

Carbon Addition as a Factor Contributing to Variability in Soil Test Sulfate-Sulfur Results

A. GUNLOGSON, K. PIOTROWSKI, S. GAWTRY, AND B. BARBER
 SOIL TESTING AND RESEARCH ANALYTICAL LABORATORIES
 UNIVERSITY OF MINNESOTA

Introduction

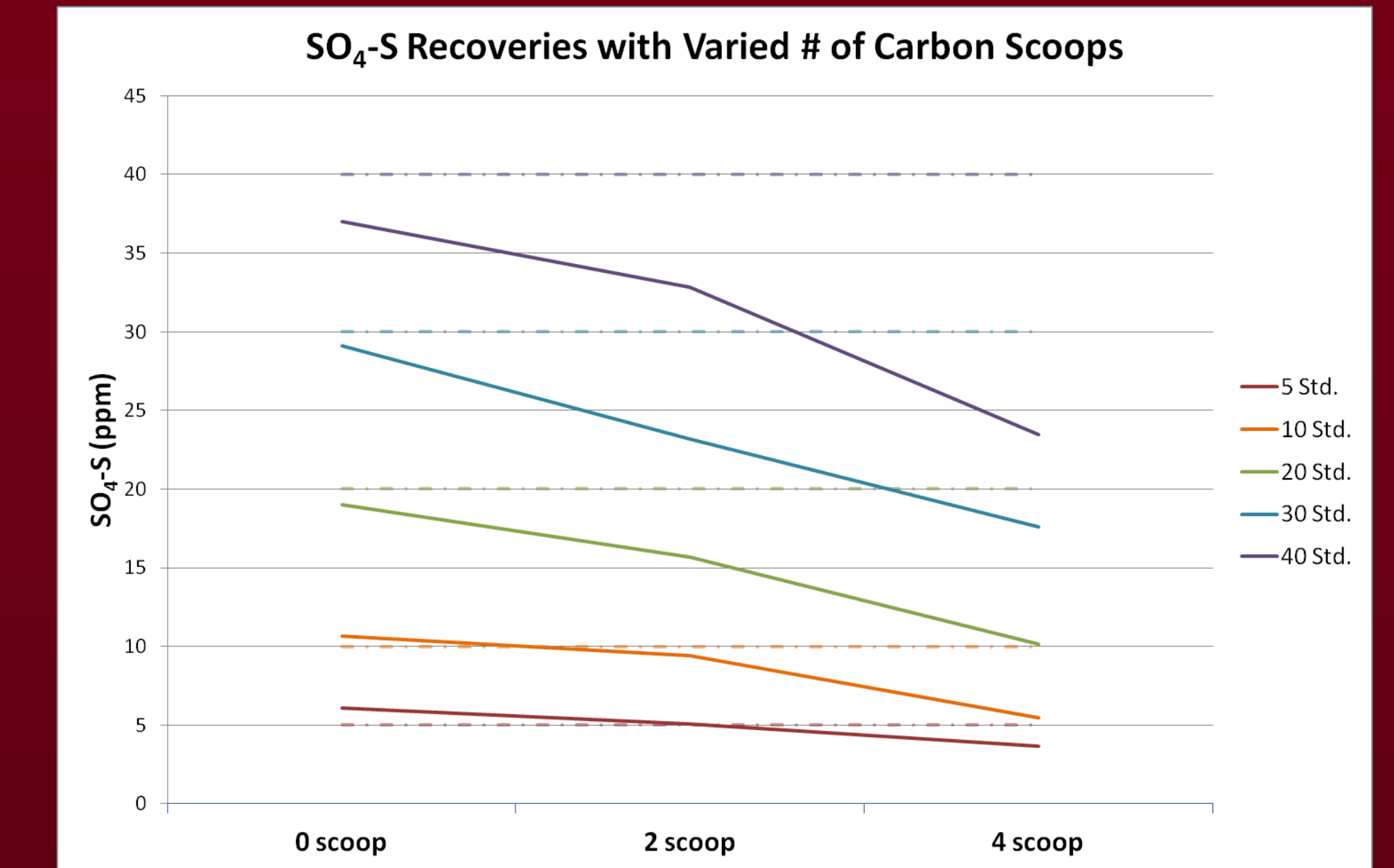
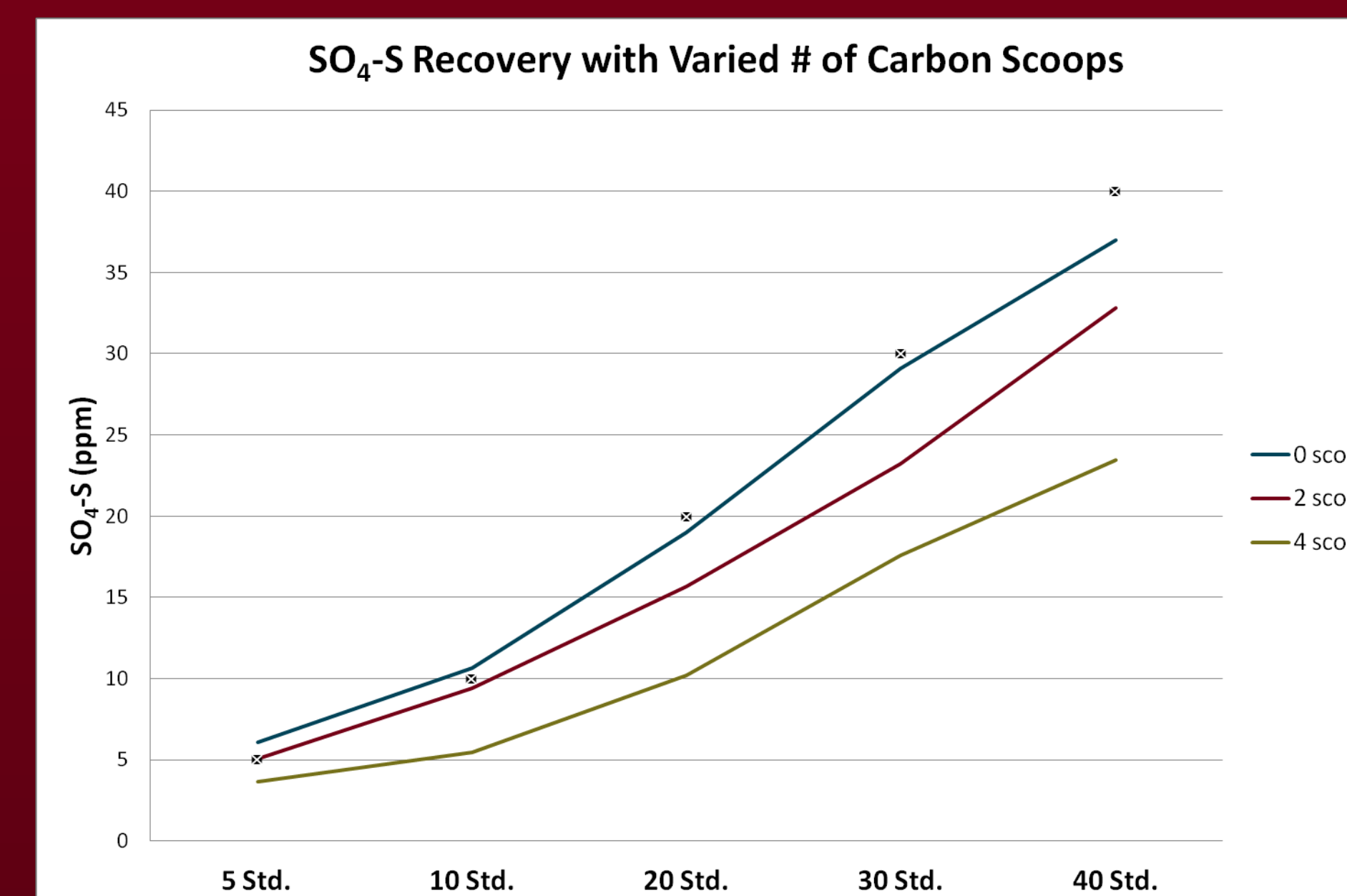
Changes in industrial practices have led to reduced atmospheric deposition of sulfur. This fact, considered along with increased yield responses to sulfur fertilization, leads to the quantification of soil sulfate-sulfur as an important step in environmental monitoring and fertility research. This poster details efforts to modify and improve laboratory techniques for soil sulfate-sulfur measurement in agronomic systems. Concurrent with a recent upgrade from manual determinations of extractable soil sulfate-S via a Klett colorimeter (ca. 1950), to an automated turbidimetric procedure using a Lachat Quikchem 8500 series 2 Flow Injection Analyzer, we embarked on an ongoing series of comparisons of the factors influencing precision and accuracy of results within the NCR-13 standardized method currently in use. Our data shows a marked decrease in sulfate-sulfur recovery associated with the use of activated carbon (used to clarify sample filtrate). Our data also shows differential effects across the analytical range.



Figure 1.
Lachat Quikchem 8500 Flow Injection Analyzer



Figure 2.
Simultaneous extraction of dozens of sulfate-sulfur samples.



Figures 6-7.

Two representations of differential sulfate-S recovery when adding varied amounts of activated carbon to sulfate-sulfur calibration standards.

Discussion

- Adding carbon to working standards reduces recovery of $\text{SO}_4\text{-S}$ relative to the same material without carbon added.
- Adding more carbon further reduces $\text{SO}_4\text{-S}$ recovery.
- Adding the same amount of carbon leads to greater loss of recovery at high concentrations relative to low concentrations.
- In order to meet ALP certification, we calibrate with “pure” (no added carbon) standards.
- Differences in recoveries from working standards with and without carbon added (represented in Figures 3-5) are statistically significant at $p < 0.05$, as determined by a Welch’s two-sample t-test.

Methods and Materials

- Soil sulfate-sulfur is extracted with a modified form of the method presented by Combs, Denning, and Frank in NCR-13 “Recommended Chemical Soil Test Procedures for the North Central Region” (1998, Missouri Agricultural Experiment Station).
- Modifications to the published method are as follows:
 - 50% (~5.5 M) HCl is used instead of 0.1 M $\text{Ca}(\text{H}_2\text{PO}_4)_2$ to “wash” carbon before addition to extracts.
 - A 0.2-ml volumetric scoop is used to add a standard volume of carbon instead of weighing 0.15 grams.
 - The analytical instrument is calibrated using “pure” standards, with no carbon added. The NCR-13 method suggests treating calibration standards with carbon.
- Sulfate-sulfur concentration is determined turbidimetrically on a Lachat QuikChem 8500 Flow Injection Analyzer, using Method #12-116-10-1-D.

Conclusions

- Adding carbon to standard material leads to differential recoveries of sulfate-sulfur.
- Those recoveries are not consistent across the analytical range.
- Due to the differential effects of carbon addition, extreme care must be taken when adding carbon to sample extracts.
- More work must be done to determine if the same effect holds true across a range of soil types and sulfate-sulfur contents.

Figure 3. Mean Difference = 1.26 ppm

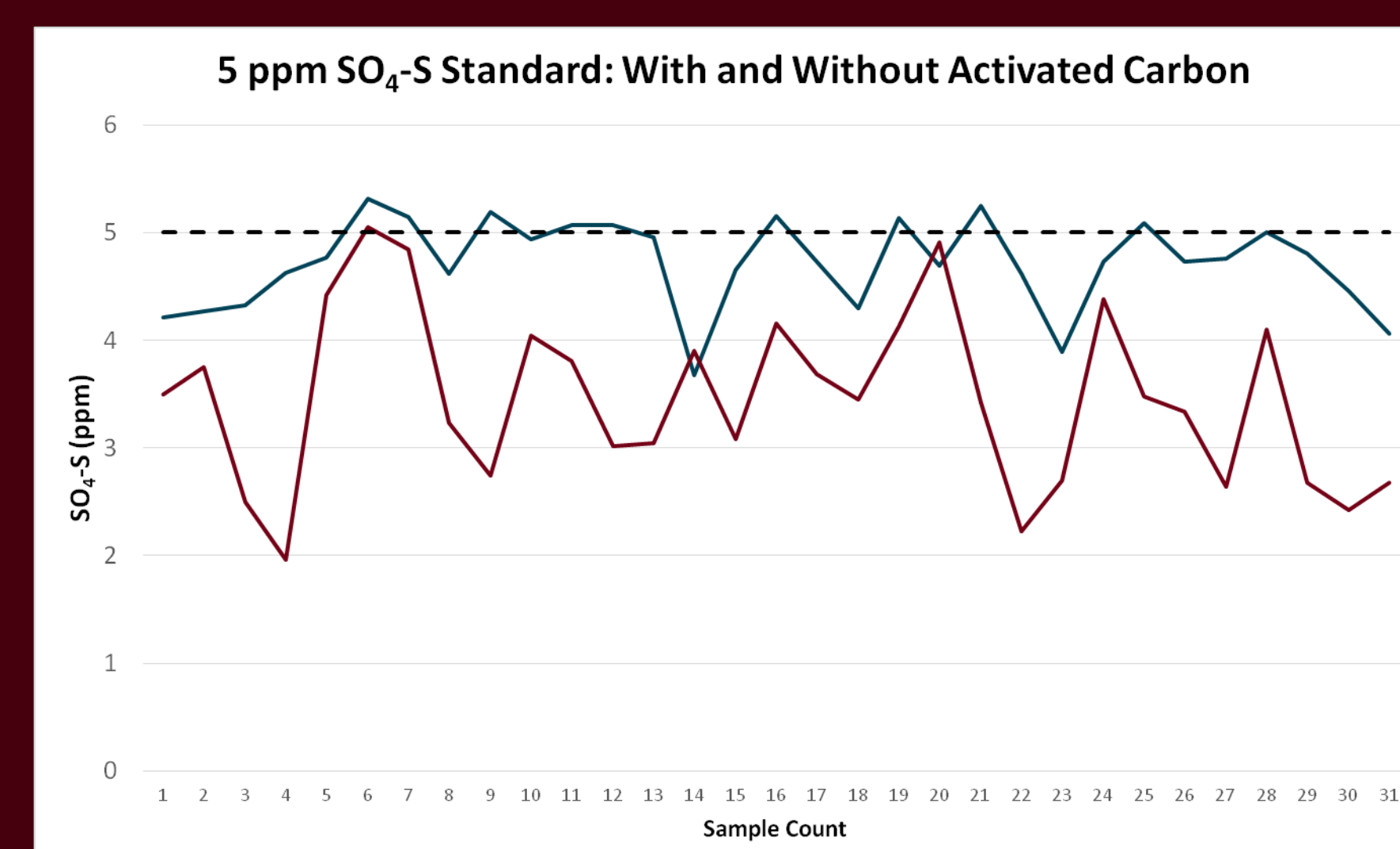


Figure 4. Mean Difference = 6.75 ppm.

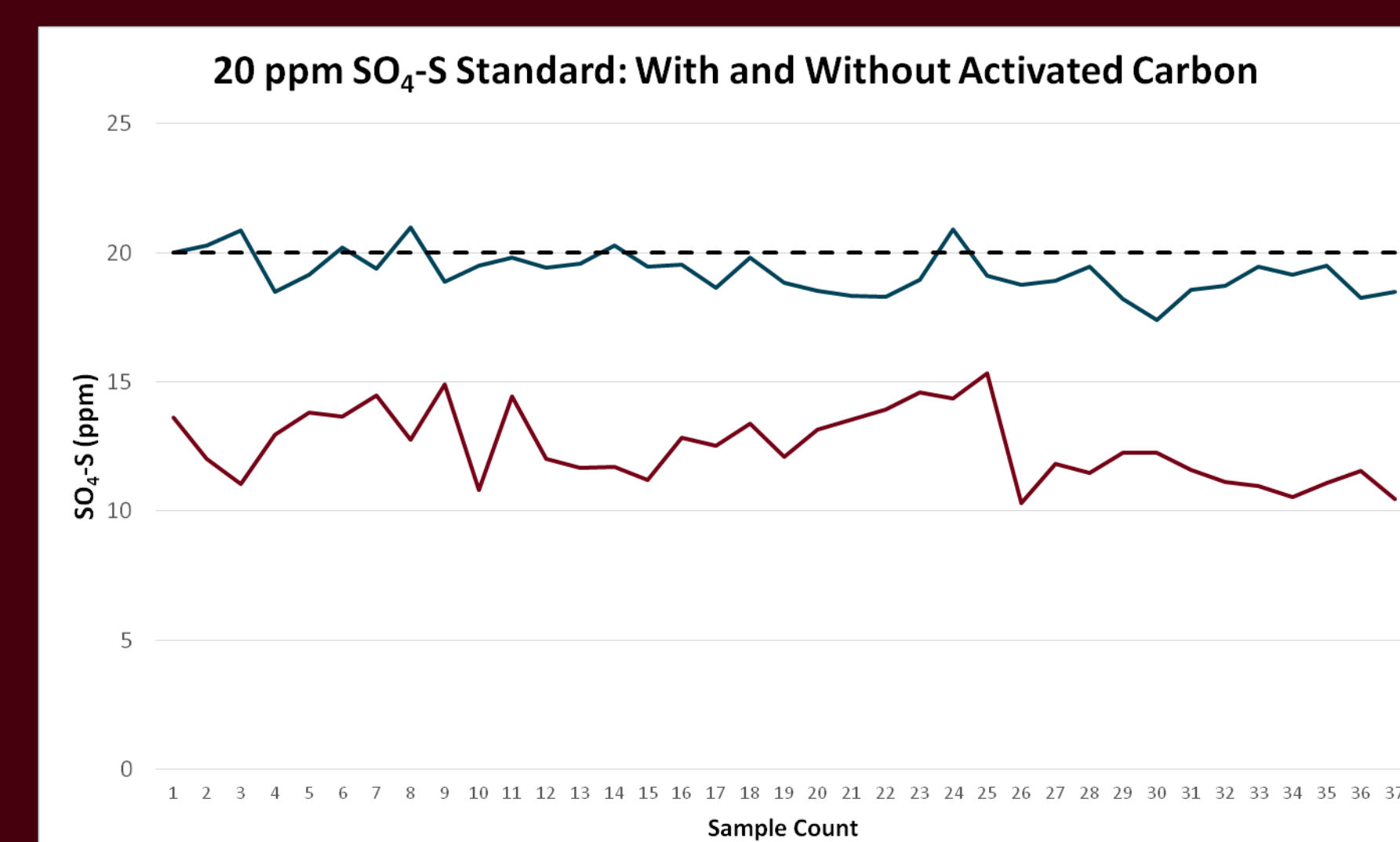
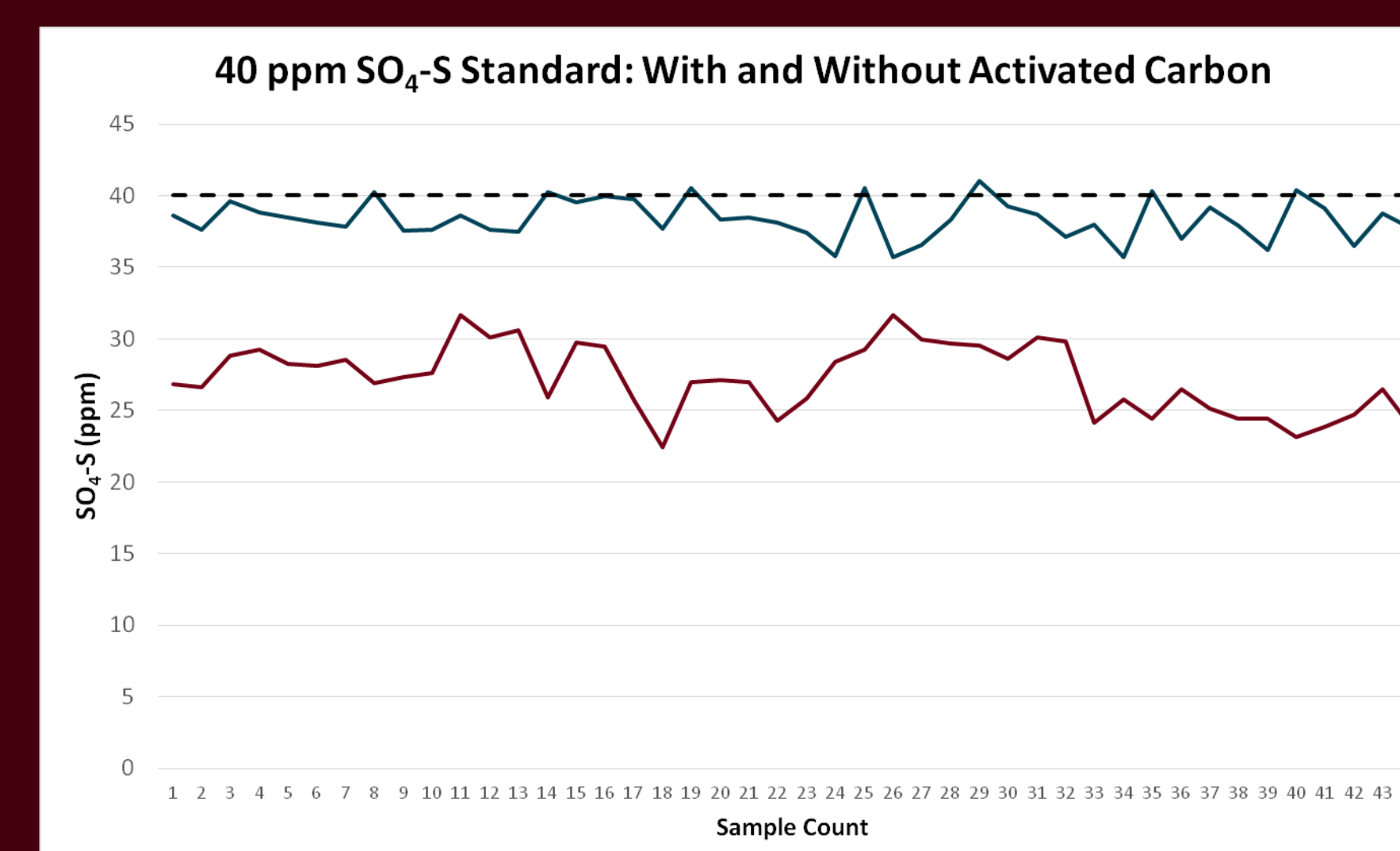


Figure 5. Mean Difference = 11.11 ppm



Figures 3-5.

Graphs showing sulfate-sulfur recoveries on three different standard concentrations with and without carbon addition.

— "X" ppm $\text{SO}_4\text{-S}$ standard: no added carbon
 — "X" ppm $\text{SO}_4\text{-S}$ standard: shaken with activated carbon
 - - Standard Concentration

Sources

- *Determination of Sulfate by Flow Injection Analysis*. QuikChem Method 12-116-10-1-D. Loveland, CO: Lachat Instruments, 2003.
- Franzen, D.W. "Sulfate-Sulfur." In *Recommended Chemical Soil Test Procedures for the North Central Region*. Columbia, MO: Missouri Agricultural Experiment Center, 1998.
- Rehm, G., and M. Schmitt. *Sulfur in Minnesota Soils*. U of MN Extension Publication FO-00794-GO. 1989.