Marcellus Shale Coalition
Dissolved Methane Method Study

National Environmental Monitoring Conference
July 13-17, 2015
Chicago, Illinois

Presented by Rock J. Vitale, CEAC
Environmental Standards, Inc.
Agenda

- Study Objectives
- Laboratory Coordination
- Sample Collection
- Results
- Conclusions
The Problem

- Public concern
- Data variability observed by MSC members
- Several published procedures for dissolved light gases
- No US EPA-published method
- Lack of standardization
MSC Dissolved Methane Method Workgroup

- Formed to study this issue in early 2013
- Compared notes and reviewed data/information:
  - Dissolved methane split sample data
  - Laboratory analytical protocols
  - Brainstormed as to cause of variability
- Requested funding from the MSC Board for a Round Robin Study
- Competitive RFP issued to MSC members to complete the Study
- MSC engaged Environmental Standards, Inc. to conduct the Study
Study Sponsors, Executor, and Participants

- Select members of the MSC Dissolved Methane Method Work Group
- Environmental Standards, Inc.
- 15 Participating Laboratories (14 commercial, one government)
Study Objectives

- Design and oversee a credible inter-laboratory study that can withstand scientific scrutiny
- Determine the variability across 15 laboratories using controlled samples
- Identify the critical laboratory variables that influence the quantitation of dissolved methane in groundwater
- Recommend Best Practices for the analysis of dissolved methane in groundwater
- Not a sample collection procedure study
Laboratory Coordination

- **Laboratory Key Elements Questionnaire**
  - 102 questions identifying critical laboratory variables
  - Detailed preparation and analytical procedures

- **Standard Operating Procedures**
  - Sample receiving
  - Sample and standard storage
  - Dissolved gases sample preparation
  - Dissolved gases analytical methodology
  - Integration of chromatographic peaks
On-Site Sampling

- Two domestic wells, confidential locations
- Vial verification
- Direct fill method employed
- 396 vials collected
- Preserved and unpreserved
- Use of butyl rubber-Teflon faced septa
- Three measured time intervals collected
- Effervescence observed
- Each laboratory received 6 samples
  - Three samples per well
  - Nine vials per well per laboratory
- Instructed laboratories to analyze based on their SOP and Questionnaire response
- Each laboratory performed analysis within 48 hours of receipt
- Laboratory deliverables: Certificate of Analysis, EDD, and Level IV data package
Data Analysis

- Compiled analytical data
- Compiled data from key elements questionnaire
- Evaluated SOPs
- Performed exploratory and statistical data analysis to identify “groupings” of data and controlling variables
  - Wilcoxon/Kruskal-Wallis Tests
- Performed data verification of selected labs’ Level IV data package
Well 1 - Dissolved Methane Results

Dissolved Methane Results (ug/L)

Sample Collection Time

Methane Result
Average (21071.39)
Standard Deviation (7053.13)
%RSD (33.47)
Trend (Linear)
Well 2 - Dissolved Methane Results

Sample Collection Time vs. Dissolved Methane Results (µg/L)

- Methane Result
- Average (23565.13)
- Standard Deviation (8533.72)
- %RSD (36.21)
- Trend (Linear)
Preserved vs. Unpreserved

Dissolved Methane Results (ug/L)

Sample Collection Time

Well 1
Well 2

Preserved Result
Unpreserved Result
Preliminary Conclusions

- Range of reported concentrations verifies MSC observations
  - Methane variability is a concern when applying regulatory standards to groundwater

- Based upon the 102 process questions and SOPs reviewed, there is a very broad range of laboratory approaches

- No “smoking gun” to explain the variability of reported concentrations
Recommendations

- The study recommendations include procedures specific for instrument calibration, sample handling/preparation, analysis, and calculations.
  - Seek collaboration from MSC laboratory members to develop a consensus procedure.
- Of most importance is development of a certified performance sample that each laboratory can use to gauge their analysis.
Additional Studies Recommended

- Repeat study at lower dissolved methane concentrations
- Sample collection study to determine how varying procedures affect dissolved methane concentrations
- Round robin study using a controlled dissolved gases analytical procedure developed by the MSC
Environmental Standards, Inc.
“Setting the Standards for Innovative Environmental Solutions”

**Headquarters** 1140 Valley Forge Road | PO Box 810 | Valley Forge, PA 19482 | 610.935.5577
**Virginia** 1412 Sachem Place | Charlottesville, VA 22901 | 434.293.4039
**Tennessee** 8331 East Walker Springs Lane, Suite 402 | Knoxville, TN 37923 | 865.376.7590
**Texas** 2000 S. Dairy Ashford Road, Suite 450 | Houston, TX 77077 | 281.752.9782
**New Mexico** PO Box 29432 | Santa Fe, NM 87592 | 505.660.8521
**Illinois** PO Box 62 | Geneva, IL 60134 | 630.262.3979

www.envstd.com | solutions@envstd.com