Exponent™

Results of the 1998 Sediment and Surface Water Investigation at the Former Barksdale Works, Ashland, Wisconsin

Prepared for

E.I. du Pont de Nemours and Company
Building 27
Lancaster Pike & Route 141
Wilmington, Delaware 19805

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ACRONYMS AND ABBREVIATIONS

2,4-DNT	2,4-dinitrotoluene
2,6-DNT	2,6-dinitrotoluene
DNT	dinitrotoluene
EPA	U.S. Environmental Protection Agency
GPS	geographic positioning system
NG	nitroglycerine
NOEC	no-observed-effect concentration
QAPP	quality assurance project plan
SAP	sampling and analysis plan
TNT	trinitrotoluene
TSS	total suspended solids
WDNR	Wisconsin Department of Natural Resources

1. INTRODUCTION

E.I. du Pont de Nemours and Company has investigated the presence of explosive compounds in surface water and sediment of the former Barksdale trinitrotoluene (TNT) manufacturing facility in Ashland, Wisconsin (Figure 1). The objective of this investigation is to respond to concerns expressed by the Wisconsin Department of Natural Resources (WDNR) (Saari 1998). The specific goal is to determine if explosive compounds are being exported from the site to Chequamegon Bay at concentrations of ecological concern. The results of the investigation indicate that **explosive compounds are not being exported** from the site via surface water or sediment at concentrations of ecological concern. Furthermore, all measured concentrations within the site are **below ecological effect levels**.

The Barksdale Explosive Works operated from 1904 to 1971, with peak periods of production during the two World Wars. The principal manufactured product was TNT. Lesser amounts of 2,4-dinitrotoluene (2,4-DNT), 2,6-dinitrotoluene (2,6-DNT), and nitroglycerine (NG) were also produced either directly or as a byproduct of TNT manufacture. Boyd Creek, which flows from east to west across the site and discharges into Chequamegon Bay (Figure 1), traverses some of the production areas and received discharges from manufacturing operations. After manufacturing ceased in 1971, cleanup activities were conducted through 1984. Cleanup included removal of buildings and stored materials, and removal or incineration of contaminated soil. Bretting Manufacturing currently owns the site and uses it for warehousing, pasturage, and as a game reserve. This investigation of sediment and surface water was initiated following detection of low levels of dinitrotoluene (DNT) in groundwater at the eastern edge of the site. Groundwater is being evaluated and reported separately from this investigation. Additional background information on site history and geology can be found in the site conditions report (DuPont 1997).

The approach to this site investigation encompassed three main activities:

- A site visit to evaluate overall ecological conditions and to gather information for development of a sampling plan
- A literature search to determine concentrations of explosive compounds in surface water and sediment that are of potential concern with regard to ecological effects
- Sampling of surface water and sediment, and interpretation with respect to export of explosive compounds from the site.

These activities were carried out in the order listed. Thus, ecological effects levels were evaluated prior to the collection or analysis of any samples.

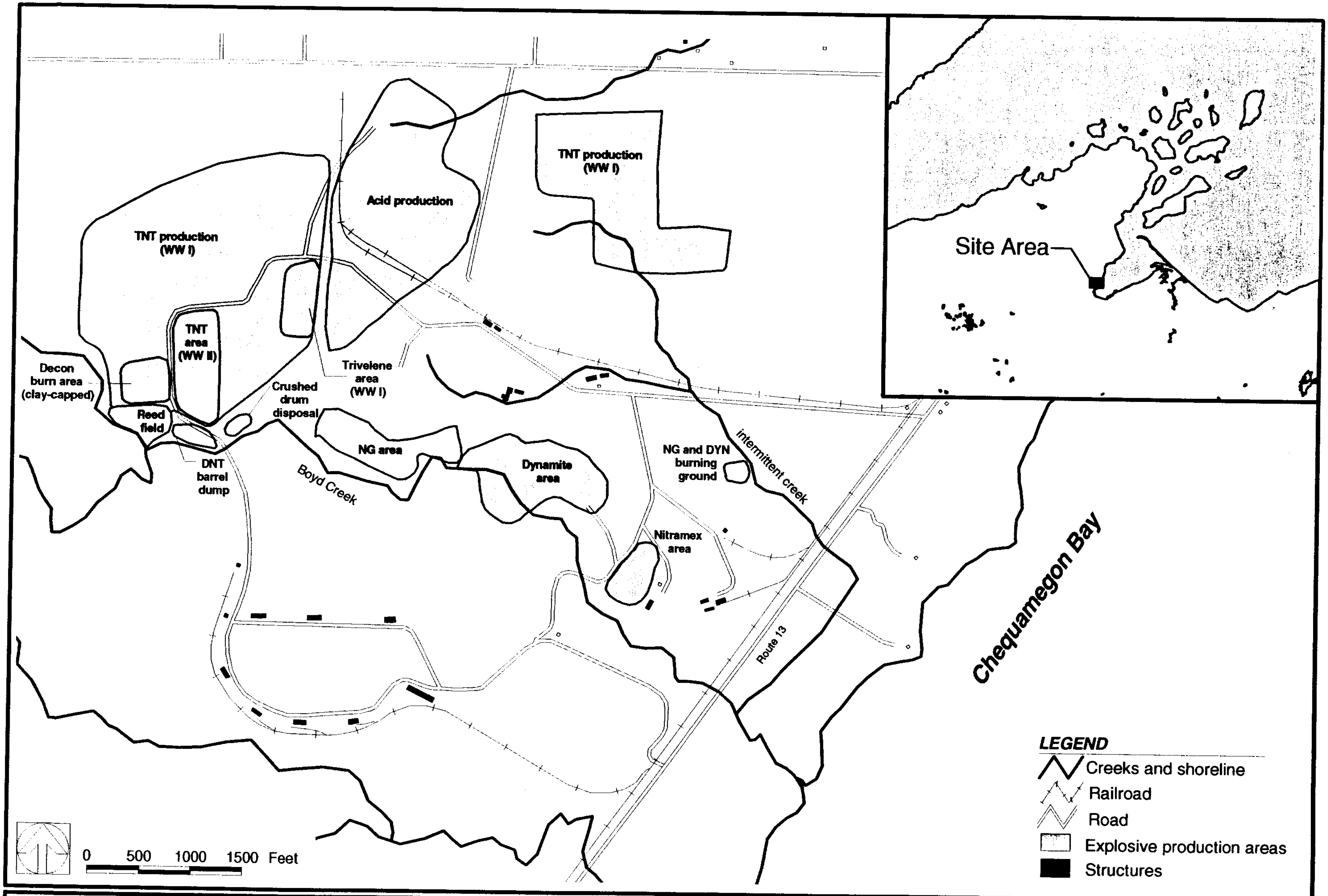

Figure 1. Explosive production areas.

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Results of the site visit and the literature search, as well as tentative sampling locations and rationale, were presented to WDNR during a meeting on May 28, 1998, in Ashland, Wisconsin. This information is also summarized in this report in Sections 2 and 3. Section 4 of this report presents the results of the surface water and sediment sampling, and Section 5 includes an evaluation of those results with regard to the investigation's goal.

2. SITE VISIT AND SAMPLING PLAN

A visual survey of Boyd Creek was conducted in April 1998 to characterize overall environmental conditions and identify appropriate sampling locations. During this site visit, Boyd Creek was examined from near its mouth to (and including) the small tributary that bounds the TNT production areas at the upstream end (the "ravine" area). General environmental conditions in and adjacent to the stream were noted, consistent with U.S. Environmental Protection Agency (EPA) Rapid Bioassessment Protocols (U.S. EPA 1998). This survey included observations of vegetative cover over and adjacent to the creek, creek bottom material, water flow conditions, and presence and type of point and nonpoint inflows.

Overall, Boyd Creek appears to offer good physical habitat for fish. Observations supporting this conclusion include the following:

- Trees and other vegetation shade the creek for almost the entire length of the creek, limiting summertime temperature elevations. Only in the ravine area is vegetative cover completely absent. Overhanging banks also shade the water in many locations.
- Both banks of the creek are vegetated for a distance of 20 m or more from the creek throughout its length (excepting road crossings; the only public road crossing is Route 13, and on-site roads are narrow and rarely carry any traffic).
- A variety of bottom types are found in the creek, ranging from poorly sorted mixtures of clay, sand, and gravel to cobble and exposed sandstone.
- The flow regime includes both fast and slow water, with riffles and pools.
- There are no evident ditches entering the creek or any runoff from bare ground in the former manufacturing area. In several locations there are swampy areas or small impoundments (e.g., beaver ponds) adjacent to the creek that may serve as recharge reservoirs. Heavy rain was falling at the time of the site visit, and had been falling for the previous 12 to 18 hours, but there was no sign of sheet flow into the creek from these or other locations upstream of Route 13. The only place at which sheet flow was observed entering Boyd Creek was at the old railroad grade just north of Route 13; this is now evidently used as a snowmobile track in the winter, and erosion is occurring on bare ground adjacent to the creek.

- Boyd Creek appears to have less adjacent pasturage than either Bono Creek (immediately to the north) or Whittlesey Creek (to the south).

These observations indicate that conditions are appropriate to support the presence of fish. Although Boyd Creek is not classified as a Wisconsin trout stream (WDNR 1980), trout have been observed in the creek in recent years (Peterson 1998, pers. comm.).

Observations made during the site visit were used to develop the sampling plan. This plan provided for separate characterization of three areas of the site:

- The main stem of Boyd Creek
- The tributary in the ravine area at the head of Boyd Creek
- The intermittent stream from the World War I production area that has been diverted to join Boyd Creek downstream of Route 13 (i.e., near the mouth).

Physical conditions in Boyd Creek and on the banks are uniform from the mouth upstream to the ravine area. Physical conditions in the ravine area are also uniform, but different from those in the main stem (the principal differences being an absence of overhanging vegetation, lower flow, and more submerged vegetation). This difference was accommodated in the sampling plan by adopting a stratified sampling strategy, with sampling locations placed uniformly throughout the length of both Boyd Creek and the ravine tributary. Because the intermittent tributary drains a different portion of the site than the main stem of Boyd Creek, the tributary was sampled separately: one sampling location was established in the tributary downstream of its culvert under Route 13. One sampling location in Boyd Creek was also established just downstream of the entry of this tributary. Because spatial variation is expected to be greater for sediment than for surface water, sediment samples were taken at more locations than were surface water samples. All sampling locations are shown in Figure 2. Sampling locations were placed throughout Boyd Creek and the ravine to allow spatial trends and the existence of possible source areas to be evaluated.

Because all explosive manufacturing activities occurred upstream (west) of Route 13, sampling locations were established at and below Route 13 to characterize surface water and sediment likely to be exported from the site. Sampling location (station) B1 is located upstream not only of Route 13 but also of the old railroad grade, which was the only observed location of surface runoff. Station B1 is therefore considered to be the primary indicator of off-site transport, unaffected by possible interference from runoff at the railroad grade or Route 13. Station B0, although located downstream of Route 13, is placed to reveal any impact on Boyd Creek sediments from the intermittent stream. Triplicate water samples were collected at station B1 during the same period that water samples were collected at upstream locations (B4 and B7) so that spatial and temporal variability could be contrasted.

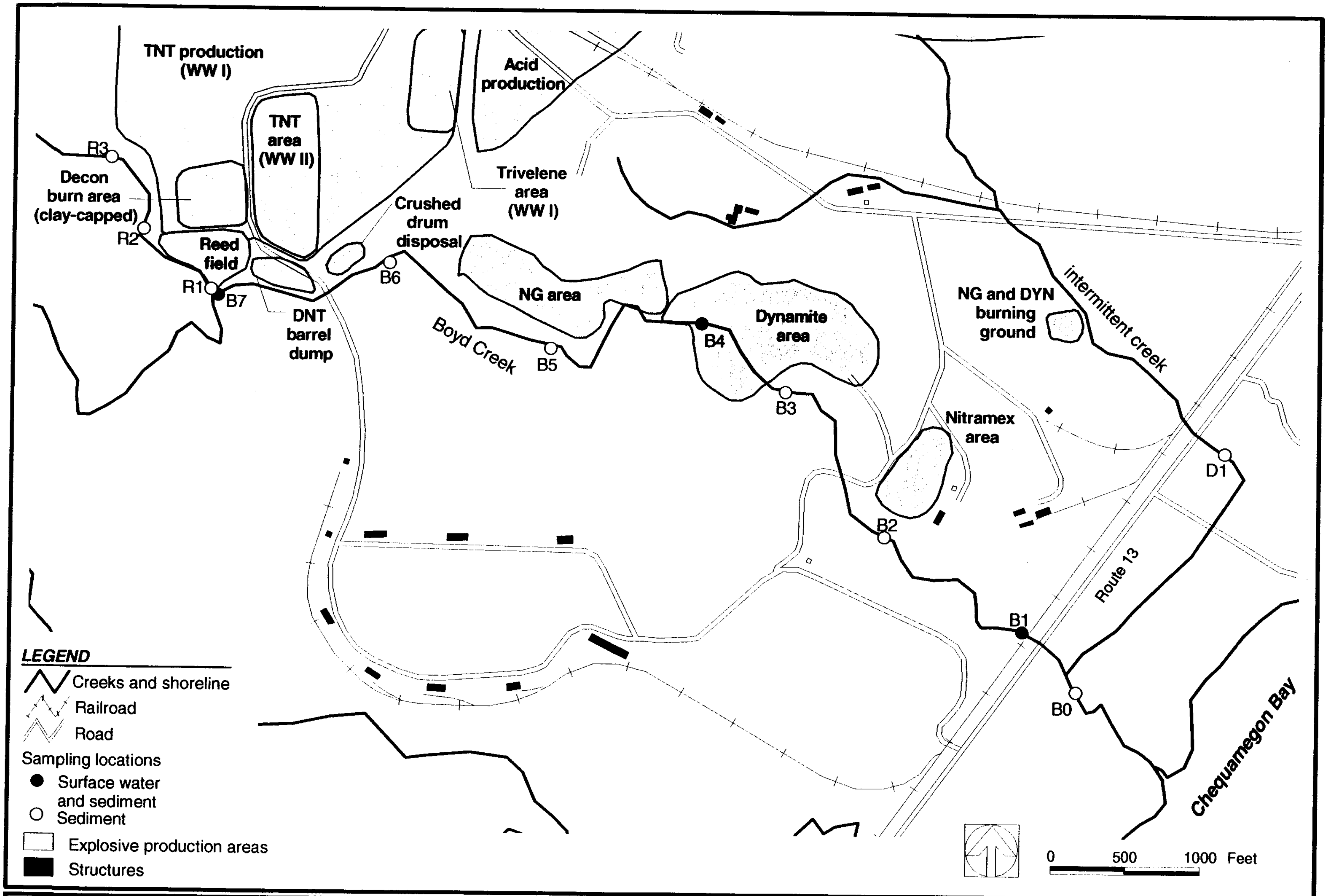

Figure 2. Sediment and surface water sample locations.

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Sampling locations shown in Figure 2 are as measured during the actual sampling effort using a differentially corrected geographic positioning system (GPS). The slight discrepancies between the sampling locations and the stream course shown in Figure 2 may be a consequence of inaccuracies in both the GPS measurements or movement of the stream location since it was originally plotted by the U.S. Geological Survey.

Only surface sediment (to a depth of 10 centimeters) was sampled and analyzed. The high proportion of gravel and cobble in the streambed prohibited coring, and characterization of fine surface sediment is most appropriate for estimating ecological exposures to sediment contaminants because concentrations generally increase with decreasing grain size. At the time of sampling (August 1998), no water was flowing in the intermittent stream, and the sediment sample was taken from a standing pool in the streambed.

Water and sediment samples were analyzed for TNT, 2,4-DNT, 2,6-DNT, and NG. Sediment samples were also analyzed for total organic carbon and grain size. Both of these sediment characteristics may affect the sorption, and therefore the concentration, of explosive compounds. Water samples were analyzed for suspended solids and various ionic constituents (e.g., nitrate) that may be associated with breakdown of explosive compounds.

Further details regarding sampling and analysis methods and procedures are documented in the sampling and analysis plan and quality assurance project plan (SAP/QAPP; Exponent 1998).

3. ECOLOGICAL EFFECTS LEVELS

Although Wisconsin has established water quality criteria for 2,4-DNT (NR 105.08, Wis. Adm. Code), neither the state nor EPA has established water or sediment quality criteria for TNT, 2,6-DNT, or NG. Relevant ecological screening values for TNT and DNT isomers were therefore sought by:

- Conducting a literature search for available peer-reviewed and gray literature
- Interviewing investigators in the field who may have unpublished data
- Computing sediment criteria based on water quality criteria and available information on chemical partitioning.

The literature reviewed is listed in Appendix A.

Ecological screening values for TNT and DNT in surface water and sediment are listed in Tables 1 and 2. Potential screening values for sediment were derived in several ways: direct measurements of sediment toxicity to benthic organisms, measurements of soil or sediment toxicity to aquatic organisms, field observations of correlated explosive concentrations and biological effects, and calculations using chemical partitioning information and water quality or effects data. Direct measurements of sediment toxicity are considered most reliable; the reliability of other effects measures decreases in the order listed. The sediment effects levels listed in Table 2 are categorized accordingly. Estimated effects levels are available for both TNT and 2,4-DNT in water and sediment; in the absence of any specific values for 2,6-DNT, 2,6-DNT will, for the purpose of data interpretation, be considered to be equivalent to 2,4-DNT in toxicity.

TABLE 1. ECOLOGICAL SCREENING VALUES FOR SURFACE WATER

Compound	Criterion	Value (μg/L)	Source
2,4-DNT	Cold Water Communities	5.3	NR 105.08 Wis. Adm. Code
2,4-DNT	Limited Aquatic Life	110	NR 105.08 Wis. Adm. Code
TNT	Estimated Maximum Criterion[a]	557	ORNL (1987)
TNT	Estimated Continuous Criterion[a]	40	ORNL (1987)

[a] Calculated per EPA guidelines.

TABLE 2. ECOLOGICAL SCREENING VALUES FOR SEDIMENT

Compound	Value (mg/kg dry)	Type	Source
TNT	84	A	Lowest no-observed-effect concentration (NOEC) for several endpoints for *Leptocheirus plumulosus* (Green et al. 1998)
TNT	55	A	Lowest NOEC for several endpoints for *Neanthes arenaceodentata* (Green et al. 1998)
TNT	304	B	EC_{50} for effects of Umatilla lagoon sediment on aquatic organisms (Griest et al. 1993)
2,4-DNT	19	C	Calculated from 2,4-DNT sorption to illite (Haderlein et al. 1996; Johnson 1980) and WI water quality criteria, Cold Water Communities, for 2,4-DNT (NR 105.08 Wis. Adm. Code)
2,4-DNT	400	C	Calculated from 2,4-DNT sorption to illite (Haderlein et al. 1996; Johnson 1980) and WI water quality criteria, Limited Aquatic Life, for 2,4-DNT (NR 105.08 Wis. Adm. Code)

Note: A - sediment toxicity to benthic organisms
B - soil or sediment toxicity to aquatic organisms
C - partitioning information and water quality criteria or effects data

4. ANALYTICAL RESULTS AND INTERPRETATION

Surface water and sediment samples were collected from the Barksdale site on August 17, 18, and 19, 1998. Exponent collected the samples, Quanterra carried out laboratory analyses, and Environmental Standards conducted data validation. Sampling and analysis were performed in accordance with the SAP and QAPP (Exponent 1998). Results of the quality assurance review are documented separately (Environmental Standards 1998). No problems were encountered during sampling or analysis.

Analytical results for explosives in surface water and sediment are shown in Tables 3 and 4, below. All analytical results are presented in Appendix B.

TABLE 3. MEASURED EXPLOSIVE COMPOUNDS IN SURFACE WATER

Station	Field replicate	2,4,6-TNT (μg/L unfiltered)	2,4-DNT (μg/L unfiltered)	2,6-DNT (μg/L unfiltered)	NG (μg/L unfiltered)
B1	1	0.018 *U*	0.16 *J*	0.39	0.088 *U*
B1	2	0.018 *U*	0.20 *J*	0.28	0.088 *U*
B1	3	0.018 *U*	0.17 *J*	0.22 *J*	0.088 *U*
B4		0.018 *U*	0.19 *J*	1.1	0.088 *U*
B7		0.018 *U*	0.025 *U*	0.016	0.088 *U*

Note: *J* - estimated
U - undetected at detection limit shown

TABLE 4. MEASURED EXPLOSIVE COMPOUNDS IN SEDIMENT

Station	Field replicate	2,4,6-TNT (mg/kg dry)	2,4-DNT (mg/kg dry)	2,6-DNT (mg/kg dry)	NG (mg/kg dry)
B0		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B1	1	0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B1	2	0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B2		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B3		0.034 *J*	0.15 *J*	0.033 *J*	0.092 *U*
B4		0.24 *J*	0.11 *J*	0.033 *J*	0.092 *U*
B5		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B6		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
B7		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
D1		0.13 *J*	0.45	0.082 *J*	0.092 *U*
R1		0.005 *U*	0.018 *U*	0.012 *U*	0.092 *U*
R2		12	0.29	0.20 *J*	0.092 *U*
R3		1.4	0.47	0.27	0.092 *U*

Note: *J* - estimated
U - undetected at detection limit shown

Nitroglycerin was not detected in any surface water or sediment sample.

TNT was not detected in any surface water sample. DNT isomer concentrations were lowest or undetected in surface water at the sampling station farthest upstream (B7), but were detected at concentrations well above the detection limit at the sampling stations in the middle and at the downstream end of Boyd Creek (B4 and B1, respectively). The locations where DNT was detected in surface water are shown in Figure 3. Replicate samples taken over a period of several hours at station B1 were consistent, indicating that the variation seen along the length of Boyd Creek is not a result of variability introduced by field and laboratory procedures. Total suspended solids (TSS) were less than 5 mg/L at stations B1 and B4, whereas station B7 had TSS of 21 mg/L. The increased DNT concentrations at downstream stations are therefore not due to an increased load of sediments containing sorbed DNT.

TNT and DNT were detected in sediment samples in three general locations: in the central part of the main stem of Boyd Creek (stations B3 and B4), in the upper end of the ravine (stations R2 and R3), and in the intermittent stream just downstream of Route 13. These locations are shown in Figure 4. Detected concentrations were much higher than detection limits, indicating that the results are not the result of analytical variability in the region of the detection limit.

Stations B3 and B4 are just below and within (respectively) a former TNT production area. Appearance of detected concentrations of explosive compounds in both surface water and sediment at station B4 suggests that an ongoing source is present in this area. Several water quality constituents—bicarbonate alkalinity, chloride, nitrate, sodium, and sulfate—are also highest at station B4.

Detected concentrations of TNT and DNT in the two upstream ravine stations indicate that residual explosive material remains in the sediment at these locations. An ongoing source to surface water does not appear to exist in this area because TNT and DNT were undetected in surface water immediately downstream of the ravine area (at station B7); in contrast, DNT was detected in the water much farther downstream of the apparent source at station B4.

Mobility of sediment-associated TNT and DNT appears to be limited to nonexistent. Concentrations fall by approximately a factor of 10 at stations immediately below the locations where these explosive compounds are detected. The detected concentrations in the intermittent stream are also not seen in Boyd Creek at station B0, immediately below the inflow of the intermittent stream. The distinct limits to the spatial extent of sediment-associated TNT and DNT suggest that if any transport is occurring, degradation or other processes are acting to rapidly reduce concentrations relative to the rate of transport.

All detected concentrations of TNT and DNT in surface water and sediment are one-tenth or less of the concentrations at which ecological effects are expected (Tables 1 and 2).

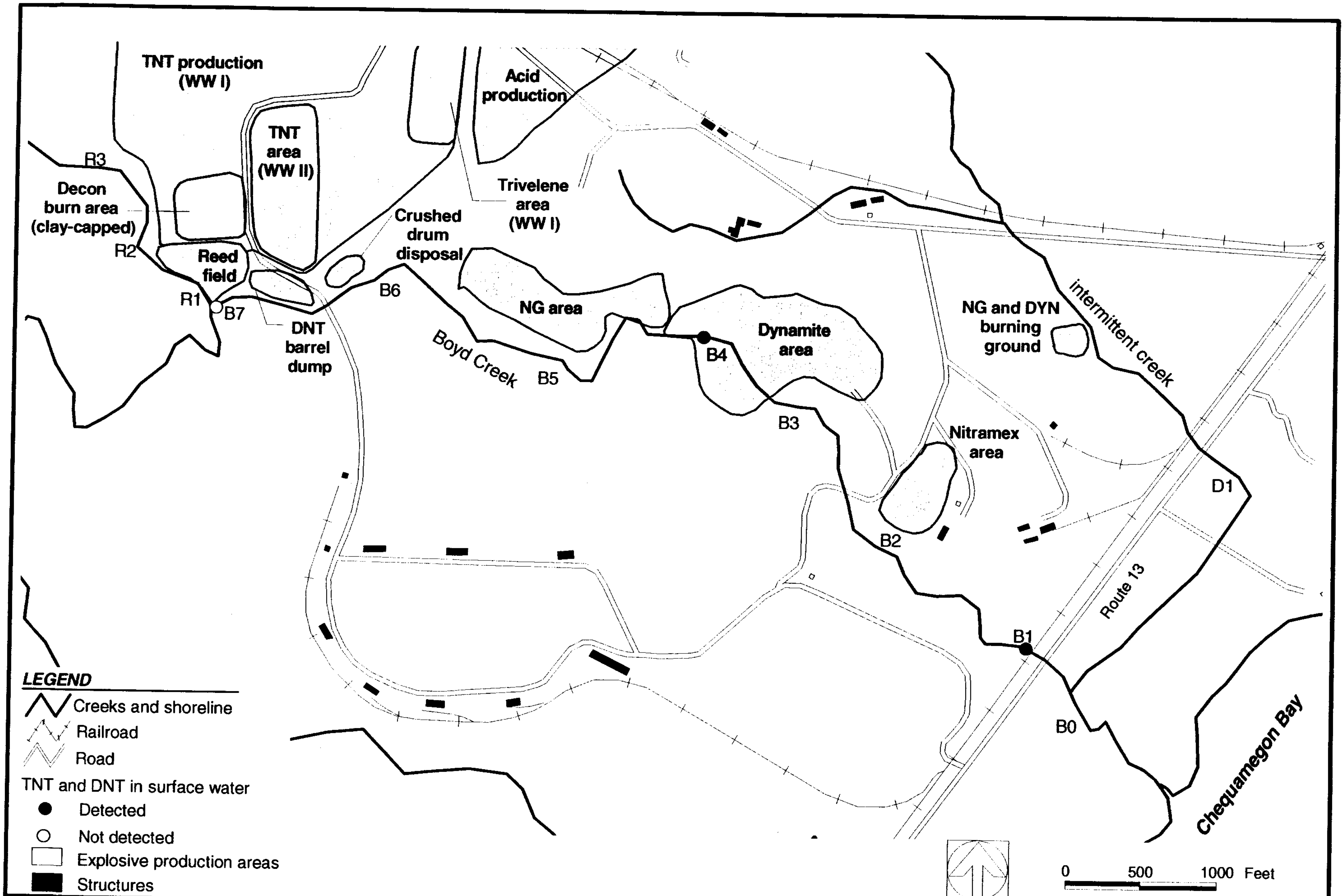

Figure 3. Locations of detected TNT and DNT in surface water samples.

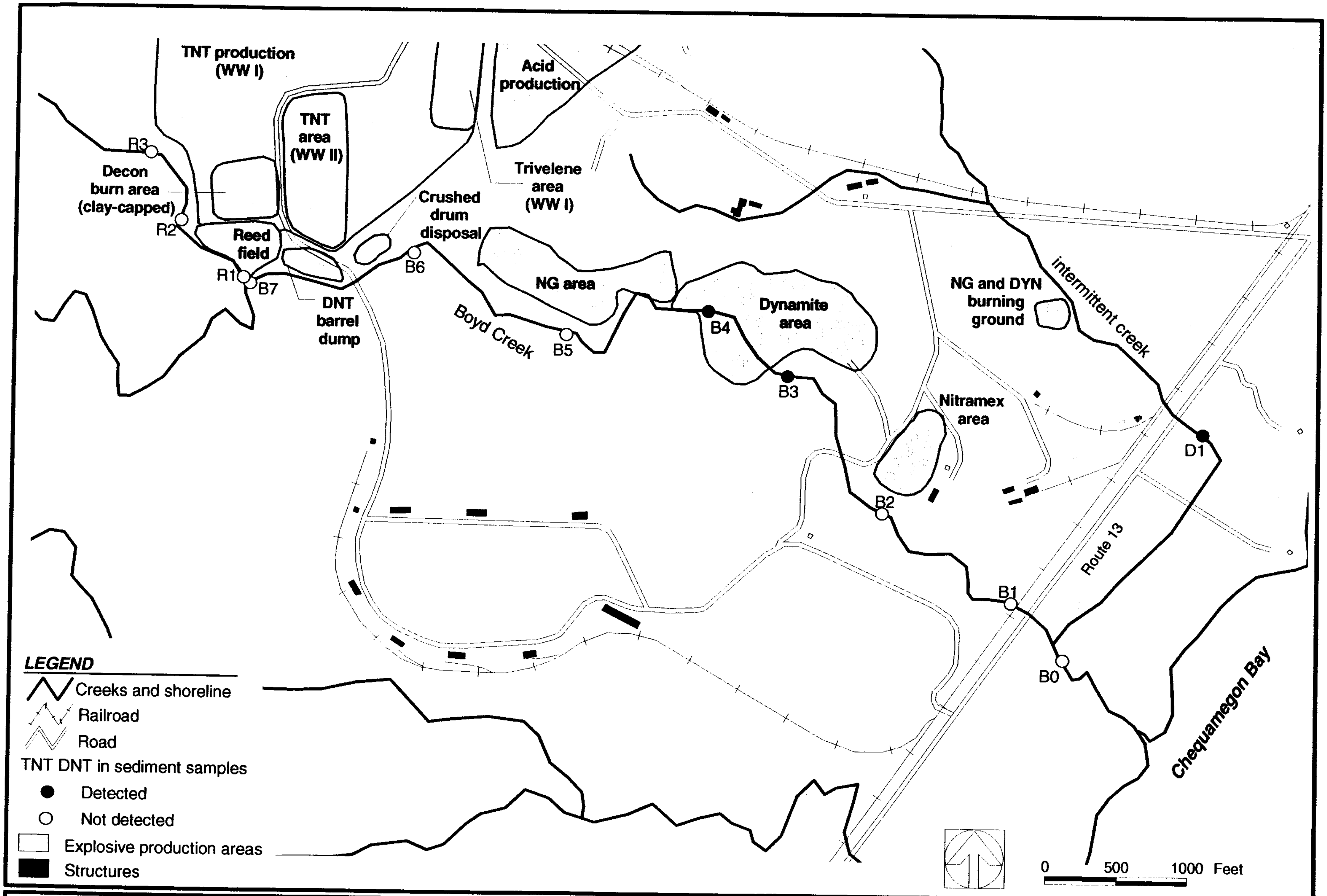

Figure 4. Locations of detected TNT and DNT in sediment samples.

Thus, explosive compounds do not appear to be either exported from the Barksdale site, nor present within the Barksdale site, at concentrations of concern.

5. CONCLUSIONS

The specific goal of this investigation is to determine if explosive compounds are being exported from the site to Chequamegon Bay at concentrations of potential ecological concern. The sampling and analyses that were carried out resulted in the collection of data that directly address that goal, and the results clearly show that concentrations of TNT and DNT isomers are not being exported from the site at concentrations of ecological concern. Furthermore, no concentrations of TNT or DNT were observed anywhere on the site that approach levels of ecological concern.

Data collected during this investigation allow several other conclusions to be drawn regarding site features or characteristics. For the sake of completeness, all conclusions of this investigation are listed below.

- Physical characteristics of Boyd Creek appear adequate to support fish such as trout.
- TNT and DNT are not being exported from the Barksdale site to Chequamegon Bay at concentrations of concern for ecological receptors.
- TNT and DNT are not present within the Barksdale site at concentrations of concern for ecological receptors.
- The former TNT production area in the central portion of Boyd Creek may be a continuing source of low concentrations of TNT and DNT to surface water and sediment.
- Sediment-associated TNT and DNT is extremely limited in spatial extent, possibly as a result of either very low mobility or degradation rates that exceed the transport rate.

The data produced by this investigation addressing potential ecological effects are considered sufficiently definitive that no further investigation of this issue is warranted.

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Appendix A

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Appendix B

Analytical Results

TABLE B-1. ANALYTICAL RESULTS FOR WATER SAMPLES

Analyte	Units	Measurement Basis	Station: / Date: / Field rep:	B1 8/18/98 1	B1 8/18/98 2	B1 8/18/98 3	B4 8/18/98	B7 8/18/98
Explosives								
2,4-Dinitrotoluene	μg/L	unfiltered		0.16 *J*	0.2 *J*	0.17 *J*	0.19 *J*	0.025 *U*
2,6-Dinitrotoluene	μg/L	unfiltered		0.39	0.28	0.22 *J*	1.1	0.016
2,4,6-Trinitrotoluene	μg/L	unfiltered		0.018 *U*	0.018 *U*	0.018 *U*	0.018 *U*	0.018 *U*
Nitroglycerin	μg/L	unfiltered		0.088 *U*	0.088 *U*	0.088 *U*	0.088 *U*	0.088 *U*
Water Quality								
Calcium	mg/L	unfiltered		50.1	48.3	47.8	73.8	31.8
Magnesium	mg/L	unfiltered		30.1	28.6	27.6	57.5	9.8
Potassium	mg/L	unfiltered		0.6 *U*	0.6 *U*	0.6 *U*	0.6 *U*	0.6 *U*
Bicarbonate alkalinity	mg/L	unfiltered		197	191	192	291	124
Bromide	mg/L	unfiltered		0.05 *U*	0.05 *U*	0.05 *U*	0.05 *U*	0.05 *U*
Carbonate alkalinity	mg/L	unfiltered		5 *U*	5 *U*	5 *U*	5 *U*	5 *U*
Fluoride	mg/L	unfiltered		0.068 *U*	0.068 *U*	0.068 *U*	0.068 *U*	0.068 *U*
Sodium	mg/L	unfiltered		11.1	10.7	10.5	18.6	0.036 *U*
Sulfate	mg/L	unfiltered		66.8	61.8	60.5	149	2.1
Total chloride	mg/L	unfiltered		4.7	4.6	4.7	8.9	1.3
Nitrate nitrogen	mg/L	unfiltered		0.64 *J*	0.59 *J*	0.47 *J*	2.7 *J*	0.036 *U*
Nitrite nitrogen	mg/L	unfiltered		0.022 *U*	0.022 *U*	0.022 *U*	0.022 *U*	0.022 *U*
Total suspended solids	mg/L	unfiltered		5 *U*	5 *U*	5 *U*	5	21

Note: *J* - estimated
U - undetected at detection limit shown

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TABLE B-2. ANALYTICAL RESULTS FOR SEDIMENT SAMPLES IN BOYD CREEK

Analyte	Units	Meas. basis	Station: / Date: / Field Rep:	B0 8/17/98	B1 8/17/98 1	B1 8/18/98 2	B2 8/18/98	B3 8/18/98	B4 8/18/98	B5 8/18/98	B6 8/18/98	B7 8/18/98
Explosives												
2,4-Dinitrotoluene	mg/kg	dry		0.018 *U*	0.018 *U*	0.018 *U*	0.018 *U*	0.15 *J*	0.11 *J*	0.018 *U*	0.018 *U*	0.018 *U*
2,6-Dinitrotoluene	mg/kg	dry		0.012 *U*	0.012 *U*	0.012 *U*	0.012 *U*	0.033 *J*	0.033 *J*	0.012 *U*	0.012 *U*	0.012 *U*
Trinitrotoluene (2,4,6-Trinitrotoluene)	mg/kg	dry		0.005 *U*	0.005 *U*	0.005 *U*	0.005 *U*	0.034 *J*	0.24 *J*	0.005 *U*	0.005 *U*	0.005 *U*
Nitroglycerin	mg/kg	dry		0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*
Physical properties												
Total organic carbon	mg/kg	dry		443	501	295	646	290	682	789	714	1020
Percent moisture	percent	wet		19.7	18.6	19.6	17.9	15.4	18.1	15.9	13.5	18.8
Percent clay	percent	wet		1.9	0.9	1		0.8	0.9		1.2	2.7
Percent fines (clay & silt)	percent	wet		2.2	12.2	9.8	3.6	1.4	2.8	2	3.2	8.7
Percent gravel	percent	wet		0	2	1.9	0.9	7.1	0.3	8.6	13.3	0
Percent sand	percent	wet		97.8	85.8	88.3	95.5	91.5	96.9	89.4	83.5	91.3
Percent silt	percent	wet		0.3	11.3	8.8		0.6	1.9		2	6

Note: *J* - estimated
U - undetected at detection limit shown

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TABLE B-3. ANALYTICAL RESULTS FOR SEDIMENT SAMPLES IN THE RAVINE AND INTERMITTENT STREAM

Analyte	Units	Meas. basis	Station: Date:	D1 8/19/98	R1 8/18/98	R2 8/19/98	R3 8/19/98
Explosives							
2,4-Dinitrotoluene	mg/kg	dry		0.45	0.018 *U*	0.29	0.47
2,6-Dinitrotoluene	mg/kg	dry		0.082 *J*	0.012 *U*	0.2 *J*	0.27
Trinitrotoluene (2,4,6-Trinitrotoluene)	mg/kg	dry		0.13 *J*	0.005 *U*	12	1.4
Nitroglycerin	mg/kg	dry		0.092 *U*	0.092 *U*	0.092 *U*	0.092 *U*
Physical properties							
Total organic carbon	mg/kg	dry		9,020	14,900 *J*	10,400	9,880
Percent moisture	percent	wet		30.3	33.7	30.2	33.6
Percent clay	percent	wet		4.4	14.1	27	42.4
Percent fines (clay & silt)	percent	wet		13.5	30.3	45.8	65
Percent gravel	percent	wet		17.6	4.1	4.3	0.1
Percent sand	percent	wet		68.9	65.6	49.9	34.9
Percent silt	percent	wet		9.1	16.2	18.8	22.6

Note: *J* - estimated
U - undetected at detection limit shown