METHOD 8515

COLORIMETRIC SCREENING METHOD FOR TRINITROTOLUENE (TNT) IN SOIL

1.0 SCOPE AND APPLICATION

1.1 Method 8515 is a procedure for screening soil samples to determine when 2,4,6-trinitrotoluene (TNT, CAS No. 118-96-7) is present at concentrations above 1 ppm.

1.2 Results obtained using this method should be used to locate samples with TNT concentrations between 1 and 30 ppm. Extracts of samples reading >30 ppm should be diluted and re-evaluated in the test.

1.3 Using the test kit from which this method was developed, 95% of samples containing 0.7 ppm of TNT or less will produce a negative result.

1.4 Method 8515 can be used to screen soil samples for the presence of TNT and other chemically related nitroaromatic compounds e.g., dinitrotoluenes (DNT) and 1,3,5-trinitrobenzene (TNB). It should be used as an indicator or screening test for the presence of TNT. Method 8515 does not measure RDX or HMX.

1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Test kits are commercially available for this method. The manufacturer's directions should be followed.

2.2 In general, the method is performed using an extract of a soil sample. The sample is treated with color-change reagents and is read in a portable spectrophotometer. The colorimetric nature of the test is based on the visual detection of the reaction product that is formed when polynitroaromatic compounds react with acetone by ketone substitution in the presence of base. This substitution product is measured at 540 nm using a spectrophotometer. The concentration of TNT in an unknown sample is determined by evaluating the intensity of the color that is developed.

3.0 INTERFERENCES

3.1 Chemically similar compounds and compounds which might be expected to be found in conjunction with TNT contamination were tested to determine the concentration required to produce an equivalent TNT result. These data are shown in Table 1.

4.0 APPARATUS AND MATERIALS

4.1 TNT Soil Test System (EnSys, Inc.), or equivalent. Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

4.2 UV/Vis Spectrophotometer, Hach DR/2000, or equivalent.

5.0 REAGENTS

5.1 Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 See Section 6.0 of Method 8330.

6.3 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

7.0 PROCEDURE

7.1 Follow the manufacturer's instructions for the test kit being used. Those test kits used must meet or exceed the performance specifications indicated in Tables 2-3.

7.2 Dry soil samples in air at room temperature or colder to a constant weight, being careful not to expose the samples to direct sunlight.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for the quality control procedures specific to the test kit being used. Additionally, guidance provided in Chapter One should be followed.

8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

8.3 Do not use test kits past their expiration dates.

8.4 Use the test kits within their specified storage temperature and operating temperature limits.

8.5 Verify operation of the colorimeter/spectrophotometer by use of appropriate standards.

8.6 Method 8515 is intended for field or laboratory use. The appropriate level of quality assurance should accompany the application of this method to document data quality.

9.0 METHOD PERFORMANCE

9.1 Inter- and intra-assay precision data are provided in Tables 2 and 3, respectively.

9.2 This method has been applied to a series of soil samples whose TNT concentration had been determined by HPLC (Method 8330). These results are provided in Table 4. A high degree of correlation was observed between the HPLC method and the field method.

10.0 REFERENCES

1. T. F. Jenkins and M. W. Walsh, "Development of Field Screening Methods for TNT, 2,4-DNT, and RDX in Soil", Talanta, 1992, 39 (4), 419-428.

2. T. F. Jenkins, "Development of a Simplified Field Method for the Determination of TNT in Soil", Special Report 90-38 (November, 1990) USA Cold Regions Research and Engineering Laboratory.

3. TNT Soil Test System Instructions for Use, EnSys, Inc.

4. P. P. McDonald; S. P. Arrowood, D. P. Johnson, J. P. Mapes; “TNT Soil Test Kit Validation Results”, Ensys, Inc., April 1993.

11.0 SAFETY

11.1 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the samples, extracts and standard solutions specified in this method. The only extra caution that should be taken is when handling the analytical standard neat material. Follow Section 7.2 for drying the neat material at ambient temperature. If samples are taken back to the laboratory for analysis by Method 8330, follow the additional safety procedures specified in that method.

TABLE 1

TNT Soil Test System Sensitivity to Explosive Compounds	
Compound	Minimum Sensitivity (ppm)
2,4,6-Trinitrotoluene	1
2,4-Dinitrotoluene	1.1
2,6-Dinitrotoluene	0.6
1,3,5-Trinitrobenzene	1
2-Nitrotoluene	>100
3-Nitrotoluene	>100
4-Nitrotoluene	>100
4-Amino-2,6-dinitrotoluene	>100
Nitrobenzene	>100

TABLE 2

Inter-assay Precision of the TNT Soil Test		
Spike Concentration (ppm)	Average Result (ppm ± SD)	%RSD
0	0.0 ± 0.2	-
5	5.1 ± 0.4	7.8%
10	10.1 ± 0.5	4.5%
20	20.1 ± 0.8	4.2%

These data were generated from 22 replicates in 10 matrices (9 soil extracts and 1 acetone control). (Reference 4.)

TABLE 3

Intra-assay Precision in the TNT Soil Test		
Spike Concentration (ppm)	Average Result (ppm ± SD)	%RSD
0	0.0 ± 0.2	-
10	10.2 ± 0.2	1.9%

These data were generated from the 10 ppm TNT control provided with the EnSys kit. (Reference 4.)

TABLE 4

Comparison of TNT Soil Test System with Method 8330 (HPLC)			
Sample ID	Screening Test Result (ppm)	HPLC Results (ppm)	Does screening test agree with HPLC determination?
012	18.9	21.5	yes
028	26.2	29.0	yes
022	34	25.2	no
021	34.6	23.8	no
023	37.7	28.1	yes
024	56.5	58.5	yes
027	192	191	yes
025	120	110	yes
026	120	131	yes
016	49	49	yes
013	174	175	yes
015	150	135	yes
020	295	287	yes
019	712	719	yes