# COMPARISON OF FIELD SCREENING TECHNOLOGIES IMPLEMENTED DURING PHASE I REMEDIATION OF EXPLOSIVE WASHOUT LAGOON SOILS

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## INTRODUCTION

This report presents a comparison of field screening methods used to determine 2,4,6-trinitrotoluene (TNT) and hexhydro-1,3,5-trinitro-1,3,5-triazine (RDX) concentrations in soil during Phase I of the Explosives Washout Lagoon Soil Remediation at the Umatilla Army Depot Activity (UMDA), Hermiston, Oregon. Field screening methods employed during this activity are described and a comparison of the results, accuracy, precision, flexibility, analysis time, sample size, skill level, costs, and other considerations are discussed.

UMDA was established as an Army ordnance depot in 1941 for the storing and handling of munitions. From the 1950s until 1965, UMDA operated an onsite explosive washout plant that processed munitions to remove and recover explosives. Operation of the plant included flushing and draining the explosive washout system. The wastewater was discharged through an open metal trough into two unlined infiltration basins known as the Explosive Washout Lagoons.

The Explosive Washout Lagoons were characterized as a potentially hazardous site in the initial installation assessment. In 1981, an approximate 45-acre plume of RDX was identified in the shallow groundwater aquifer apparently resulting from discharges to the lagoons. Subsequent investigations confirmed the presence of explosives in the soil and groundwater.

During Phase I of the Washout Lagoon Soil Remediation, soil from the lagoons, berms, trough, building, and surrounding areas were removed by the U.S. Army Corps of Engineers (USACE) contractor, Wilder Environmental, Inc. (Wilder). Soils with a concentration of TNT or RDX above 30 mg/Kg were excavated using a pre-established grid system and stockpiled in a containment structure (1). Wilder used EPA SW-846 Method 8510 and 8515 colormetric analyses based on Jenkins (2) and Walsh and Jenkins (3) for field screening to determine soil TNT and RDX concentrations. For the purpose of this report, field screening methods for RDX and TNT used by Wilder will be referred to as the Jenkins Method. When the field screening results indicated a clean grid, (RDX and TNT results were below 30 mg/Kg) confirmation samples were sent to a Wilder subcontract laboratory, Precision Analytical (Precision) for EPA SW-846 Method 8330 analyses of TNT and RDX. Depth of excavation and completeness of Phase I remediation were based on Precision Analytical results. Additionally, samples from observed hot spots were also field screened and a portion of these hot spot samples were also analyzed by Precision.

BVWS provided U.S. EPA oversight during a majority of the Phase I Soil Remediation. BVWS activities included field screening using DTECH® and Quantix® immunoassay field screening methods, providing samples to Ensys® Laboratory for their EPA SW-846 Method 8510 and 8515 colormetric field screening method, and providing samples to DataChem Laboratories, Inc. (DataChem) for EPA SW-846 Method 8330 explosives analysis.

Field screening and laboratory analyses during the Phase I Remediation were performed to ensure cleanup requirements were being met and to compare field screening techniques to determine appropriate methods for future explosive remediation activities, including groundwater remediation and Phase II soil bio-

remediation at the Explosive Washout Lagoons.

## METHODOLOGY

All soil sample collection was performed by Wilder. Five point composite samples for each grid were taken following excavation as directed by the USACE representative. All analytical testing was conducted from splits of soil collected. For each sample, a sufficient volume of soil was collected in a ziplock bag, thoroughly composited by mixing the soil within the bag then placed in appropriately labeled sample containers. Original Plans and Specifications (4) required each lift of soil excavation to be one foot. Primarily because of relatively high TNT and RDX concentrations in the lagoons and inner berm grids, approximately two foot excavations were conducted on the upper lifts. Once a grid exhibited field screening analysis concentrations below 30 mg/Kg for both TNT and RDX, a composite split from the sample was sent to Precision for confirmation analysis. At the completion of excavation activities five discrete samples per grid in the lagoons and inner berms were collected (40 samples total) and analyzed by Precision. During excavation activities fine-grained soils initially colored brown that contained relatively high concentrations of TNT and RDX, would change to a distinctive red to orange color within 24 to 48 hours of exposure to atmospheric conditions. Observed hot spots (red/orange soil) were also sampled and selectively excavated.

Samples collected from the grids and hotspots were split three ways by Wilder. One split was field screened by the Jenkins Method using EPA SW-846 Method 8515 and Method 8510 (2, 3); then sent to Precision if field screening indicated a grid was below cleanup levels. If the grid was not below cleanup levels, then the remaining soil from the split was added to the excavation stockpile. Two sample splits were released to BVWS. One split was shipped to DataChem for EPA SW-846 Method 8330 analysis. Soil from the final split was field screened using DTECH® and Quantix® commercial methods. A portion of the remaining soil was sent to Ensys® for analysis and remaining soil samples were archived.

DTECH® TNT and RDX immunoassay methods can be used for both soil and water. The soil detection range without dilutions is from 0.5 to 5.0 mg/Kg for TNT and from 0.5 to 6.0 mg/Kg for RDX. These ranges can be expanded with prepackaged 10:1, 100:1, 1000:1, and 10,000:1 dilution kits. Water detection ranges are from 5 to 45 µg/Lfor TNT and RDX.

The general procedure for DTECH® TNT and RDX field screening is:

- Extract TNT/RDX from soil using acetone. The sample extraction can be used for TNT and RDX analyses.
- Dilute extraction with acetone if necessary.
- Transfer extraction to water-based solution.
- Prepare reference and test solutions.
- Filter both solutions through membrane.
- Flush the membrane.
- Add a color solution.
- Read development of test relative to reference after approximately 10 minutes using a color chart or DTECHTOR®.
- Determine concentration range.

DTECH® results can be obtained in two ways. When a color chart is used, the reference side of the test well is matched to a reference color on the chart, the sample test side is then compared to the color chart to determine the concentration range. However, during Phase I remediation, the colors printed on the chart tended to have a gray tint and did not effectively match the color of the test, creating a high degree of subjectivity.

The alternative method for obtaining DTECH® results is to use the DTECHTOR® to read the difference in absorbance between the test and reference side. The DTECHTOR® produces a number which is correlated to a concentration range. BVWS utilized the DTECHTOR® during Phase I. In a conservative approach three DTECHTOR® measurements were taken and the highest measurement was selected for the concentration range.

In an effort to obtain an actual concentration for comparison purposes BVWS assumed a linear correlation between the DTECHTOR® measurement and the concentration range. The DTECHTOR® measured value was used to generate an estimated concentration. Both the estimated concentration and the concentration range were used during the determination of precision and accuracy of results.

DTECH® analyses appear to be temperature dependant. At 0° F the test developed consistently in 10 minutes. As the field conditions became warmer, the test developed quicker, and the reference side of the test well would develop fully in less than 10 minutes. The test side would continue to develop, leading to a smaller absorption difference between the two wells. At higher temperature ranges, DTECHTOR® readings could significantly drop in a relatively short time, potentially creating a change in the concentration range.

The Quantix® TNT immunoassay method can be used for water and soil. Quantix® does not currently produce an RDX field screening test product. The detection range without diluting is from 0.26 to 100 mg/Kg in soil. Quantix® also provides a 100:1 dilution solution for higher TNT concentrations.

The general procedure for the utilizing Quantix® TNT kit is:

- Extract TNT from soil using acetone.
- Dilute with water (if necessary).
- Transfer extraction or dilution to dilutant tube (water).

- Create standard curve in first 14 wells of a plate which contains 96 wells.
- Transfer test solution to remaining wells. (Each sample test solution is placed in two separate wells, creating a replicate test).
- Add conjugate and let sit for 30 minutes.
- Wash wells.
- Add substrate and chromogen (color solution) and let sit for 30 minutes without exposure to light.
- Add stop solution.
- Read absorption with Microreader.
- Print results.

Quantix® results are obtained by reading the absorption in each well of a plate with a Microreader. The Microreader creates a standard curve with each batch. Each sample is replicated in an adjacent well. These wells are read and the average absorbance for both wells is compared to the standard curve for that batch to determine sample concentration. The absorbance and concentration of each well and an average absorbance and concentration for each sample is provided in a printout from the Microreader.

The Ensys® TNT test is a commercially available colormetric method used for soil. The Ensys® field screening method was not used by BVWS, rather the Ensys® testing was performed by Ensys® at their facilities in North Carolina. The detection limit without dilution is from 1 to 30 mg/Kg. Ensys® also conducted RDX colormetric screening of soil. This method is still in development and not currently commercially available. Therefore, information about this method is limited. According to Ensys®, the general testing procedures for TNT is:

- Dry sample to < 10 percent moisture. This step is not required but increases the accuracy.
- Weight sample.
- Extract TNT from soil using acetone.
- Dilute with acetone (if necessary).
- Filter into sample cuvette.
- Read absorbance ($Abs_{IN}$).
- Add developer solution and shake.
- Read absorbance ($Abs_{SAMP}$).
- Calculate concentration.

Ensys® results are calculated using the following equation:

$$TNT\ (ppm) = \frac{((Abs_{SAMP} - 4(Abs_{IN}))}{0.0323}$$

The Jenkins test is a colormetric method that can be used to determine TNT and RDX in soil and water. A preconcentration step is required for water samples (5). This method is not a packaged field screening product. This method was not performed by BVWS personnel, although BVWS observed field screening procedures. The quantification range without dilution is from 1.11 to 22.3 mg/Kg for TNT and from 1 to 20 mg/Kg for RDX.

Jenkins results are calculated using the following equations:

$$TNT\ (ppm) = \frac{((Abs_{SAMP}) - 2\ (Abs_{IN}))}{Response\ Factor}$$

$$RDX\ (ppm) = \frac{Abs_{SAMP}}{Response\ Factor}$$

The general procedure utilized by Wilder for the Jenkins method for TNT includes:

- Produce a standard curve (one per day) and determine a response factor.
- Weigh sample.
- Extract TNT and RDX from soil with acetone. The same extraction can be used for TNT and RDX.
- Dilute with acetone (if necessary).
- Filter extraction.
- Read absorbance ($Abs_{IN}$).
- Add KOH and shake.
- Filter sample.
- Read absorbance ($Abs_{SAMP}$).
- Calculate concentration.

General procedures for RDX analysis include:

- Produce a standard curve (one per day) and determine a response factor. (Some problems were encountered in establishing the proper RDX standard curve. The curve was generally a good fit, but occasionally flat)
- Weigh sample.
- Extract soil with acetone.
- Dilute with acetone (if necessary).
- Filter extract through ion exchange resin.
- Mix with acetic acid.
- Add Zinc dust and filter.
- Add water and powder pillow and shake.
- Let stand for 10-15 minutes.
- Read absorbance ($Abs_{SAMP}$).
- Calculate concentration.

A daily calibration is not needed if only one compound is being analyzed. An initial calibration, with daily checks of a single known concentration is sufficient. Since the Jenkins method was used daily for both RDX and TNT analysis, daily calibrations were performed.

Acetone with up to 3 percent water is recommended for dilutions. Using pure acetone for dilutions may cause low results, especially for dry soils and large dilutions.

Of the field screening methods evaluated, DTECH® and Jenkins can be used for single sample or batch type analysis. Each DTECH® kit provides sufficient materials for four tests. Generally, up to 8 tests were conducted in each batch based on ease of product use. Jenkins RDX

testing can be run in batches of up to 6 or 7 samples. Jenkins TNT testing must be run as a single sample, but extractions can be conducted in batches. The Quantix® kit is designed to run up to 41 samples in a batch. Less can be run, yet because a standard curve should be run with each batch, running smaller batches increases the per sample cost. Ensys® samples are run individually but extraction can be done in batches.

DTECH® required between 90 and 120 minutes to extract and analyze 8 samples for TNT and RDX. Conducting less sample tests does not significantly reduce the analyses time.

Quantix® required approximately 150 to 210 minutes to run between 20 and 40 samples for TNT. The minimum time for a smaller Quantix® test batch is approximately 2 hours. Testing time does not significantly increase with the number of samples run, but the extraction and dilution preparation increase the overall procedure duration.

Based on discussions with Ensys®, approximately 10 samples can be run in 30 to 35 minutes in a laboratory setting. In field operations, analysis time will take longer, approximately 45 to 50 minutes for 10 samples, not including the recommended drying step.

The Jenkins method requires approximately 30 minutes to weigh and extract 6 samples. The TNT analysis requires approximately 5 minutes per sample. A batch of 6 RDX samples requires approximately 30 minutes to complete the analysis step.

Soil samples are by nature heterogeneous. This can be only partially overcome by thorough mixing the sample prior to splitting and analysis. Therefore, the larger the amount of soil tested, the more representative the result. The colormetric methods, Jenkins and Ensys®, recommend 20 grams and 10 grams of soil, respectively, as compared to the immunoassay methods, DTECH® and Quantix®, which use approximately 4.5 grams and 4.2 grams of soil, respectively. Colormetric results are therefore potentially more representative of actual soil explosive concentration. The drawback to using more sample material is that more acetone must be used during the extraction step which requires proper disposal. Acetone requirements per sample are: 100 mL for Jenkins method, 50 mL for Ensys®, 9 mL for DTECH®, and 21 mL for Quantix®.

The DTECH® procedure is the simplest method and required the least training. Detailed instructions are provided. All reagents are premeasured and transferring of liquids is by eyedroppers and standardized pipettes.

The Quantix® method is more difficult than DTECH® and should be initially attempted with guidance as a trial batch. The extraction bottles and the dilution tubes are premeasured, which prevents error and all liquids are transferred using standardized pipettes. The Quantix® method requires the use of instruments, including the Microreader, the washer, and repeating pipettes. The instructions provided do not clearly explain all steps in the process.

The Ensys® procedure was not conducted during this test. Ease of use of the Ensys® product is unknown.

The Jenkins method is not a prepackaged product and requires general chemical laboratory skills including the assembly of materials, chemicals, explosive standards, the use of a spectrophotometer and relatively simple calculations to determine concentrations. This method also requires the proper cleaning of appropriate laboratory glassware.

The DTECH® field screening product contains three separate kits, an extraction kit which costs approximately $25, a TNT test kit which costs approximately $100, and an RDX test kit which costs approximately $100. Dilution bottles were provided for this project at no charge. Each kit contained all the material and equipment to run four samples, resulting in a per sample cost of approximately $60 for TNT and RDX results. When considering costs per sample, unusable tests must be factored in. Unusable tests may be significant if concentrations are unknown and many dilutions have to be run on single samples. Each dilution requires a separate test. The DTECHTOR® can be purchased for approximately $300. Utilizing the DTECHTOR® is recommended to reduce subjectivity.

The Quantix® method only determines TNT. The cost of a single soil TNT kit is $840 which will run approximately 40 samples ($21 per sample), assuming only one standards curve is created. The Quantix® TNT kit does not include everything required for field screening. A lab station which contains the Microreader, Well Washer, pipettes and tips required for screening cost $5880. This lab station was loaned to BVWS at no charge during the Phase I field activities.

An Ensys® TNT soil test kit costs $380 for 20 samples. The acetone required for the extraction is not provided. The approximate cost per sample is between $20 and $25. The Hach spectrophotometer, balance and cuvettes can be rented for $160 per day or $400 per week from Ensys®, or purchased separately for approximately $2,000. These materials are required for this method.

All materials for the Jenkins method must be purchased separately, including acetone, filters, syringes, vials, KOH, zinc dust, powder pillows, acetic acid, and the Hach spectrophotometer. The Hach spectrophotometer can be purchased for approximately $1600 or can be rented on a daily or weekly basis at prices similar to Ensys®. Materials to run TNT and RDX are estimated to cost between $10 and $15 per sample. Explosive standard must also be obtained to conduct calibration.

DTECH® requires little set up time and little room to run, approximately half the area of a desk is sufficient area. All kits are packaged together and easily transportable, but there is a significant amount of

packaging that requires disposal. This product requires no electricity or refrigeration. Although the product does not require refrigeration, the materials and the test in general become relatively unstable at temperature extremes. DTECH® kits should not be frozen.

Due to small DTECH® detections ranges samples may have to be diluted and the correct dilution must be chosen. Numerous tests were unusable because the wrong dilution resulted in values above or below the detection ranges. The degree of red color in the extract provided a good identification of the concentration of TNT. Operator intuition was valuable in determining dilution requirements. There was no color indication for RDX concentration in extracts. If the primary objective of field screening was to determine whether the soil is above or below a known concentration, then choosing the right dilution would not be a problem.

Quantix® kits require a somewhat larger area to run the test comfortably. A power source is required for the Microreader and the washer. Deionized water is also needed for the wash step. Quantix® kits must be kept refrigerated, but they should not be frozen. Traveling cases are provided, but assembling all the materials and equipment requires time and effort.

Ensys® requires slightly less space to run than Quantix®. A Hach spectrophotometer that is battery or electrically powered can be obtained so a constant power source may not be required. Room temperature storage is recommended, but not required. The Hach spectrometer can run in temperatures between 40-100°F.

The Jenkins method required more room, approximately 2 large desk areas to efficiently set up and run the analyses. Acetone used during extraction and must be properly disposed. Time must be spent setting up the work area to efficiently run the analyses. Once the work area is set up, significant effort would be required to relocate because materials are not prepackaged. Since the method is not a packaged kit all materials must be ordered separately. Most filters, pillow packs, and other method requirements can be purchased through laboratory supply catalogs. Acetone can be purchased at local hardware stores. For most government projects, the U.S. Army Environmental Center will provide explosive standards. Standards are available commercially, but may be difficult to locate and obtain. The Hach spectrometer used by Wilder was adaptable for electricity or a battery, so a constant power source was not required. Deionized water is required for the rinsing of vials and cuvettes. The analysis should be run at room temperature.

## FIELD SCREENING COMPARISON

DataChem Laboratory results were used as a baseline to evaluate accuracy comparison. A total of 155 samples were analyzed by DataChem. No sample had RDX concentrations below detection limits, 11 samples had TNT concentrations below detection limits. The highest detections were 2,800 mg/Kg for RDX and 9,300 mg/Kg for TNT. TNT analysis was performed on 125 samples by DTECH®, 151 samples by Quantix®, 149 samples by Ensys®, and 104 samples by Jenkins method. RDX analysis was performed on 122 sample by DTECH®, 149 samples by Ensys®, and 66 samples by Jenkins method. A comparison of sample method and range is in Table 1.

The DTECH® method provides a range in which the sample concentration is expected, while the other methods produce a single concentration.

In an effort to obtain an actual concentration BVWS assumed a linear correlation between the DTECHTOR® measurement and the concentration range based on field observations. The DTECHTOR® measured value was used to generate an estimated concentration. Both the estimated concentration and the concentration range were used to determine precision and accuracy of results.

Accuracy is a measure of bias in the testing and analyses procedures. The closer a value of a measurement agrees with the true value, the more accurate the measurement. For comparison of field screening results, DataChem reported concentrations were considered the "true value". All Datachem results were validated (6) and determined useable; no DataChem results were rejected.

The accuracy of field screening results were estimated by two separate methods. One method compared the relative percent difference (RPD) between DataChem results and each field screening method results. The second method used linear regression graphs of the field screening concentration versus the DataChem concentrations.

The RPD between DataChem concentrations and field screening concentrations was calculated where:

$$RPD = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100$$

$D_1$ = Field Screening Concentration
$D_2$ = DataChem Concentration

Based on standard validation procedures (7) an RPD criteria of ±50 percent is considered acceptable. The lower the absolute value of the RPD, the closer the two values. For comparison purposes, field screening results with an RPD of ± 50 percent was established as acceptable. RPD values within this range were considered accurate. Table 2 contains the percentage of the data set that met the RPD criteria; that were biased high (field screening result RPD value is greater than 50); that were biased low (field screening RPD value is less than -50); and n, the total number of RPD calculations conducted for each concentration range.

DTECH® results are reported in a concentration range. For the purpose of this study an estimated DTECH® value was determined by plotting the value of the DTECHTOR® reading on a graph of DTECHTOR® values versus concentrations. The DTECH® estimated value was then compared to DataChem concentrations to determine an RPD value.

The DTECH® reported range for each sample was also used to determine accuracy. For each sample the DTECH® range value was determined accurate if the DataChem concentration was within the reported DTECH® range.

As shown in Table 2, TNT accuracy based on RPD calculations were relatively low for all field screening methods at DataChem concentrations less than 30 mg/Kg. At DataChem concentration groups of 30 to 100 mg/Kg, 100 to 1,000 mg/Kg and greater than 1,000 mg/Kg, the Jenkins field screening method was the most accurate followed by Ensys® with Quantix® and DTECH® having lower RPD based accuracy percentages.

RDX accuracy based on RPD calculations were relatively low for all field screening methods at all DataChem concentration groups. Ensys® field screening was the most accurate with Jenkins and DTECH® having lower RPD accuracy percentages. All three method results were consistently biased low. Quantix® does not currently have a commercial RDX field screening method.

TNB (1,3,5-trinitrobenzene) is reported to cause positive interference of TNT results in colormetric field screening methods (2). To determine the effects, if any, of TNB interference on TNT results and potential HMX interference on RDX results, an RPD based accuracy determination was conducted. This determination compared the combined DataChem TNT and TNB against colormetric field screening TNT results. At TNT concentrations less than 30 mg/Kg, RPD based accuracy increased relative to TNT RPD based accuracy for both Jenkins (+30.1 percent) and Ensys® (45.2 percent). At DataChem concentration groups greater than 30 mg/Kg TNT + TNB RPD based accuracy decreased or stayed the same for both colormetric field methods relative to TNT RPD based accuracy.

Manufacturers of the immunoassay test report a cross reactivity with TNB as 47 percent for Quantix® and 23 percent for DTECH®. The TNB concentration was multiplied by the percent cross reference reactivity and added to the TNT concentrations. Tetryl; 2-amino-4,6-dinitrotoluene; 2,4-dinitrotoluene; 4-amino-2,6-dinitrotoluene; 2,4-dinitroaniline; and 1,3-dinitrobenzene are also known to affect immunoassay TNT test, but concentrations and/or cross reactivity of these compounds are low and were not included in the interference evaluation.

Quantix® accuracy increased by 33.6 percent for concentrations less than 30 mg/Kg and decreased by 5 percent for concentrations from 10 to 100 mg/Kg and by 4.6 percent for concentrations from 100 to 1,000 mg/Kg. Quantix® accuracy for concentrations greater than 1,000 mg/Kg did not change. DTECH® accuracy decreased by 2.2 percent for concentrations less than 30 mg/Kg and 13.3 percent for concentrations from 30 to 100 mg/Kg. Accuracy for higher concentrations did not change.

A similar comparison was conducted for combined DataChem RDX and HMX versus field screening results for RDX. RDX + HMX RPD based accuracy increased only slightly for Jenkins (2.3 percent) at DataChem concentrations of less than 30 mg/Kg RDX. For all other DataChem concentration groups field screening method RDX + HMX RPD based accuracy decreased relation to RDX RPD based accuracy.

A second evaluation of accuracy included linear regression graphs in which the individual field screen method result was plotted versus the DataChem concentrations for each sample. Graphs were compared in each of the four DataChem concentration groups (<30, 30-100, 100-1000, >1000 mg/Kg). These graphs contain two best fit lines, one of which has a zero y-intercept. Under ideal conditions true accuracy would have a slope of 1 on the best fit line and an $R^2$ (correlation coefficient) of 1.0. A slope of less than 1 indicates that field screening results are generally less than DataChem results for each sample. A slope of greater than 1 indicates that field screening results are generally greater than DataChem results. The closer the $R^2$ value is to 1.0, the better the accuracy is for each data group. Because DTECH® sample range could not be used only DTECH®, estimate values were used to calculate linear regression values.

Table 3 contains linear regression parameters for the best-fit line (n = number of samples, slope and $R^2$) for each of the field methods for both TNT and RDX in the four DataChem concentration groups. TNT linear regression parameters indicate a low accuracy for all field methods in the less than 30 mg/Kg concentration range. Accuracy based on linear regression analyses improved in the 30 to 100 mg/Kg, 100 to 1000 mg/Kg, and greater than 1,000 mg/Kg concentration groups. These data indicate a high degree of variability and a wide scatter of points, indicating both analytical variation associated with the soil matrix and the variability of each field screening method.

RDX linear regression parameters indicate that most field screening results were consistently lower than associated DataChem concentrations.

Precision is the measure of mutual agreement among replicate or duplicate measurements of the same analyte. The closer the numerical values of the measurement are to each other, the more precise the measurement. Replicates and duplicate analysis assist in measuring the precision. A replicate analysis is when two tests are performed on the same extraction. The precision criteria

for replicate analyses requires an RPD ± 50 percent.

A duplicate analysis is when one sample is extracted twice and each extraction is tested. Due to natural soil conditions and contaminant characteristics, even a well mixed soil sample is not entirely homogenous. Therefore, the precision goal for duplicate analyses was met if a factor of less than 5 times existed between the sample and the duplicate.

For the purposes of this evaluation the DTECH® replicate analyses must have the same range to meet precision requirements. DTECH® duplicates met the precision criteria if a factor of less than 5 times existed between the midpoints of the sample and duplicate ranges.

The percentages of samples that met the replicate precision criteria for each field screening method are: 80 percent and 100 percent for DTECH® TNT and RDX, 100 percent and 96 percent for ENSYS® TNT, and RDX and 88 percent for Quantix® TNT. All duplicate precision criteria were met. Wilder performed the Jenkins method field screening and were not required to perform duplicate or replicate analyses, therefore precision information is not provided for this method.

## SUMMARY AND CONCLUSIONS

A summary of comparisons are presented in Table 4. Based on the comparisons, no single method significantly out-performed other methods. Accuracies for all the field screening methods were comparable, with Jenkins and Ensys® being higher in the >30 ppm TNT ranges and DTECH® being higher in the <30 ppm range. None of the available methods were accurate for RDX. Therefore, it is likely that field conditions will determine which field screening method will be chosen.

DTECH's® primary strengths are its ease of use; lack of space, setup, and power requirements; and availability of TNT and RDX tests for soil and water. DTECH® tests can be conducted under remote field conditions, where other methods requiring space and power and lacking transportability cannot. For a small number of samples, DTECH® is the cheapest field screening method, primarily because it does not require an initial investment. Although, depending on the amount of dilution- related unusable tests, the cost per sample may increase. DTECH® is also the only manufacturer that provides a packaged RDX field screening kit for water.

Quantix® is the most efficient method for running large numbers of samples. Up to 40 samples can be analyzed in 3.5 hours. Quantix® also has a large detection range so dilutions are not required as often. This results in less unusable test with concentrations above or below the detection range due to choosing an improper dilution level. Diluting with water may cause inaccuracies at high concentration because of the differences of TNT solubility in acetone and water. It is not known at what concentration dilution method becomes significant.

Quantix® and DTECH® immunoassay methods are preferred over Ensys® and Jenkins colormetric methods when TNB concentrations are expected to be high and may interfere with field screening objectives because of lower cross reactivity. In some field screening applications, it may be important to know more than TNT and RDX concentrations. The color development of the extract in colormetric tests can give the operator an indication of what type of compounds are present in the soil. In general, TNT and TNB tend to turn red, tetryl orange; dinitrobenzene (DNB) purple; 2,4-dinitrotoluene (2,4-DNT) blue; 2,6-DNT pink; and humic material yellow. In immunoassay tests, there is no color development of the extract, so the presence of other compounds cannot be visually identified.

Ensys® was not used by BVWS, therefore little is known about the conditions and requirements for this method.

Jenkins had the highest accuracy for TNT concentrations above 30 ppm and is the cheapest method for large sampling efforts. Jenkins is the overall preferred method if site conditions allow.

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Table 1
Samples per Range

| | < 30 mg/Kg | | 30-100 mg/Kg | | 100-1000 mg/Kg | | > 1000 mg/Kg | |
|---|---|---|---|---|---|---|---|---|
| | TNT | RDX | TNT | RDX | TNT | RDX | TNT | RDX |
| DataChem | 111 | 73 | 17 | 46 | 22 | 33 | 5 | 3 |
| DTECH | 88 | 59 | 15 | 35 | 17 | 25 | 5 | 3 |
| Quantix | 107 | NA | 17 | NA | 22 | NA | 5 | NA |
| Ensys | 106 | 71 | 17 | 43 | 21 | 32 | 5 | 3 |
| Jenkins | 73 | 44 | 13 | 20 | 15 | 2 | 3 | 0 |

Table 3
Linear Regression Parameters

| TNT | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | < 30 mg/Kg | | | 30-100 mg/Kg | | | 100-1000 mg/Kg | | | > 1000 mg/Kg | | |
| | N | Slope | $R^2$ | N | Slope | $R^2$ | N | Slope | $R^2$ | N | Slope | $R^2$ |
| DTECH, est | 88 | 1.36 | 0.45 | 15 | 0.79 | 0.42 | 17 | 1.81 | 0.33 | 5 | 1.05 | 0.91 |
| Quantix | 107 | 2.08 | 0.02 | 17 | 0.78 | 0.27 | 22 | 1.29 | 0.007 | 5 | 0.48 | -0.46 |
| Ensy | 106 | 2.87 | 0.08 | 17 | 1.02 | 0.12 | 21 | 1.22 | 0.35 | 5 | 1.04 | 0.94 |
| Jenkins | 73 | 2.33 | 0.41 | 13 | 0.91 | 0.65 | 15 | 1.34 | 0.45 | 3 | 1.15 | 0.55 |
| **RDX** | | | | | | | | | | | | |
| | < 30 mg/Kg | | | 30-100 mg/Kg | | | 100-1000 mg/Kg | | | > 1000 mg/Kg | | |
| | N | Slope | $R^2$ | N | Slope | $R^2$ | N | Slope | $R^2$ | N | Slope | $R^2$ |
| DTECH, est | 59 | 0.1 | 0.30 | 35 | 0.24 | -0.13 | 25 | 0.21 | 0.18 | 3 | 0.17 | 0.89 |
| Ensys | 71 | 0.85 | -0.06 | 43 | 0.68 | 0.18 | 32 | 0.65 | 0.25 | 3 | 0.70 | 0.85 |
| Jenkins | 44 | 0.44 | 0.14 | 20 | 0.55 | 0.67 | 2 | 1.06 | 0.59 | No Data | | |

# Table 2
## Relative Percent Difference Accuracy Determinations

**TNT RPD ACCURACY**

| | <30 mg/Kg | | | | 30-100 mg/Kg | | | | 100-1000 mg/Kg | | | | >1000 mg/Kg | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Bias | | | | Bias | | | | Bias | | | | Bias | | |
| | RPD[1] | + | - | n | RPD[1] | + | - | n | RPD[1] | + | - | n | RPD[1] | + | - | n |
| DTECH estimate | 47.7 | 35.2 | 17.1 | 88 | 60.0 | 0 | 40.0 | 15 | 47.1 | 17.6 | 35.3 | 17 | 40.0 | 0 | 60.0 | 5 |
| DTECH range[2] | 54.5 | 30.7 | 14.8 | 88 | 26.7 | 6.7 | 66.6 | 15 | 35.3 | 23.5 | 41.2 | 17 | 20.0 | 20.0 | 60.0 | 5 |
| Quantix | 23.4 | 68.2 | 8.4 | 107 | 58.8 | 0 | 41.2 | 17 | 54.6 | 22.7 | 22.7 | 22 | 40.0 | 20.0 | 40.0 | 5 |
| Ensys | 20.8 | 78.3 | 0.9 | 106 | 82.3 | 11.8 | 5.9 | 17 | 66.7 | 19.0 | 14.3 | 21 | 80.0 | 0 | 20.0 | 5 |
| Jenkins | 31.5 | 64.4 | 4.1 | 73 | 92.3 | 0 | 7.7 | 13 | 80.0 | 13.3 | 6.7 | 15 | 100.0 | 0 | 0 | 3 |

**RDX RPD ACCURACY**

| | <30 mg/Kg | | | | 30-100 mg/Kg | | | | 100-1000 mg/Kg | | | | >1000 mg/Kg | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Bias | | | | Bias | | | | Bias | | | | Bias | | |
| | RPD[1] | + | - | n | RPD1 | + | - | n | RPD[1] | + | - | n | RPD[1] | + | - | n |
| DTECH estimate | 28.8 | 3.4 | 67.8 | 59 | 0 | 0 | 100 | 35 | 4.0 | 0 | 96.0 | 25 | 0 | 0 | 100.0 | 3 |
| DTECH range[2] | 34.5 | 1.7 | 63.8 | 59 | 0 | 2.6 | 97.4 | 37 | 4.0 | 0 | 96.0 | 25 | 0 | 0 | 100.0 | 4 |
| Ensys | 59.1 | 15.5 | 25.4 | 71 | 48.8 | 2.4 | 48.8 | 43 | 25.0 | 6.3 | 68.7 | 32 | 66.7 | 0 | 33.3 | 3 |
| Jenkins | 34.1 | 13.6 | 52.3 | 44 | 20.0 | 0 | 80.0 | 20 | 0 | 0 | 100.0 | 2 | No Data | | | |

Bias + = bias higher than DataChem Concentration.
Bias - = bias lower than DataChem Concentration.
n = Total number of samples being compared.
[1] = Percent of data set that met RPD accuracy criteria (±50%).
[2] = Percent of DataChem results that fit corresponding DTECH range results.

# Table 4
## Summary of Field Screening Comparison

| Criteria | DTECH | | Quantix | Ensys | | Jenkins | |
|---|---|---|---|---|---|---|---|
| Available Analyses | TNT and RDX in soil and water | | TNT in soil and water | TNT in soil; RDX under development | | TNT and RDX in soil and water | |
| Detection Range (Not diluted) | TNT soil = 0.5-5.0 mg/Kg<br>RDX soil = 0.5-6.0 mg/Kg<br>TNT & RDX water = 5-45 ppb | | TNT soil =0.26-100 mg/Kg<br>TNT water > 0.05 ppb | TNT soil = 1-30 mg/Kg | | TNT soil = 1.11-22.3 mg/Kg<br>TNT water = 0.91 ppb<br>RDX soil = 1.0-20 mg/Kg<br>RDX water = 3.83 ppb | |
| Type of Results | Concentration Range | | Quantitative | Quantitative | | Quantitative | |
| Samples per batch | 8 (can run single or batch) | | 20-40 (batch only) | 1 (extraction can be batched) | | TNT: 1 (single only) : RDX: 6-7 (can run single or batch; extractions can be batched) | |
| Analysis Time | 90 to 120 minutes for 8 TNT and RDX samples | | 2½ to 3½ hours for 20 to 40 TNT samples | 30 to 35 minutes for 10 TNT samples in lab; estimated 45 minutes in field | | 30 minutes to extract 6 samples; 5 minutes per TNT sample; 30 minutes for 6 RDX samples | |
| Sample Size | Approximately 4.5 grams | | Approximately 4.2 grams | 10 grams | | 20 grams | |
| Skill Level | Low | | High: training recommended | Unknown | | Medium | |
| Cost | $60 per sample for RDX and TNT plus $300 for DTECHTOR | | $21 per sample for TNT plus $5880 for lab station | $25 per sample for TNT plus $160 per day/$400 per week rental or $2,000 purchase for lab station | | $15 per sample for RDX and TNT plus $1600 for Hach spectrometer purchase. Rental cost similar to Ensys. | |
| Accuracy | TNT[1] | RDX[1] | TNT | TNT | RDX | TNT | RDX |
| < 30 mg/Kg | 47.7/54.5 | 28.8/34.5 | 23.4 | 20.8 | 59.1 | 31.5 | 34.1 |
| 30-100 mg/Kg | 50.0/26.7 | 0/0 | 58.8 | 82.3 | 48.8 | 92.3 | 20.0 |
| 100-1000 mg/Kg | 47.1/35.3 | 4.0/4.0 | 54.6 | 66.7 | 25.0 | 80.0 | 0 |
| > 1000 mg/Kg | 40.0/20.0 | 0/0 | 40.0 | 80.0 | 66.7 | 100.0 | No data |
| Precision<br>R = Replicate<br>D = Duplicate | TNT R-80%<br>D-100% | RDX R-100%<br>D-100% | R - 100%<br>D - 88% | TNT R - 100%<br>D - 100% | RDX R-100%<br>D-100% | No data | |
| Additional considerations | Small working area; little set up requirements; no electricity or refrigeration required; temperature dependant development time; significant amount of packaging; dilutions increases ranges; no check on test; easy to transport/carry. | | Larger working area(desk); set up time required; need electricity, refrigeration and deionized water; replicate run for each sample, average is the result; less temperature dependant. | Less working space than Quantix, but more than DTECH; power supply required to charge Hach spectrometer; possible TNB interference; color indication of other compounds in sample. | | Large working area (2 large desks); requires the most set up time; difficult to transport; possible TNB interference; deionized water required; must assemble materials; glassware must be rinsed between analysis; acetone must be properly disposed; color indication of other compounds. | |

[1] DTECH estimate/DTECH range