**Appendix A: Supplementary materials**

Title:

**Degradation of RDX, TNT, and HMX during EPA 8330B sample processing and analysis of soils under hydrated lime or sodium dithionite based chemical remediation treatment**

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The following table presents soil metal concentrations for the general purpose sand used in the vial experiments.

Table A. 1.

Soil metal concentrations (ICP-AES)

|  |  |  |
| --- | --- | --- |
| **Metals** | **LOD** | **General purpose sand** |
|  | (mg/kg) | (mg/kg) |
| Antimony (Sb) | 0.1 | < 0.1 |
| Silver (Ag) | 0.5 | < 0.5 |
| Arsenic (As) | 2 | < 2 |
| Baryum (Ba) | 4 | 10 |
| Cadmium (Cd) | 0.1 | < 0.1 |
| Calcium (Ca) | 20 | 930 |
| Chromium (Cr) | 1 | < 1 |
| Copper (Cu) | 1 | 1 |
| Cobalt (Co) | 1 | 2 |
| Tin (Sn) | 1 | < 1 |
| Iron (Fe) | 10 | 5100 |
| Magnesium (Mg) | 5 | 830 |
| Manganese (Mn) | 2 | 190 |
| Molybdenum (Mo) | 0.5 | < 0.5 |
| Nickel (Ni) | 0.5 | 1.5 |
| Lead (Pb) | 1 | < 1 |
| Thorium (Th) | 4 | < 4 |
| Uranium (U) | 2 | < 2 |
| Zinc (Zn) | 5 | 5 |

LOD: Method’s limit of detection

Table A. 2.

Soil water content.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Soil** | **Empty dish** | **Soil** | **Combined weight** | **Final weight** | **Water weight** | **Humidity** |
|  | **(g)** | **(g)** | **(g)** | **(g)** | **(g)** | **(wt/wt %)** |
| General purpose sand | 100.14 | 50.00 | 150.14 | 150.11 | 0.03 | 0.06 |

The following table is a description of the soil treatments used in each soil vial for each experiment series.

Table A. 3.

Soil treatment description for each series of experiments.

|  |  |
| --- | --- |
| **Series #** | **Soil treatment description** |
| 1 | Control |
| 2 | Potassium carbonate and potassium bicarbonate (pH buffer) |
| 3 | Calcium hydroxide (5 % w/w) |
| 4 | MuniRem 4 mg |
| 5 | MuniRem 90 mg |
| 6 | MuniRem 4 mg, potassium carbonate and potassium bicarbonate |
| 7 | MuniRem 90 mg, potassium carbonate and potassium bicarbonate |

The following section contains the detailed result tables for the tables and figures presented within the results and discussion section of the article.

Table A. 3.

Energetic material losses due to remaining remediation reagents

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample** | **Composition B - initial concentration** | **Energetic materials - corrected initial concentration (w/o wax)** | **Total soil energetic material concentration** | **Losses** | **Average** | **Standard deviation** |
|
|  | (mg/kg) | (mg/kg) | (mg/kg) | (%) | (%) | (%) |
| **1A** | 105.97 | 104.91 | 115.0 | -9.7 | -2.0 | 14.0 |
| **1B** | 95.74 | 94.78 | 81.4 | 14.1 |
| **1C** | 90.44 | 89.54 | 98.8 | -10.4 |
| **2A** | 97.44 | 96.4656 | 62.2 | 35.5 | 19.4 | 14.2 |
| **2B** | 98.29 | 97.3071 | 89.1 | 8.5 |
| **2C** | 101.12 | 100.1088 | 85.9 | 14.2 |
| **3A** | 97.56 | 96.5844 | 3.6 | 96.3 | 89.9 | 9.8 |
| **3B** | 109.86 | 108.7614 | 5.6 | 94.9 |
| **3C** | 102.51 | 101.4849 | 21.6 | 78.7 |
| **4A** | 92.73 | 91.8027 | 81.8 | 10.9 | 1 | 9.2 |
| **4B** | 102.62 | 101.5938 | 102.0 | -0.4 |
| **4C** | 106.14 | 105.0786 | 112.9 | -7.4 |
| **5A** | 109.99 | 108.8901 | 103.9 | 4.6 | 6 | 8.1 |
| **5B** | 94.11 | 93.1689 | 94.0 | -0.9 |
| **5C** | 100.23 | 99.2277 | 84.2 | 15.1 |
| **6A** | 99.41 | 98.4159 | 60.0 | 39.1 | 32 | 7.1 |
| **6B** | 106.72 | 105.6528 | 79.3 | 24.9 |
| **6C** | 96.09 | 95.1291 | 64.4 | 32.3 |
| **7A** | 96.71 | 95.7429 | 32.2 | 66.3 | 56 | 9.4 |
| **7B** | 102.01 | 100.9899 | 52.7 | 47.8 |
| **7C** | 92.89 | 91.9611 | 41.9 | 54.4 |

1 Sum of energetic materials analytes included in EPA 8330 standards used in our method described in the manuscript.

Table A. 4.

Comparison of average ratios (HMX concentration/initial energetic material concentration) for each experiment series.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample** | **Initial Composition B concentrations** | **Corrected energetic material concentration** | **HMX soil concentration** | **Ratio** | **Average** | **Standard deviation** |
|  | (mg/kg) | (mg/kg) | (mg/kg) |  |  |  |
| **1A** | 105.97 | 104.9103 | 11.22 | 0.1069 | 0.10508 | 0.012 |
| **1B** | 95.74 | 94.7826 | 8.73 | 0.0921 |
| **1C** | 90.44 | 89.5356 | 10.40 | 0.1162 |
| **2A** | 97.44 | 96.4656 | 8.23 | 0.0854 | 0.10855 | 0.022 |
| **2B** | 98.29 | 97.3071 | 12.65 | 0.1300 |
| **2C** | 101.12 | 100.1088 | 11.04 | 0.1103 |
| **3A** | 97.56 | 96.5844 | 3.47 | 0.0360 | 0.04986 | 0.048 |
| **3B** | 109.86 | 108.7614 | 1.15 | 0.0106 |
| **3C** | 102.51 | 101.4849 | 10.45 | 0.1030 |
| **4A** | 92.73 | 91.8027 | 8.33 | 0.0908 | 0.10633 | 0.014 |
| **4B** | 102.62 | 101.5938 | 12.02 | 0.1183 |
| **4C** | 106.14 | 105.0786 | 11.55 | 0.1099 |
| **5A** | 109.99 | 108.8901 | 11.72 | 0.1076 | 0.11168 | 0.006 |
| **5B** | 94.11 | 93.1689 | 10.20 | 0.1095 |
| **5C** | 100.23 | 99.2277 | 11.70 | 0.1179 |
| **6A** | 99.41 | 98.4159 | 9.49 | 0.0964 | 0.10388 | 0.009 |
| **6B** | 106.72 | 105.6528 | 12.00 | 0.1135 |
| **6C** | 96.09 | 95.1291 | 9.68 | 0.1017 |
| **7A** | 96.71 | 95.7429 | 6.94 | 0.0725 | 0.08504 | 0.012 |
| **7B** | 102.01 | 100.9899 | 9.67 | 0.0958 |
| **7C** | 92.89 | 91.9611 | 7.99 | 0.0869 |

Table A. 5.

Comparison of average ratios (RDX concentration/initial energetic material concentration) from each experiment series.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample** | **Initial Composition B concentrations** | **Corrected energetic material concentration** | **RDX soil concentration** | **Ratio** | **Average** | **Standard deviation** |
|  | (mg/kg) | (mg/kg) | (mg/kg) |  |  |  |
| **1A** | 105.97 | 104.91 | 57.24 | 0.5456 | 0.5126 | 0.071 |
| **1B** | 95.74 | 94.78 | 40.88 | 0.4313 |
| **1C** | 90.44 | 89.54 | 50.21 | 0.5608 |
| **2A** | 97.44 | 96.47 | 44.56 | 0.4620 | 0.4998 | 0.033 |
| **2B** | 98.29 | 97.31 | 50.24 | 0.5163 |
| **2C** | 101.12 | 100.11 | 52.17 | 0.5211 |
| **3A** | 97.56 | 96.58 | 0.00 | 0.0000 | 0.0502 | 0.056 |
| **3B** | 109.86 | 108.76 | 4.42 | 0.0406 |
| **3C** | 102.51 | 101.48 | 11.17 | 0.1100 |
| **4A** | 92.73 | 91.80 | 41.55 | 0.4526 | 0.4900 | 0.033 |
| **4B** | 102.62 | 101.59 | 50.93 | 0.5013 |
| **4C** | 106.14 | 105.08 | 54.23 | 0.5160 |
| **5A** | 109.99 | 108.89 | 55.94 | 0.5137 | 0.4914 | 0.061 |
| **5B** | 94.11 | 93.17 | 50.16 | 0.5384 |
| **5C** | 100.23 | 99.23 | 41.87 | 0.4220 |
| **6A** | 99.41 | 98.42 | 49.10 | 0.4989 | 0.4956 | 0.022 |
| **6B** | 106.72 | 105.65 | 54.46 | 0.5154 |
| **6C** | 96.09 | 95.13 | 44.94 | 0.4724 |
| **7A** | 96.71 | 95.74 | 25.28 | 0.2641 | 0.3472 | 0.081 |
| **7B** | 102.01 | 100.99 | 43.04 | 0.4261 |
| **7C** | 92.89 | 91.96 | 32.31 | 0.3513 |

Table A. 6.

Comparison of average ratios (final TNT concentration/initial energetic material concentration) from each experiment series.

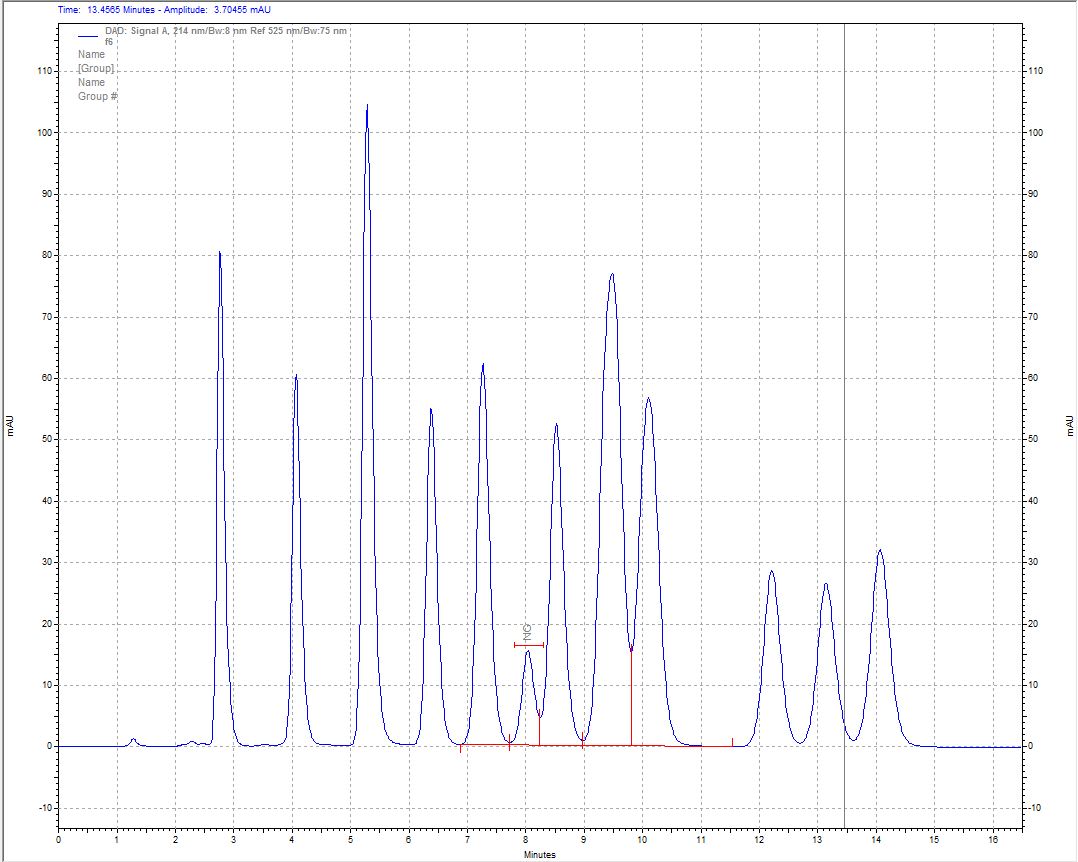
|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample** | **Initial Composition B concentrations** | **Corrected energetic material concentration** | **TNT soil concentration** | **Ratio** | **Average** | **Standard deviation** |
|  | (mg/kg) | (mg/kg) | (mg/kg) |  |  |  |
| **1A** | 105.97 | 104.91 | 46.5909 | 0.4441 | 0.4015 | 0.05907 |
| **1B** | 95.74 | 94.78 | 31.6618 | 0.3340 |
| **1C** | 90.44 | 89.54 | 38.1644 | 0.4262 |
| **2A** | 97.44 | 96.47 | 9.3803 | 0.0972 | 0.1951 | 0.08808 |
| **2B** | 98.29 | 97.31 | 26.0825 | 0.2680 |
| **2C** | 101.12 | 100.11 | 22.0211 | 0.2200 |
| **3A** | 97.56 | 96.58 | 0.0000 | 0.0000 | 0.0000 | 0.00000 |
| **3B** | 109.86 | 108.76 | 0.0000 | 0.0000 |
| **3C** | 102.51 | 101.48 | 0.0000 | 0.0000 |
| **4A** | 92.73 | 91.80 | 31.8831 | 0.3473 | 0.3930 | 0.05077 |
| **4B** | 102.62 | 101.59 | 39.0074 | 0.3840 |
| **4C** | 106.14 | 105.08 | 47.0367 | 0.4476 |
| **5A** | 109.99 | 108.89 | 35.5194 | 0.3262 | 0.3298 | 0.02833 |
| **5B** | 94.11 | 93.17 | 33.5235 | 0.3598 |
| **5C** | 100.23 | 99.23 | 30.1169 | 0.3035 |
| **6A** | 99.41 | 98.42 | 1.3793 | 0.0140 | 0.0788 | 0.05701 |
| **6B** | 106.72 | 105.65 | 12.8303 | 0.1214 |
| **6C** | 96.09 | 95.13 | 9.5911 | 0.1008 |
| **7A** | 96.71 | 95.74 | 0.0000 | 0.0000 | 0.0059 | 0.01018 |
| **7B** | 102.01 | 100.99 | 0.0000 | 0.0000 |
| **7C** | 92.89 | 91.96 | 1.6219 | 0.0176 |

Chromatograms (10.00 µg/mL Standard)

The following standard concentrations were used for calibration standards: 0.05 µg/mL, 0.50 µg/mL, 1.00 µg/mL, 2.5 µg/mL, 5.0 µg/mL, and 10.00 µg/mL. These standards were prepared from an intermediated standard at 50.00 µg/mL, itself prepared from the purchased 1000 µg/mL commercial energetic material standards.

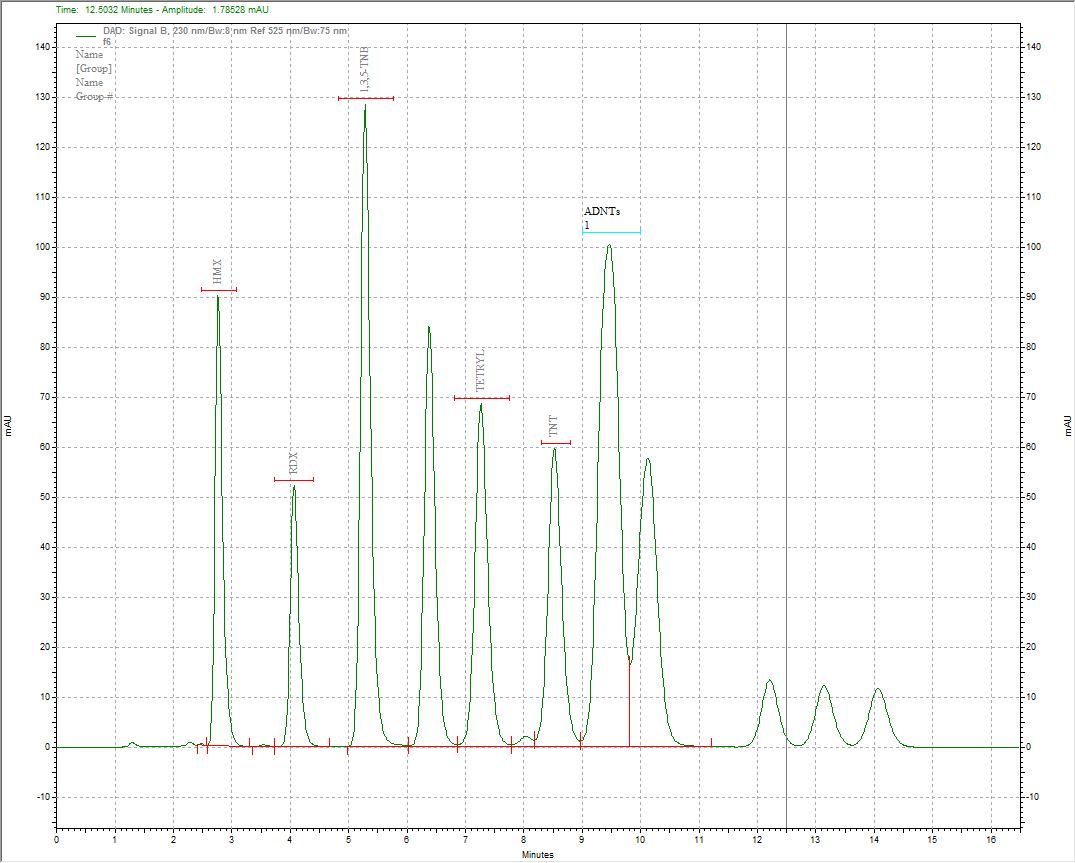
Calibration standard concentrations for ADNTs and DNTs were doubled compared to those previously mentioned for our standards due to both isomers, in each case, being integrated together: 0.1 µg/mL, 1.0 µg/mL, 2.0 µg/mL, 5.0 µg/mL, 10.0 µg/mL, and 20.0 µg/mL.

Figure A.1. shows a chromatogram at 214 nm for the 10.00 µg/mL standard. This wavelength was selected for NG analysis.



**Figure A.1:** Chromatogram (214 nm) for the 10.00 µg/mL standard.

Figure A.2. shows the chromatogram at 230 nm for the 10.00 µg/mL standard. This wavelength was selected for HMX, RDX, 1,3,5-TNB, TETRYL, TNT, and ADNTs analyses.



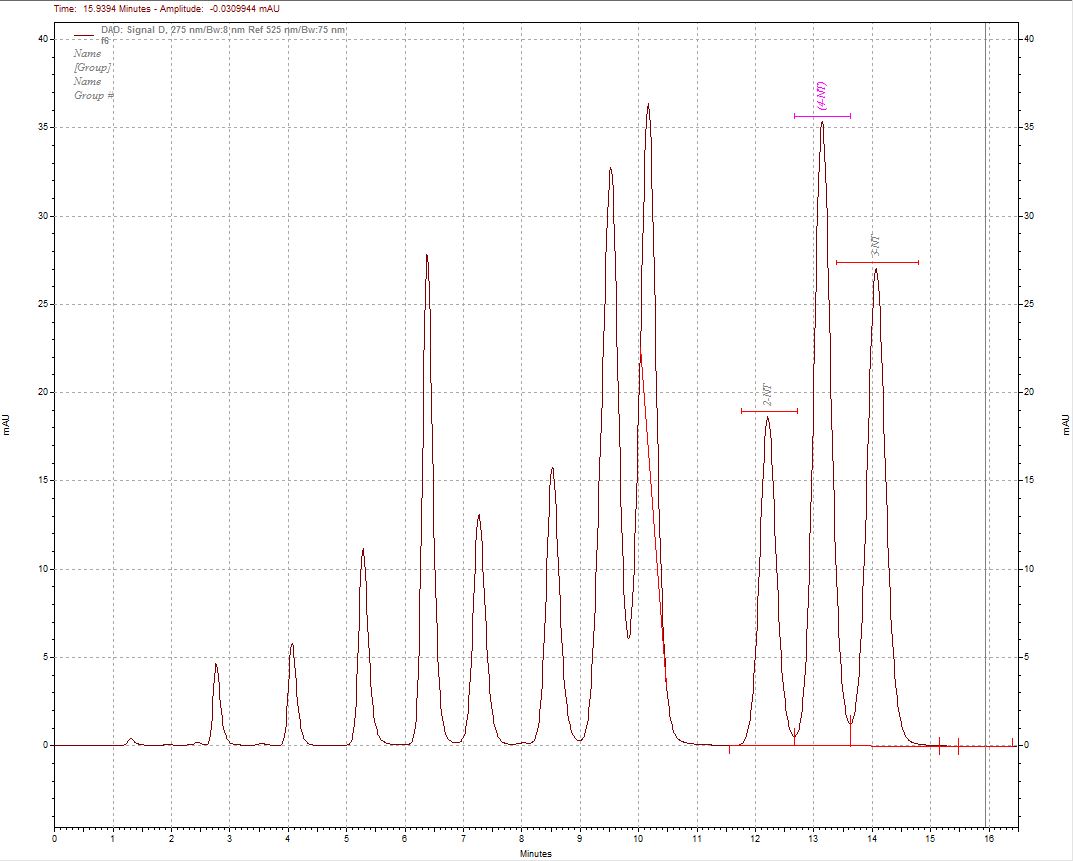
**Figure A.2:** Chromatogram (230 nm) for the 10.00 µg/mL standard.

Figure A.3 shows the chromatogram at 245 nm for the 10.00 µg/mL standard. This wavelength was selected for 1,3-DNB, and DNTs analyses.



**Figure A.3:** Chromatogram (245 nm) for the 10.00 µg/mL standard.

Figure A.4 shows the chromatogram at 275 nm for the 10.00 µg/mL standard. This wavelength was selected for 2-NT, 4-NT and 3-NTs analyses, respectively.



**Figure A.4:** Chromatogram (275 nm) for the 10.00 µg/mL standard.