

STATE OF NEW MEXICO  
ENERGY, MINERALS AND NATURAL RESOURCES DEPARTMENT  
OIL CONSERVATION COMMISSION

IN THE MATTER OF THE APPLICATION OF THE NEW MEXICO OIL AND GAS ASSOCIATION FOR AMENDMENT OF CERTAIN PROVISIONS OF TITLE 19, CHAPTER 15 OF THE NEW MEXICO ADMINISTRATIVE CODE CONCERNING PITS, CLOSED LOOP SYSTEMS, BELOW GRADE TANKS, SUMPS AND OTHER ALTERNATIVE METHODS RELATED TO THE FOREGOING AND AMENDING OTHER RULES TO CONFORMING CHANGES, STATEWIDE.

CASE NO. 14784

**SUPPLEMENTAL PRE-HEARING STATEMENT OF THE  
NEW MEXICO OIL & GAS ASSOCIATION**

This Supplemental Pre-hearing Statement is submitted on behalf of the New Mexico Oil & Gas Association (NMOGA) as instructed by the Oil Conservation Commission at the November 15, 2012, Commission hearing and pursuant to Division Rule 19.15.3.11.B NMAC.

**APPEARANCES**

**APPLICANT**

New Mexico Oil & Gas Association

**ATTORNEYS**

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Adam G. Rankin, Esq.  
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COUNSEL FOR THE NEW MEXICO  
OIL & GAS ASSOCIATION

**PROPOSED ADDITIONAL EVIDENCE**

**WITNESS:**

Clay A. Robinson  
Senior Soil Scientist,  
PhD, CPSS, PG

**ESTIMATED TIME**

1.5 Hours

**EXHIBITS**

5 Exhibits

Dr. Robinson holds a Ph.D in Soil Science from Iowa State University and will address the corrections to the "Method" column and associated asterisk for chlorides in proposed Tables I and II on page 41 of NMOGA's Exhibit 20 (filed with the "Notice Of NMOGA's Corrections To Its Proposed Amendments to Title 19, Chapter 15, Part 17"). Dr. Robinson will discuss how Tables I and II are referenced and utilized in NMOGA's proposed amendments. He will explain why the change from EPA Method 300.1 to 300.0 in Table I is appropriate for testing chlorides in impacted soils. He will explain that because this method has a defined mass for the soils, it can yield a mg/kg unit of measurement. Dr. Robinson will similarly testify that EPA SW-846 Method 1312 along with EPA Method 300.0 (as reflected in Table II) is appropriate for testing chlorides in the mixed media wastes subject to Table II. Dr. Robinson will explain that this testing method yields a mg/L unit of measurement and that it is difficult for laboratories and operators to convert the mg/L results to a mg/kg standard.

**NMOGA Exhibit 21:** to this Pre-hearing Statement is a copy of Dr. Robinson's resume.

**NMOGA Exhibit 22:** to this Pre-hearing Statement are pages from a lengthy publication from the EPA's Office of Research and Development dated August 1993 entitled "Method 300.0 Determination Of Inorganic Anions By Ion Chromatography" that identifies the scope and application of this testing method and summarizes the methodology.

**NMOGA Exhibit 23:** to this Pre-hearing Statement are pages from a lengthy EPA website publication that addresses SW-846 and Method 1312.

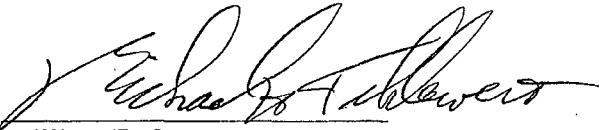
**NMOGA Exhibit 24:** to this Pre-hearing Statement are pages from a lengthy 1997 publication from the EPA's Office of Research and Development entitled "Method 300.1 Determination Of Inorganic Anions In Drinking Water By Ion Chromatography" that identifies the scope and application of this testing method and summarizes the methodology.

**NMOGA Exhibit 25:** to this Prehearing Statement is a July 24, 2008, "Memorandum" from the Director of the New Mexico Oil Conservation Division adopting EPA Method 300.0.

**PROCEDURAL MATTERS**

The New Mexico Oil and Gas Association has no procedural matters at this time.

Respectfully Submitted,

By: 

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**COUNSEL FOR**

**THE NEW MEXICO OIL AND GAS ASSOCIATION**

**CERTIFICATE OF SERVICE**

I hereby certify that on this 2nd day of January 2013, I served a copy of the foregoing **Supplemental Pre-Hearing Statement of the New Mexico Oil & Gas Association** upon following counsel of record via Hand Delivery; Electronic Mail, and U.S. Mail, postage pre-paid to:

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Los Alamos, New Mexico 87544  
(505) 662-4592  
[dneeper@earthlink.net](mailto:dneeper@earthlink.net)

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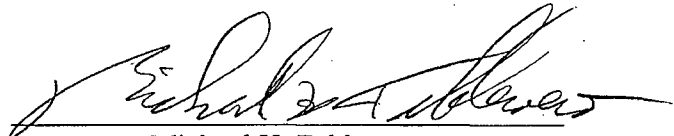
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**ATTORNEY FOR JALAPENO CORPORATION**



Michael H. Feldewert



<b>Name &amp; Title:</b> Clay A. Robinson, PhD, CPSS, PG Senior Soil Scientist	<b>Project Assignment:</b>																		
<b>Years of Experience with Firm: 1</b>	<b>Years of Experience With Other Firms: 19</b>																		
<b>Education: Degree(s) / Year / Specialization:</b> Ph.D., 1993, Iowa State University, Ames, Iowa, Soil Science (Soil Management) M.S., 1988, West Texas State University, Canyon, Texas, Agriculture B.S., 1984, West Texas State University, Canyon, Texas, Magna cum Laude	<b>Registrations / Certifications:</b> P.G., 2003, No. 1416, Licensed Professional Geoscientist: Soils, Texas Board of Professional Geoscientists  CPSS, 1999 No. 28054, Certified Professional Soil Scientist, Soil Science Society of America																		
<b>Experience Record</b> <table border="0"> <tr> <td>June 2011 - present</td> <td>Senior Soil Scientist, Stetson Engineers Inc.</td> </tr> <tr> <td>Sep., 2007 - May 2011</td> <td>Professor of Soil Science, West Texas A&amp;M University</td> </tr> <tr> <td>Sep., 2000 - 2007</td> <td>Associate Professor of Soil Science, West Texas A&amp;M University</td> </tr> <tr> <td>Sep., 2000</td> <td>Tenured, West Texas A&amp;M University</td> </tr> <tr> <td>Sep., 1994 - 2000</td> <td>Assistant Professor of Soil Science, West Texas A&amp;M University</td> </tr> <tr> <td>Sep., 1992 - 1994</td> <td>Instructor, Farm Mgr., Asst. Prof. of Agriculture, Eastern New Mexico Univ.</td> </tr> <tr> <td>Sep., 1988 - 1992</td> <td>Teaching Assistant (88-90), Research Assistant (90-92), Iowa State University</td> </tr> <tr> <td>Jan., 1986 - 1988</td> <td>Research and Teaching Assistant, West Texas State University</td> </tr> <tr> <td>May, 1984 - 1986</td> <td>Research Technician, ARCO Seed Company, Triticale Division</td> </tr> </table> <p>Assisted Dr. Bruce Buchanan in preparation for testimony before the New Mexico Oil Conservation Commission in Pit Rule hearings concerning chloride movement in the vadose zone, soil and climate characteristics, 2012.</p> <p>Cook vs Twerberg. 2012. Expert witness on agronomic effects in herbicide damage claims, Johnson County, TX. Provided report, rendered opinion, and gave deposition.</p> <p>Assisted with land classification for Navajo, Coeur d'Alene and Flathead tribes, 2010-2012.</p> <p>Copper Flat project. 2011. Topsoil assessment survey and report for environmental impact statement for a hard rock mining company, Hillsboro, NM.</p> <p>Copper Flat project. 2011. Evaluated potential impact of removing water pipeline on soil and vegetation.</p> <p>Southern Nevada Water Authority hearings. 2011. Testimony before Nevada State Engineer.</p> <p>Lonestar vs Rathjen. 2011. Provided report and rendered opinion on reason for crop failure. Cleburne, TX.</p> <p>El Paso Natural Gas vs Brian Hamilton, Fall 2010. Rendered opinion, gave depositions and testimony for arbitration on nature of soil disturbance relative to pipeline work and agronomic production characteristics.</p> <p>Stetson Engineers, Summer 2010. On-site irrigated land suitability classification (ILSC) per Navajo irrigation water rights to the San Juan River, Shiprock, NM, and ILSC per Flathead irrigation water rights near Polson, Montana.</p> <p>Rendered opinion for case related to damage associated with pipeline construction and revegetation: Were soils replaced in correct order? Wright vs. Gorman Phillips, Hemphill County, TX, 2009.</p> <p>Rendered opinion about soil/crop salinity documentation for application of oilfield brine wastes to rangeland in Hemphill County, TX for report to Texas Railroad Commission. J&amp;L Oilfield Services, Summer 2009.</p> <p>Evaluation of irrigation water availability, land requirements, and nutrient management plans for a beef backing plant in Texas County, OK. 2007. HDR Engineering. Evaluated nitrogen and phosphorus fate in alternative cropping systems. Developed water balance spreadsheets according to regulatory requirements relative to soil hydrologic groupings, crop evapotranspiration requirements, effluent/freshwater requirements, and whether current well yields would provide enough fresh water to offset salinity of effluent.</p> <p>December, 2001 to 2005, Soil background research for Fulbright &amp; Jaworski, L.L.P., in reference to questions related to BWXT-Pantex TLAP proposal for subsurface drip irrigation system to dispose treated wastewater on cropland</p> <p>Soils background research for Walker &amp; Twenhafel, L.L.P., in reference to Civil Action No. H-02-3098; Wills v. State Farm Lloyds, Inc.; In the U.S. District Court, Southern District of Texas-Houston Division</p> <p>Renewable Resources Program review, College of Applied Life Sciences, University of Louisiana-Lafayette (Curriculum, Auxiliary Units, Visits with faculty, administrators, students, alumni, and industry board), Provided written report with recommendations.</p> <p>Spring, 2000, Morton Homes, Soil and landscape assessment for problems with tree growth</p> <p>Fall, 1997, Enviro-Ag Engineering, Inc., Soil characterization/classification of a potential playa</p>		June 2011 - present	Senior Soil Scientist, Stetson Engineers Inc.	Sep., 2007 - May 2011	Professor of Soil Science, West Texas A&M University	Sep., 2000 - 2007	Associate Professor of Soil Science, West Texas A&M University	Sep., 2000	Tenured, West Texas A&M University	Sep., 1994 - 2000	Assistant Professor of Soil Science, West Texas A&M University	Sep., 1992 - 1994	Instructor, Farm Mgr., Asst. Prof. of Agriculture, Eastern New Mexico Univ.	Sep., 1988 - 1992	Teaching Assistant (88-90), Research Assistant (90-92), Iowa State University	Jan., 1986 - 1988	Research and Teaching Assistant, West Texas State University	May, 1984 - 1986	Research Technician, ARCO Seed Company, Triticale Division
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Jan., 1986 - 1988	Research and Teaching Assistant, West Texas State University																		
May, 1984 - 1986	Research Technician, ARCO Seed Company, Triticale Division																		

**Other Experiences, Qualifications, and Affiliations**

Taught fourteen undergraduate courses and associated labs, including: soils, soil fertility, soil and water conservation, soil morphology and classification, irrigation, field crops, forage crops and pastures, grain and forage crops, landscape gardening, horticulture, undergraduate statistics, environmental issues and ethics, and seminar.

Soil fertility class included soil and water analysis test methods and purposes.

Active teaching role in twelve MS and PhD graduate courses addressing soil, plants, statistics, and systems.

Coaching soil and crops judging teams.

Annually presented soils topics to about 50 farmers/consultants, 100 homeowners, and >1500 K-12 students

Author or contributing co-author for 37 refereed publications including journals, proceedings, books chapters, international journal, and encyclopedia entries, and for 30 non-refereed abstracts and proceedings. Developed 7 pedagogical publications.

Coeditor, "Know Soil, Know Life", 2012. High school environmental science/soils textbook.

Maintain DoctorDirt.org K-12 Teaching Resources and Activities.

Coeditor for high school environmental/soil science textbook, in progress

Reviewer, 2012 Next Generation Science Standards (K-12 Education)

**American Society of Agronomy**

Resident Education Division Chair for planning Annual Meetings, Long Beach CA, 2010

Associate Editor, J. Natural Resources and Life Sciences Education

2011 Outstanding Associate Editor

**Soil Science Society of America**

Chair, S591 Committee, K-12 Soils Outreach

Council of Soil Science Examiners (for soil science certification program)

STEM (Science, Technology, Engineering, and Mathematics) Task Force

**Professional Soil Scientists Association of Texas**

Soil and Water Conservation Society

International Soil Science Society

**Other Activities**

Water Retention Techniques for Roadside Vegetation Establishment in Arid Regions of Texas, TX-DOT RMC-5748. 2008. Funded for 2 years. Jim Rogers and C.A. Robinson at WTAMU, TTI at TAMU.

Synthesis and Study of the Establishment and Management of Roadside Vegetation, TX-DOT RMC-5731. 2007. Funded for 3 years. Jim Rogers and C.A. Robinson at WTAMU, TTI at TAMU, and TAMU-Kingsville.

Soil Water Monitoring of Subsurface Drip Irrigation System Under a Wheat, Sorghum, Fallow Crop Rotation and Scientific Reporting, funded for 1 year (\$24,937) with option for 4 1-year extensions (\$17,744), BWXT-PANTEX, March, 2004

Climate Characterization in the Ogallala Aquifer Region of the Texas High and Southern Plains, Ogallala Aquifer Initiative: Hydrology-Climatology Section, funded for 2 years, \$33,000 awarded, July, 2004

Irrigation and Water Best Management Practices Workshop Series, Ogallala Aquifer Initiative: Technology Transfer Section, funded for 2 years, \$17,000 awarded, July, 2004

Irrigation and Water Best Management Practices Workshop Series, Ogallala Aquifer Initiative: Technology Transfer Section, funded for 2 years, \$10,000 awarded, July, 2005

Served as major professor for eighteen (18) master's students completing a thesis. Research topics include soil spatial variability, crop and range management system impacts on soil carbon and nitrogen, environmental impact of a concentrated cattle feeding operation on adjacent rangeland. Served on 22 other M.S. Committees.

Expanded vita available upon request.



**METHOD 300.0**

**DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY**

John D. Pfaff  
Inorganic Chemistry Branch  
Chemistry Research Division

Revision 2.1  
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
U.S. ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268

BEFORE THE OIL CONSERVATION DIVISION  
CASE NO. 14784 NMOGA EXHIBIT 22  
JANUARY 9, 2013

## METHOD 300.0

### DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

#### 1.0 SCOPE AND APPLICATION

- 1.1 This method covers the determination of the following inorganic anions:

##### PART A.

Bromide	Nitrite
Chloride	Ortho-Phosphate-P
Fluoride	Sulfate
Nitrate	

##### PART B.

Bromate	Chlorite
Chlorate	

- 1.2 The matrices applicable to each method are shown below:

1.2.1 Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 11.7), leachates (when no acetic acid is used).

1.2.2 Drinking water and reagent waters

- 1.3 The single laboratory Method Detection Limit (MDL defined in Section 3.2) for the above analytes is listed in Tables 1A and 1B. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample.

- 1.4 Method A is recommended for drinking and wastewaters. The multilaboratory ranges tested for each anion are as follows:

<u>Analyte</u>	<u>mg/L</u>
Bromide	0.63 - 21.0
Chloride	0.78 - 26.0
Fluoride	0.26 - 8.49
Nitrate-N	0.42 - 14.0
Nitrite-N	0.36 - 12.0
Ortho-Phosphate-P	0.69 - 23.1
Sulfate	2.85 - 95.0

- 1.5 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

- 1.6 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 11.6.
- 1.7 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Section 9.0.

## **2.0 SUMMARY OF METHOD**

- 2.1 A small volume of sample, typically 2-3 mL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.
- 2.2 The main differences between Parts A and B are the separator columns and guard columns. Sections 6.0 and 7.0 will elicit the differences.
- 2.3 An extraction procedure must be performed to use this method for solids (See Section 11.7).
- 2.4 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Section 9.0, Quality Control.

## **3.0 DEFINITIONS**

- 3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.
- 3.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.3 **Field Duplicates (FD)** -- Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.4 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

- 11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

- 11.7 The following extraction should be used for solid materials. Add an amount of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed for 10 minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45  $\mu$  membrane type filter. This can be the type that attaches directly to the end of the syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the user's matrix through the use of fortified samples.
- 11.8 It has been reported that lower detection limits for bromate ( $\approx 7 \mu\text{g/L}$ ) can be obtained using a borate based eluent<sup>(7)</sup>. The use of this eluent or other eluents that improve method performance may be considered as a minor modification of the method and as such still are acceptable.
- 11.9 Should more complete resolution be needed between peaks the eluent (7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

## 12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.
- 12.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.
- 12.3 Report results in mg/L.
- 12.4 Report         $\text{NO}_2^-$  as N  
                   $\text{NO}_3^-$  as N  
                   $\text{HPO}_4$  as P

## 13.0 METHODS PERFORMANCE

- 13.1 Tables 1A and 2A give the single laboratory (EMSL-Cincinnati) MDL for each anion included in the method under the conditions listed.



<http://www.epa.gov/waste/hazard/testmethods/sw846/online/>  
Last updated on Thursday, November 15, 2012

## Wastes - Hazardous Waste - Test Methods

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### SW-846 On-line

#### [Table of Contents](#) | [Chapters](#)

SW-846 contains over 200 documents, including the Table of Contents, Disclaimer, Preface, Chapters One through Thirteen, and many different methods for the sampling and analysis of wastes. All of the documents found in the Third Edition of SW-846, as updated by Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA and IVB are located at this site. Just click on the appropriate document type at the top of this page, which will in turn take you to a complete listing of the applicable documents and links to each of those documents. For example, in order to locate Method 1311, the TCLP, click on the phrase "1000 Series," which will take you to a listing of the SW-846 methods in the 1000 series.

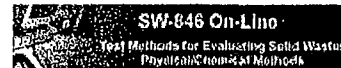
#### Table of Contents

[Table of Contents \(PDF\)](#) (15 pp, 41K)  
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[Abstract \(PDF\)](#) (1 pg, 132K)  
[Method Index and Conversion Table \(PDF\)](#) (4 pp, 94K)  
[Methods Status Table \(PDF\)](#) (26 pp, 96K)  
[Preface \(PDF\)](#) (2 pp, 33K)  
[Acknowledgements \(PDF\)](#) (1 pg, 47K)  
[Appendix -- Company References \(PDF\)](#) (3 pp, 51K)

#### Chapters

[Chapter One -- Quality Control \(PDF\)](#) (33 pp, 274K)  
[Chapter Two -- Choosing the Correct Procedure \(PDF\)](#) (80 pp, 591K)  
[Chapter Three -- Inorganic Analytes \(PDF\)](#) (28 pp, 272K)  
[Chapter Four -- Organic Analytes \(PDF\)](#) (20 pp, 96K)  
[Chapter Five -- Miscellaneous Test Methods \(PDF\)](#) (2 pp, 16K)  
[Chapter Six -- Properties \(PDF\)](#) (1 pg, 15K)  
[Chapter Seven -- Characteristics Introduction and Regulatory Definitions \(PDF\)](#) (7 pp, 35K)  
[Chapter Eight -- Methods for Determining Characteristics \(PDF\)](#) (4 pp, 13K)  
[Chapter Nine -- Sampling Plan \(PDF\)](#) (79 pp, 684K)  
[Chapter Ten -- Sampling Methods \(PDF\)](#) (1 pg, 13K)  
[Chapter Eleven -- Ground Water Monitoring \(PDF\)](#) (1 pg, 9.8K)  
[Chapter Twelve -- Land Treatment Monitoring \(PDF\)](#) (16 pp, 220K)  
[Chapter Thirteen -- Incineration \(PDF\)](#) (19 pp, 228K)

#### Highlighted Resources



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## Wastes - Hazardous Waste - Test Methods

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### 1000 Series Methods

[Method 1010A \(PDF\)](#) (1 pg, 19K)

Test Methods for Flash Point by Pensky-Martens Closed-Cup Tester

[Method 1020B \(PDF\)](#) (1 pg, 17K)

Standard Test Methods for Flash Point by Setaflash (Small Scale) Closed-Cup Apparatus

[Method 1030 \(PDF\)](#) (13 pp, 116K)

Ignitability of Solids

[Method 1040 \(PDF\)](#) (17 pp, 207K)

Test Method for Oxidizing Solids

[Method 1050 \(PDF\)](#) (20 pp, 722K)

Test Methods to Determine Substances Likely to Spontaneously Combust

[Method 1110A \(PDF\)](#) (6 pp, 36K)

Corrosivity Toward Steel

[Method 1120 \(PDF\)](#) (12 pp, 118K)

Dermal Corrosion

[Method 1310B \(PDF\)](#) (18 pp, 133K)

Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test

[Method 1311 \(PDF\)](#) (35 pp, 288K)

Toxicity Characteristic Leaching Procedure

[Method 1312 \(PDF\)](#) (30 pp, 1.2MB)

Synthetic Precipitation Leaching Procedure

[Method 1313 \(PDF\)](#) (30 pp, 422.37K)

Liquid-Solid Partitioning as a Function of Extract pH using a Parallel Batch Extraction Procedure

[Method 1316 \(PDF\)](#) (20 pp, 292.06K)

Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio using a Parallel Batch Extraction Procedure.

#### SW-846 On-Line

Test Methods for Evaluating Solid Wastes  
Physical/Chemical Methods

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## METHOD 1312

### SYNTHETIC PRECIPITATION LEACHING PROCEDURE

#### 1.0 SCOPE AND APPLICATION

1.1 Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

#### 2.0 SUMMARY OF METHOD

2.1 For liquid samples (*i.e.*, those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter, is defined as the 1312 extract.

2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter.

2.3 If compatible (*i.e.*, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

#### 3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

#### 4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at  $30 \pm 2$  rpm. Suitable devices known to EPA are identified in Table 2.

##### 4.2 Extraction Vessels

4.2.1 Zero Headspace Extraction Vessel (ZHE). This device is for use only when the sample is being tested for the mobility of volatile analytes (*i.e.*, those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device and effectively precludes headspace. This type of vessel allows for initial liquid/solid

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at  $90 \pm 5$  degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Sulfuric acid/nitric acid (60/40 weight percent mixture)  $H_2SO_4/HNO_3$ . Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute  $H_2SO_4/HNO_3$  acid mixture may be prepared and used in steps 5.4.1 and 5.4.2 making it easier to adjust the pH of the extraction fluids.

#### 5.4 Extraction fluids.

5.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is  $4.20 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.

NOTE: Solutions are unbuffered and exact pH may not be attained.

5.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is  $5.00 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

5.4.3 Extraction fluid #3: This fluid is reagent water (Step 5.2) and is used to determine cyanide and volatiles leachability.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

#### 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 There may be requirements on the minimal size of the field sample depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent



solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the sample is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).

6.6 1312 extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Step 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Step 8.0 (Quality Control) for acceptable sample and extract holding times.

## 7.0 PROCEDURE

### 7.1 Preliminary Evaluations

Perform preliminary 1312 evaluations on a minimum 100 gram aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include: (1) determination of the percent solids (Step 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Step 7.1.2); and (3) determination of whether the solid portion of the waste requires particle size reduction (Step 7.1.3).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a representative subsample (100 g minimum) and proceed to Step 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device

7.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Step 7.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \% \text{ solids (Step 7.1.1)} \times \text{weight of waste filtered (Step 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Step 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at  $30 \pm 2$  rpm for  $18 \pm 2$  hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at  $23 \pm 2^\circ\text{C}$  during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the  $18 \pm 2$  hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 7.2.7. For final filtration of the 1312 extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the 1312 extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.2.12 is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 7.2.12 with the initial liquid phase of the sample obtained

in Step 7.2.7. This combined liquid is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Step 7.2.7, is not or may not be compatible with the filtered liquid resulting from Step 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the 1312 extract, and combine the results mathematically, as described in Step 7.2.14.

7.2.14 Following collection of the 1312 extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The 1312 extract shall be prepared and analyzed according to appropriate analytical methods. 1312 extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to  $\pm 0.5\%$ ), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

$V_1$  = The volume of the first phase (L).

$C_1$  = The concentration of the analyte of concern in the first phase (mg/L).

$V_2$  = The volume of the second phase (L).

$C_2$  = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

### 7.3 Procedure When Volatiles Are Involved

Use the ZHE device to obtain 1312 extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

**METHOD 300.1 DETERMINATION OF INORGANIC ANIONS IN DRINKING  
WATER BY ION CHROMATOGRAPHY**

Revision 1.0

John D. Pfaff (USEPA, ORD, NERL) - Method 300.0, (1993)

Daniel P. Hautman (USEPA, Office of Water) and David J. Munch (USEPA, Office of Water) -  
Method 300.1, (1997)

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300.1-1

**BEFORE THE OIL CONSERVATION DIVISION  
CASE NO. 14784 NMOGA EXHIBIT 24  
JANUARY 9, 2013**

## METHOD 300.1

### DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY

#### 1.0 SCOPE AND APPLICATION

- 1.1 This method covers the determination of the following inorganic anions in reagent water, surface water, ground water, and finished drinking water. As a result of different specified injection volumes (See conditions in Tables 1A and 1B), these anions are divided between the common anions listed in Part A and the inorganic disinfection by-products listed in Part B. These different injection volumes are required in order to compensate for the relative concentrations of these anions in drinking water and maintain good chromatographic peak shape throughout the expected dynamic range of the detector. Bromide is included in both Part A, due to its importance as a common anion, as well as Part B due to its critical role as a disinfection by-product precursor.

##### PART A.-- Common Anions

Bromide	Nitrite
Chloride	ortho-Phosphate-P
Fluoride	Sulfate
Nitrate	

##### PART B.-- Inorganic Disinfection By-products

Bromate	Chlorite
Bromide	Chlorate

- 1.2 The single laboratory Method Detection Limits (MDL, defined in Sect. 3.11) for the above analytes are listed in Tables 1A, 1B and 1C. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.
- 1.2.1 In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more than 5 nS noise/drift per minute of monitored response over the background conductivity.
- 1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

- 1.4 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Sect. 9.4.1.
- 1.5 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Sect. 9.0.
- 1.6 Bromide and nitrite react with most oxidants employed as disinfectants. The utility of measuring these anions in treated water should be considered prior to conducting the analysis.

## **2.0 SUMMARY OF METHOD**

- 2.1 A small volume of sample, 10  $\mu\text{L}$  for Part A and 50  $\mu\text{L}$  for Part B, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.
- 2.2 The ONLY difference between Parts A and B is the volume of sample analyzed by the ion chromatographic system. The separator columns and guard columns as well as eluent conditions are identical.

## **3.0 DEFINITIONS**

- 3.1 ANALYSIS BATCH -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices). The analysis batch must include an Initial Calibration Check Standard, an End Calibration Check Standard, Laboratory Reagent Blank, and a Laboratory Fortified Blank. Within an ANALYSIS BATCH, for every group of ten field samples, at least one Laboratory Fortified Matrix (LFM) and either a Field Duplicate, a Laboratory Duplicate or a duplicate of the LFM must be analyzed. When more than 10 field samples are analyzed, a Continuing Calibration Check Standard must be analyzed after the tenth field sample analysis.
- 3.2 CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

# New Mexico Energy, Minerals and Natural Resources Department

**Bill Richardson**  
Governor

Joanna Prukop  
Cabinet Secretary  
Reese Fullerton  
Deputy Cabinet Secretary

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Division Director  
Oil Conservation Division



July 24, 2008

## MEMORANDUM

**Clarification of analytical test method, EPA Method 300.1, for chloride in regards to 19.15.36 NMAC and 19.15.17 NMAC.**

On June 16, 2008 a new regulation regarding the permitting, design and construction, operations and closure of pits, closed-loop systems, below-grade tanks, and sumps, 19.15.17 NMAC, went into effect. On February 14, 2007 the new surface waste management regulation, 19.15.36 NMAC, also went into effect. Each of these rules, 19.15.17 NMAC and 19.15.36 NMAC, established EPA test methods for chlorides.

The Oil Conservation Division (OCD) has received several inquiries regarding implementation of EPA Method 300.1 for chloride, as specified in the above referenced rules. In order to address these inquiries and to provide clarification to operators and laboratories, OCD wishes to identify the following test methods as "other approved methods" that OCD will consider acceptable in lieu of EPA Method 300.1, as specified in 19.15.17 NMAC and 19.15.36 NMAC:

EPA Method 300.0 (extraction utilizing deionized water)  
Standard Method 4500B

If you have any questions regarding this matter, please contact Brad A. Jones of my staff at (505) 476-3487 or [brad.a.jones@state.nm.us](mailto:brad.a.jones@state.nm.us).

Sincerely,

A handwritten signature in black ink, appearing to read "Mark E. Fesmire", is written over a horizontal line.

Mark E. Fesmire  
Director, Oil Conservation Division

ME/baj

cc: Daniel Sanchez, Enforcement & Compliance Manager, OCD, Santa Fe, NM  
Wayne Price, Bureau Chief, Environmental Bureau, OCD, Santa Fe, NM  
Chris Williams, District Supervisor, District I, OCD, Hobbs, NM  
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A handwritten signature in black ink, appearing to read "Mark E. Fesmire", is written over a horizontal line.

BEFORE THE OIL CONSERVATION DIVISION  
CASE NO. 14784 NMOGA EXHIBIT 25  
JANUARY 9, 2013