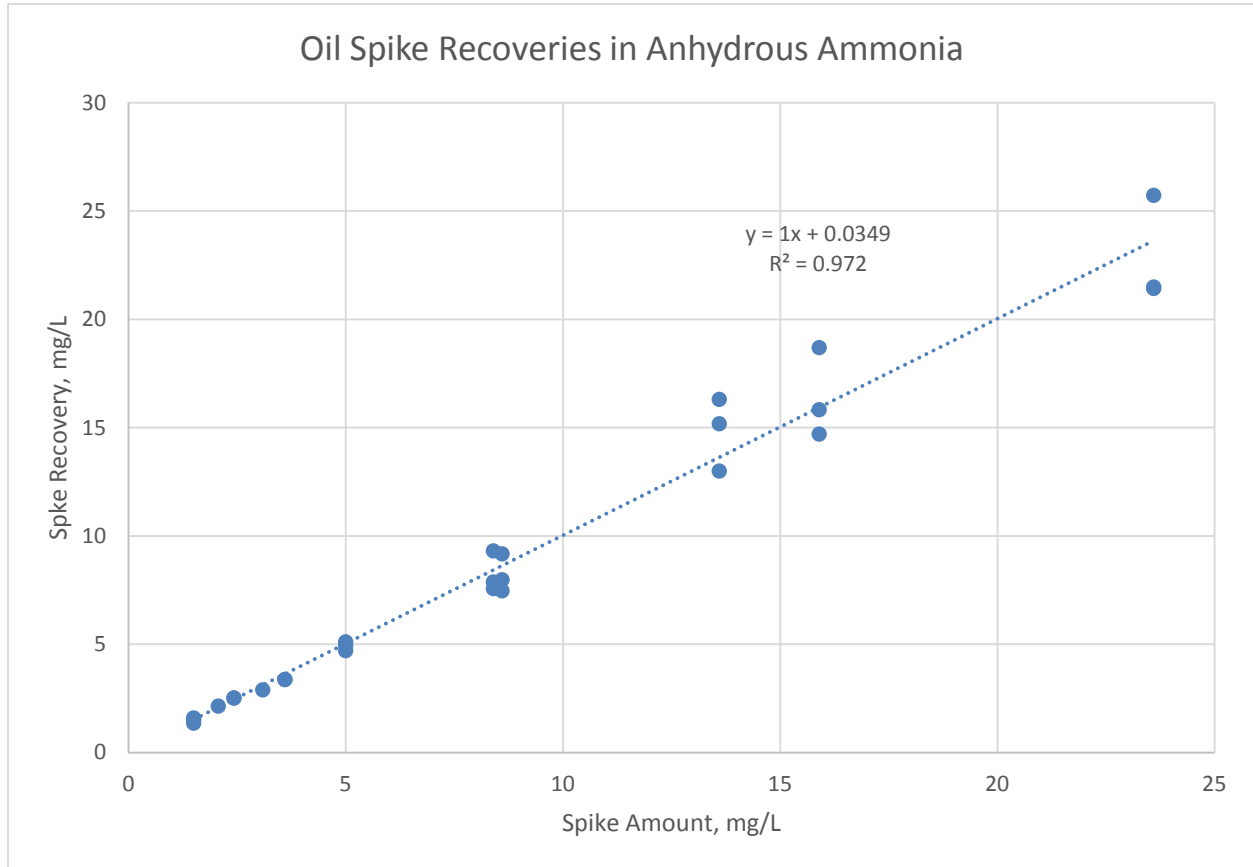


Figure 1: Oil Spike Recoveries in Anydrous Ammonia by OSS Method