

# **SAND REPORT**

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## **GICHD Mine Dog Testing Project – Soil Sample Results #5**

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## GICHD Mine Dog Testing Project –Soil Sample Results #5

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### **Abstract**

A mine dog evaluation project initiated by the Geneva International Center for Humanitarian Demining is evaluating the capability and reliability of mine detection dogs. The performance of field-operational mine detection dogs will be measured in test minefields in Afghanistan containing actual, but unfused landmines. Repeated performance testing over two years through various seasonal weather conditions will provide data simulating near real world conditions. Soil samples will be obtained adjacent to the buried targets repeatedly over the course of the test. Chemical analysis results from these soil samples will be used to evaluate correlations between mine dog detection performance and seasonal weather conditions. This report documents the analytical chemical methods and results from the fifth batch of soils received. This batch contained samples from Kharga, Afghanistan collected in June 2003.

## **Acknowledgements**

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## 1.0 Introduction

The Geneva International Center for Humanitarian Demining (GICHD) initiated a project to evaluate the capability and reliability of mine detection dogs to find landmines in test minefields. Details describing the background, scope, objectives and project execution can be found in the Terms of Reference (GICHD, 2000). The project seeks to evaluate weather data, surface soil sample chemical residue results, and mine dog performance to determine conditions optimal for successful landmine detection. Quarterly samples are planned for collection in Afghanistan (Kharga). Periodic reports will be produced documenting the results of samples submitted to Sandia National Laboratories. The first set of soil samples received by Sandia National Laboratories were obtained from Afghanistan prior to placement of the test landmines to determine if the sites contained explosive signature chemical residues that might confound future tests (Phelan and Barnett, 2001). Analytical results from samples taken in April/May 2003, October 2002, March 2002 and July 2001 have been previously documented (references 5-8).

The purpose of this report is to document the surface soil chemical residue results from sample set #5 received at Sandia National Laboratories on September 10, 2003. The Swedish Defense Research Establishment (FOI) and the Geneva International Center for Humanitarian Demining (GICHD) collected these samples in Afghanistan June 2003.

Samples were packaged in an insulated cardboard box with plastic ice packs. Upon receipt, the temperature of the samples was measured by thermocouples and was found to be about 10°C. The samples were placed into a freezer at -10°C until sample preparation began on September 16, 2003. A total of 151 samples were received from Kharga, Afghanistan. The first fifty-two samples (Kharga A7) analyzed were not marked with a series number or sampling date. The assumption was made that sampling location and sampling date was the same for all samples received. Sample analysis procedures were completed by January 7, 2004.

Sample preparation, extraction and quantification were performed using protocols developed for the US Environmental Protection Agency (EPA, 1998). Details on this method are described in Section 2. The analytical results are presented and discussed in Section 3.

## 2.0 Sample Analysis Method

Chemical residues of explosive related compounds in soils were analyzed using EPA Method 8095. The soil samples were received in 40 mL amber screw cap vials. The samples were mixed by vigorously shaking each vial. Approximately 0.8 g ( $\pm$  0.01 g) of soil was removed from each 40 mL vial and placed into a 5 mL amber screw cap vial with care to avoid stones and organic material. A surrogate (25  $\mu$ L aliquot of 10 mg/L of 3,4-dinitrotoluene) was placed into each extraction vial as a quality control

check on extraction efficiency. Acetonitrile ( $4 \text{ mL} \pm 1 \mu\text{L}$ ) was added by pipetting to create a 4:1 solvent to soil ratio. A batch containing 20 samples was placed into a  $10^\circ\text{C}$  water bath cooled ultrasonicator for 18 hours. The samples were then syringe filtered using  $0.45\text{-}\mu\text{m}$  nylon filters and placed into an autosampler vial. The filtered soil extracts were analyzed by gas chromatography (GC) with a  $1\text{-}\mu\text{L}$  autoinjection into a split/splitless injector containing a single taper liner  $4\text{-mm}$  i.d. x  $78\text{-mm}$  long. Primary column analyte separation was performed using a RTX-5 column manufactured by Restek ( $0.53\text{-}\mu\text{m}$  i.d.,  $15\text{-m}$  long,  $0.1\text{-}\mu\text{m}$  film thickness). The GC parameters include a programmed temperature profile set for  $70^\circ\text{C}$  for 2 minutes,  $10^\circ\text{C}/\text{min}$  ramp to  $200^\circ\text{C}$  and then held constant at  $200^\circ\text{C}$  for 7 minutes. Confirmation analyses were performed using an RTX-225 column (Restek,  $0.53 \mu\text{m-i.d.}$ ,  $15\text{-m}$  long,  $0.1\text{-}\mu\text{m}$  film thickness). The temperature profile for the RTX-225 was programmed for  $100^\circ\text{C}$  for 2 minutes,  $10^\circ\text{C}/\text{min}$  ramp to  $200^\circ\text{C}$  and then held constant at  $200^\circ\text{C}$  for 7 minutes. The electron capture detector was operated at  $225^\circ\text{C}$  for both column types with a nitrogen makeup of  $60 \text{ mL}/\text{min}$ .

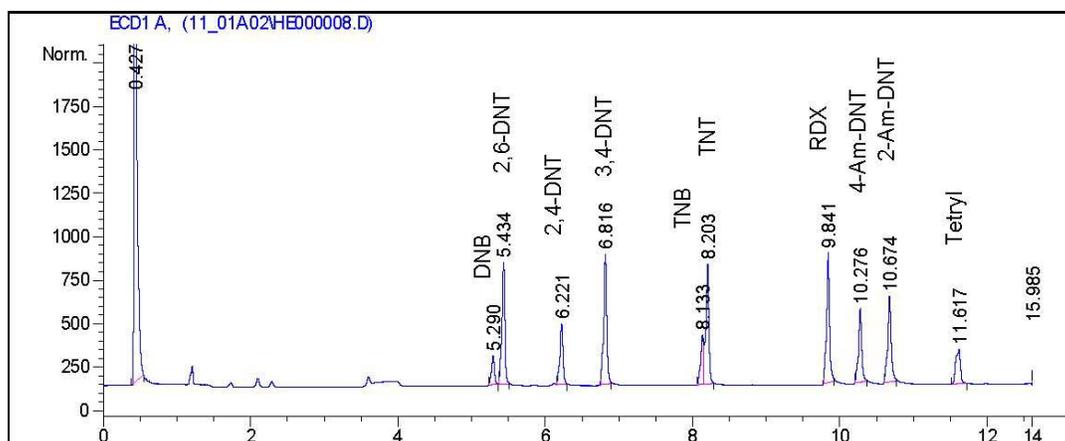
Nine sets of 20 samples were prepared and each autosampler run schedule included the following vials:

- 1 each inlet passivation,  $1000 \text{ pg}/\mu\text{L}$  (all analytes),
- 3 each blank,
- 1 each continuing calibration verification (CCV),
- 1 each laboratory method blank (LMB),
- 1 each laboratory control standard (LCS),
- 1 each matrix spike (MS),
- 1 each matrix spike duplicate (SD),
- 5 each soil extract samples,
- 1 each continuing calibration verification (CCV),
- 5 each soil extract samples,
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- 5 each soil extract samples,
- 1 each continuing calibration verification (CCV).
- 5 each soil extract samples,
- 1 each continuing calibration verification (CCV).

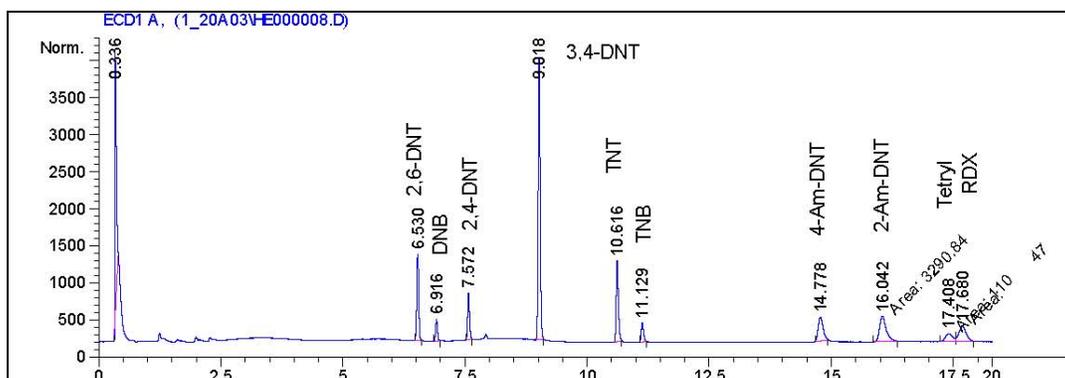
Calibration standards of 5, 10, 25, 50, 75, and  $100 \text{ pg}/\mu\text{L}$  were prepared for each batch of samples. Table 1 shows a list of the analytes quantified and the acronyms used in this report. The analyte tetryl was added to the chemical analyses because of the presence of tetryl in a mine in one of the locations. Tetryl is challenging to analyze because chemical instability leads to larger analytical error and method detection limits. Quadratic fit calibration equations were used to quantify the peak area of the sample chromatograms. Figure 1 shows a calibration standard using the RTX-5 column and Figure 2 shows the same standard on an RTX-225 column. If initial results exceed  $100\text{pg}/\text{ml}$  for any sample, the sample was diluted to be between 20 and  $100 \text{ pg}/\text{ml}$  and rerun.

**Table 1: Analyte List**

Analyte	Acronym
1,3-Dinitrobenzene	DNB
2,6-Dinitrotoluene	2,6-DNT
2,4-Dinitrotoluene	2,4-DNT
3,4-Dinitrotoluene (surrogate)	3,4-DNT
1,3,5-Trinitrobenzene	TNB
2,4,6-Trinitrotoluene	TNT
Hexahydro-1,3,5-trinitro-s-triazine	RDX
4-Amino-2,6-Dinitrotoluene	4-A-DNT
2-Amino-4,6-Dinitrotoluene	2-A-DNT
Tetryl	Tetryl



**Figure 1: RTX-5 Column Chromatogram - 50 pg standard**



**Figure 2: RTX-225 Column Chromatogram - 50 pg standard**

The Laboratory Method Blank (LMB) is an acetonitrile extract of an uncontaminated soil to evaluate the presence of naturally occurring interferents. The Laboratory Control Spike (LCS) is an uncontaminated soil spiked with the full list of analytes at 250 ng/g to evaluate bias in the soil extraction process. Both the LMB and the LCS used clean soil from Sandia National Laboratories. The Matrix Spike (MS) is similar to the LCS but used a randomly chosen sample from the suite of samples collected for analysis from the actual site. The Matrix Spike Duplicate (MSD) is used to assess variability of the analyte recoveries from the actual site matrix. The Continuing Calibration Verification (CCV) is a mid point calibration (50 pg/ $\mu$ L) standard placed every ten samples in the auto injection run to monitor instrument drift.

## **3.0 Sample Results and Discussion**

### **3.1 Quality Assurance/Quality Control Sample Results**

Surrogate recovery values for 3,4-DNT were within the acceptable range for all soil samples. The first laboratory method blank (LMB 1), had trace levels of TNT and TNB at or near the method detection limit (MDL). A confirmation of these analytes in the LMB 1 was not performed. The analyte levels are very low and have no impact on the data. In addition, the subsequent batch LMB's had no explosive analytes confirmed indicating that LMB 1 was an anomaly. Peaks found on only one column (not confirmed by the second column) are considered artifacts and do not represent detection of that analyte. Recoveries on the Laboratory Control Samples were within acceptable ranges for all analytes.

The confirmation column is used to confirm the presence of an analyte found on the primary column. If the confirmation column did not find an analyte within  $\pm 40\%$  of the value reported from the primary column, then the presence of the analyte on the primary column was not reported.

### **3.2 Method Detection Limits**

The Minimum Detectable Limits (MDL) for the analytes are shown at the bottom of each page of Table 2 and were determined from soil obtained at Sandia National Laboratories (SNL) in Albuquerque, New Mexico, USA. All of the analyte MDLs, except for tetryl, were determined in February 2000. The MDL for tetryl was determined in this effort using both the SNL soil and the Kharga, Afghanistan soil. The tetryl MDL for the combined data set are also shown at the bottom of each page of Table 2. As expected, the tetryl MDL is about ten times greater than for the other analytes. The MDL values shown in Table 2 will probably be similar to values that would be found for the Afghanistan soils because the SNL and Kharga soils are both sandy loam soils. Soils with greater organic carbon, clay size fraction or other extreme properties would likely show different results and would need to be specifically evaluated.

### 3.3 Sample Results

Table 2 shows the sample results for the all analytes with acceptable quality control results identified.

**Table 2: Qualified Sample Results (ng/g)**

	2,6-DNT	DNB	2,4-DNT	TNT	TNB	4-Amino-DNT	2-Amino-DNT	RDX	Tetryl
Kharga A7 1	U	U	U	U	U	U	U	U	U
Kharga A7 2	U	U	U	27	U	U	U	66	U
Kharga A7 6	U	U	U	29	U	U	U	41	U
Kharga A7 10	U	U	U	1108	147	U	U	549	U
Kharga A7 12	U	U	U	U	U	U	U	U	U
Kharga A7 13	U	U	U	44,477	U	U	U	36,268	U
Kharga A7 15	U	U	240	66	34	U	U	U	U
Kharga A7 17	U	U	U	U	U	U	U	U	U
Kharga A7 19	U	U	U	1109	U	U	U	3,956	U
Kharga A7 21	U	U	U	2,487	U	U	U	U	U
Kharga A7 22	U	U	U	1098	U	U	U	11787	U
Kharga A7 26	U	U	U	U	U	U	U	U	U
Kharga A7 27	U	U	U	9	U	U	U	30	U
Kharga A7 28	U	U	U	18	U	U	U	27	U
Kharga A7 298	U	U	U	376,098	U	U	U	337,254	U
Kharga A7 30	U	U	250	29	45	U	U	U	U
Kharga A7 34	U	U	U	U	U	U	U	U	U
Kharga A7 37	U	U	U	5	U	U	U	48	U
Kharga A7 38	U	U	U	U	U	U	U	U	U
Kharga A7 40	U	U	U	5	U	U	U	U	U
Kharga A7 42	U	U	13	U	U	U	U	U	U
Kharga A7 43	U	U	U	5	18	U	U	U	U
Kharga A7 46	U	U	U	9,932	U	U	U	U	U
Kharga A7 47	U	U	U	49	U	U	U	27	U
Kharga A7 48	U	U	U	99	U	U	U	401	U
Kharga A7 50	U	U	U	U	U	U	U	U	U
Kharga A7 53	U	U	U	U	U	U	U	U	U
Kharga A7 70	U	U	U	17	U	U	U	U	U
Kharga A7 102	U	U	U	440,414	231,450	U	U	U	U
Kharga A7 103	U	U	U	11	U	U	U	24	U
Kharga A7 104	U	U	258	5,166	1,201	U	U	U	U
Kharga A7 107	U	U	U	5	U	U	U	U	U
Kharga A7 110	U	U	U	2,134,141	U	U	U	2,191,183	U
Kharga A7 111	U	U	U	29	U	14	10	46	U
Kharga A7 112	U	U	U	341,595	287493	U	U	U	U
Kharga A7 116	U	U	U	U	U	U	U	U	U
Kharga A7 118	U	U	U	591,477	363,070	U	U	U	U
Kharga A7 120	U	U	U	U	U	U	U	U	U
Kharga A7 121	U	U	U	10	U	U	U	85	U
MDL (95%)	6	9	5	6	32	4	7	13	48
U-Undetectable									

**Table 2 (continued)**

	2,6-DNT	DNB	2,4-DNT	TNT	TNB	4-Amino-DNT	2-Amino-DNT	RDX	Tetryl
Kharga A7 124	U	U	U	11	U	49	39	U	U
Kharga A7 125	U	U	U	4,267,880	U	U	U	3,848,678	U
Kharga A7 127	U	U	U	U	U	U	U	U	U
Kharga A7 129	U	U	U	3,890	U	U	U	10,588	U
Kharga A7 225	U	U	U	267,633	186,929	U	U	U	U
Kharga A7 227	U	U	U	U	U	U	U	U	U
Kharga A7 233	U	U	U	9	U	U	U	U	U
Kharga A7 236	U	U	U	U	U	U	U	U	U
Kharga A7 237	U	U	U	U	U	U	U	U	U
Kharga A7 238	U	U	U	11	U	U	U	U	U
Kharga A7 240	U	U	U	15	U	U	U	U	U
Kharga A7 242	U	U	U	8	U	U	U	U	U
Kharga A7 243	U	U	U	U	U	U	U	U	U
Kharga A7 Series 3 99	U	U	U	33	U	U	U	33	U
Kharga A7 Series 3 201	U	U	22	21	U	79	51	U	U
Kharga A7 Series 3 202	U	U	U	1,041	U	U	47	684	U
Kharga A7 Series 3 204	U	U	U	U	U	U	U	U	U
Kharga A7 Series 3 209	U	U	U	U	U	U	U	U	U
Kharga A7 Series 3 210	U	U	U	8	U	U	U	U	U
Kharga A7 Series 3 213	U	U	U	U	328,243	U	U	U	389,136
Kharga A7 Series 3 215	U	U	U	U	U	U	U	1854	U
Kharga A7 Series 3 217	U	U	U	15	U	U	U	U	U
Kharga A7 Series 3 218	U	U	U	U	U	U	U	12	U
Kharga A7 Series 3 219	U	U	U	U	U	U	U	U	U
Kharga A7 Series 3 222	U	U	260	U	13	U	U	U	U
Kharga A7 Series 3 226	U	U	U	U	U	U	U	U	U
Kharga A7 Series 3 229	U	U	U	14	U	U	U	163	U
Kharga Series 4 2	U	U	U	U	U	U	U	U	U
Kharga Series 4 4	U	U	242	U	6	U	U	U	U
Kharga Series 4 6	265	U	U	U	U	U	U	U	U
Kharga Series 4 8	267,090	U	U	U	U	U	U	U	U
Kharga Series 4 10	U	U	235	U	U	U	U	U	U
Kharga Series 4 12	255	U	U	63	U	U	U	38	U
Kharga Series 4 13	U	U	U	U	87,276	U	U	U	U
Kharga Series 4 16	U	U	U	159,123	U	U	U	154,476	U
Kharga Series 4 20	U	262	U	U	U	U	U	U	U
Kharga Series 4 25	U	U	U	U	U	U	U	U	264
Kharga Series 4 26	U	U	247	U	10	U	U	U	U
Kharga Series 4 33	U	U	U	61	U	15	14	96	U
Kharga Series 4 34	U	285	8.4	36	51	U	U	U	276
Kharga Series 4 35	U	U	U	471	U	92	62	1,136	U
Kharga Series 4 42	U	271	448	59	72	U	U	U	U
Kharga Series 4 46	240	U	U	U	126	U	U	U	U
MDL (95%)	6	9	5	6	32	4	7	13	48
U-Undetectable									

**Table 2 (continued)**

	2,6-DNT	DNB	2,4-DNT	TNT	TNB	4-Amino-DNT	2-Amino-DNT	RDX	Tetryl
Kharga Series 4 48	U	290	925	U	U	U	U	U	U
Kharga Series 4 52	237	U	U	U	201	U	U	U	U
Kharga Series 4 53	U	U	U	U	34,308	U	U	U	U
Kharga Series 4 54	U	U	U	7	U	U	U	U	U
Kharga Series 4 55	U	U	U	6	U	U	U	U	U
Kharga Series 4 56	U	U	U	7	U	U	U	U	U
Kharga Series 4 59	U	269	6,177	U	U	U	U	U	U
Kharga Series 4 60	U	250	11.4	U	U	U	U	U	U
Kharga Series 4 61	U	269	U	U	U	U	U	U	U
Kharga Series 4 62	U	U	U	214	U	U	U	140	U
Kharga Series 4 67	U	U	U	7	U	U	U	U	U
Kharga Series 4 74	223	U	U	U	93	U	U	U	U
Kharga Series 4 77	U	U	232	U	U	U	U	U	U
Kharga Series 4 78	U	U	U	10	U	U	U	44	U
Kharga Series 4 79	268	U	U	U	U	U	U	U	U
Kharga Series 4 80	U	U	80	9	U	13	15	62	U
Kharga Series 4 82	U	U	U	29	U	U	U	U	U
Kharga Series 4 84	U	U	276	U	9	U	U	U	U
Kharga Series 4 88	278	U	U	U	U	U	U	U	U
Kharga Series 4 90	U	U	242	U	8	U	U	U	U
Kharga Series 4 91	U	277	86	U	U	U	U	U	U
Kharga Series 4 92	267	U	U	U	U	U	U	U	U
Kharga Series 4 94	U	U	218	U	5	U	U	U	U
Kharga Series 4 101	245	U	U	U	1,975	U	U	76	U
Kharga Series 4 103	U	U	U	U	U	U	U	U	U
Kharga Series 4 104	U	U	U	23,121	U	U	U	17726	U
Kharga Series 4 106	U	U	U	6	U	U	U	U	U
Kharga Series 4 110	U	U	U	U	10,894	U	U	U	U
Kharga Series 4 113	U	260	U	U	U	U	U	U	U
Kharga Series 4 118	253	U	U	U	U	U	U	U	U
Kharga Series 4 120	U	U	U	U	1,550,543	U	U	U	U
Kharga Series 4 126	U	268	U	U	U	U	U	U	U
Kharga Series 4 128	U	U	U	101,214	U	U	U	69,934	U
Kharga Series 4 129	229	U	237	1,570	69	72	U	101	U
Kharga Series 4 131	33,529	U	37,976	U	U	U	U	U	284
Kharga Series 4 133	U	255	U	U	U	U	U	U	U
Kharga Series 4 134	85	U	73	U	U	U	U	U	298
Kharga Series 4 136	U	U	U	35	U	U	U	U	U
Kharga Series 4 138	U	U	U	936	U	U	U	1,639	U
Kharga Series 4 141	U	U	U	U	U	U	U	U	279
Kharga Series 4 142	U	U	U	28	U	U	U	94	U
Kharga Series 4 143	5,078	U	U	U	U	U	U	U	U
Kharga Series 4 145	275	U	U	U	U	U	U	U	U
MDL (95%)	6	9	5	6	32	4	7	13	48
U-Undetectable									

**Table 2 (continued)**

	2,6-DNT	DNB	2,4-DNT	TNT	TNB	4-Amino-DNT	2-Amino-DNT	RDX	Tetryl
Kharga Series 4 147	272	U	U	198	800	U	U	178	U
Kharga Series 4 149	U	U	U	1,235,189	1,443,413	U	U	U	U
Kharga Series 4 154	256	U	U	U	U	U	U	U	U
Kharga Series 4 155	U	U	280	U	6	U	U	U	U
Kharga Series 4 156	U	244	U	U	U	U	U	U	U
Kharga Series 4 158	267	U	U	U	U	U	U	U	U
Kharga Series 4 159	U	U	233	761	232	U	U	U	U
Kharga Series 4 162	U	U	U	U	U	U	U	U	U
Kharga Series 4 163	6	U	U	U	U	U	U	U	278
Kharga Series 4 164	U	U	U	19	U	U	U	U	U
Kharga Series 4 167	U	264	U	U	U	U	U	U	U
Kharga Series 4 173	U	259	26	U	U	U	U	U	U
Kharga Series 4 174	U	262	U	U	U	U	U	U	U
Kharga Series 4 175	U	265	111	U	U	U	U	U	U
Kharga Series 4 176	U	U	U	U	U	U	U	U	U
Kharga Series 4 177	U	262	U	U	U	U	U	U	U
Kharga Series 4 179	37	U	41	U	U	U	U	162	272
Kharga Series 4 181	U	U	U	572	U	U	U	860	U
Kharga Series 4 183	U	263	10	U	U	U	U	U	U
Kharga Series 4 184	U	U	U	U	U	U	U	U	U
Kharga Series 4 190	U	U	U	U	U	U	U	U	U
Kharga Series 4 192	177	U	297	U	U	U	U	U	291
Kharga Series 4 193	U	U	U	U	8	36	34	U	U
Kharga Series 4 197	U	U	U	2139	U	U	U	1354	U
Kharga Series 4 198	U	278	10	23	22	U	U	U	U
Kharga Series 4 201	U	266	U	U	U	U	U	U	U
MDL (95%)	6	9	5	6	32	4	7	13	48
U-Undetectable									

The principal degradation byproducts of TNT are 4-A-DNT and 2-A-DNT. Samples that contain one or both of these degradation products when TNT values are also reported increases confidence in the presence of TNT. Samples where low levels of TNT are reported, but 4-A-DNT and 2-A-DNT are absent may be due to levels that are below the MDL. When the sample values become less than about 10 ng/g, uncertainty in the values increases.

Most soil chemical residues of explosive signature compounds shown in this set of samples showed typical landmine signature chemical presence. Extremely high analyte values for TNT and RDX were found in several samples. These are not indicative of landmine signatures and may be a result of contamination of surface soil by low order detonations or other methods that disperse explosive materials. Reports describing typical landmine soil chemical residues are limited. Jenkins et al. (2000) have reported this information on many landmine types over several annual seasons. In that

effort, values for surface soil residues were typically very low with results frequently below the MDL. Only about 22% of the samples have reported values above the MDL. Median values for TNT were about 4 ng/g, DNT was about 16 to 32 ng/g, and 4-A-DNT and 2-A-DNT were 17 to 44 ng/g.

This data will be provided to GICHD for use in the evaluation of mine dog performance in conjunction with the weather data and full observations.

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