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Application Note

High Throughput Analysis of Hydraulic Fracturing Waste Water

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INTRODUCTION

There are important organic and inorganic components to the testing of fracking related samples. Besides EPA methods such as SW-846 and 200.7, the focus here is the inorganic elemental analysis associated with fracking samples and how the increasing number of those samples can be better and more efficiently dealt with by using the CETAC *ASXPRESS® PLUS* rapid sample introduction system.

BACKGROUND ON FRACKING

Technique and Analytical Challenges

Hydraulic fracturing (fracking) is a relatively new method of extracting natural gas from dense shale deposits situated deep under low-permeable rock layers miles below the surface. Huge volumes of hydraulic fluid need to be pumped into wells to fracture the shale deposit and liberating the gas.

Although about 90% of this fluid is water, a variety of additives are used, including:

- Proppants such as sand or bauxite
- Gelling agents such as cellulose polymers and cross-linkers
- Foam additives
- Scale inhibitors such as ammonium chloride and phosphonates
- Corrosion inhibitors such as methyl and isopropyl alcohol
- Biocides
- Friction reducers such as light crude oil distillates
- Surfactants
- Acids such as HCl

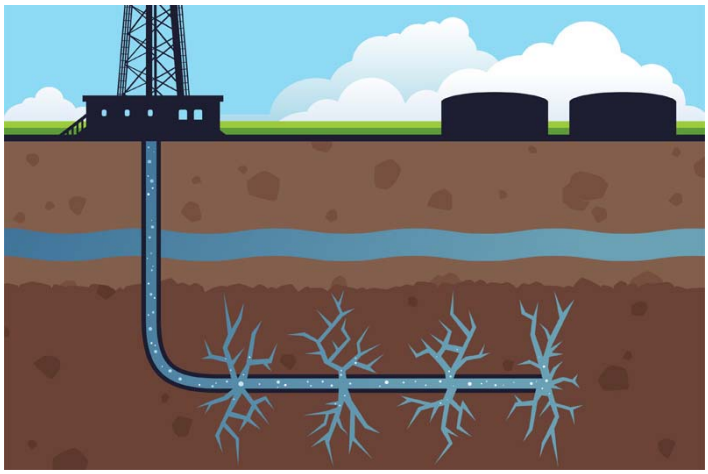

Figure 1: Schematic diagram of the fracking process

This process leads to environmental concerns about drinking water quality in the surrounding area and the tremendous amount of waste water produced.

Many samples are generated that need to be tested, for example, by quantifying the content of trace and ultra trace metals and metalloids.

Political Background and Progress on Legal Framework

The 2005 Energy Bill explicitly exempts fracking from federal review under the Safe Drinking Water Act (SDWA). Under this provision, drilling companies are under no obligation to make public the chemicals that they use, although many of them are recognized or suspected carcinogens. This current state of legislation is likely to change as time moves forward and the political climate evolves.

The U.S. Environmental Protection Agency (USEPA) is currently investigating cases of suspected contamination in towns located near fracking activities.

ASXPRESS PLUS – FUNCTION PRINCIPLE

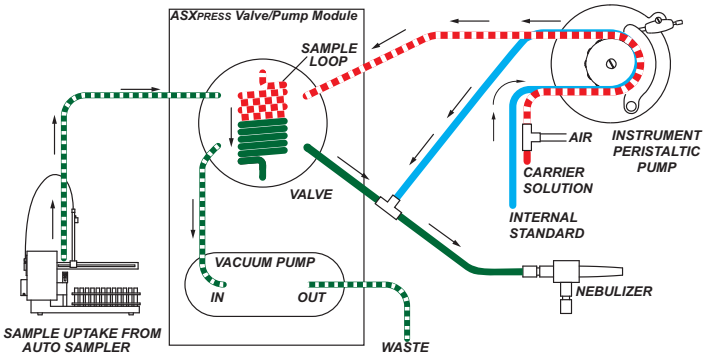

Figure 2: Function principle of the *ASXPRESS PLUS* system and connections.

Advantages and Features

- Robust coupling to any ICP-OES or ICP-MS system due to universal setup and GUI controlled software running without any need for an user intervention after installation.
- Increased efficiency due to shortened sample uptake / stabilization time.
- Minimized carryover and memory effects due to air bubble wash step and no exposition of the sample to peristaltic pump tubing.
- Constant flow to nebulizer (no fast uptake) to ensure plasma stability and easy in-line addition of internal standards.

ANALYTICAL METHOD

ICP-OES Conditions:

- Thermo iCAP 6000 series ICP-OES system with dual view configuration
- RF Power: 1150 W
- Nebulizer Gas Flow: 0.7 L/min
- Purge gas mode: Boost, pump rate: 45 rpm
- Humidified argon supply and *GE SeaSpray* nebulizer for high TDS matrices

Sample Matrix:

- 5 mL HCl + 2 mL HNO_3 for 50 mL sample
- ISTDs: Y @ 1.000 mg/L and In @ 10.000 mg/L
- CCB & CCV every 10 samples
- USEPA 6010B / 200.7 / fracking combined

ASXPRESS PLUS Configuration:

- 5 mL, 2 mm I.D. aqueous loop and 1.5 mm I.D. port sized aqueous valve
- Extra loop rinse:
  - Loop rinse delay: 4.0 s
  - Loop evacuation delay: 3.0 s
- Loop load: 10 s
- Equalization time: 2.0 s
- Time to evacuate probe: 2.0 s
- Rinse station fill: 11 s
- Pump timeout: 60 s
- *ASXPRESS* Firmware: 2.50

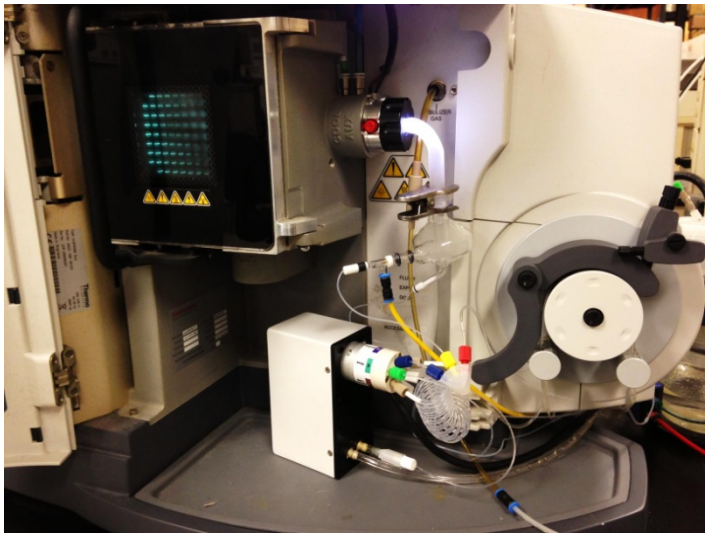

Figure 3: CETAC's *ASXPRESS PLUS* rapid sample introduction system coupled to a Thermo iCAP 6000 series ICP-OES system.

RESULTS

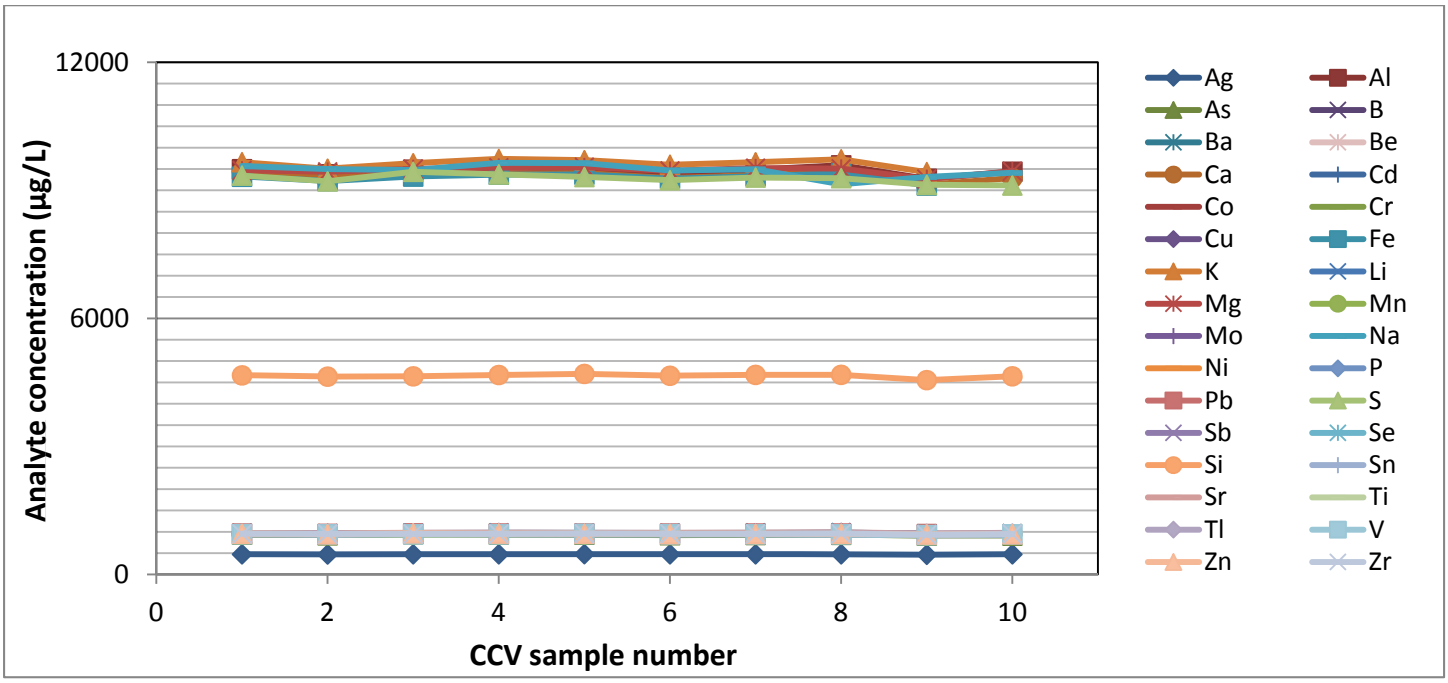

Figure 4: Found concentrations in 10 CCV samples recorded during analysis of fracking waste water showing the figures of merit of the developed system and method.

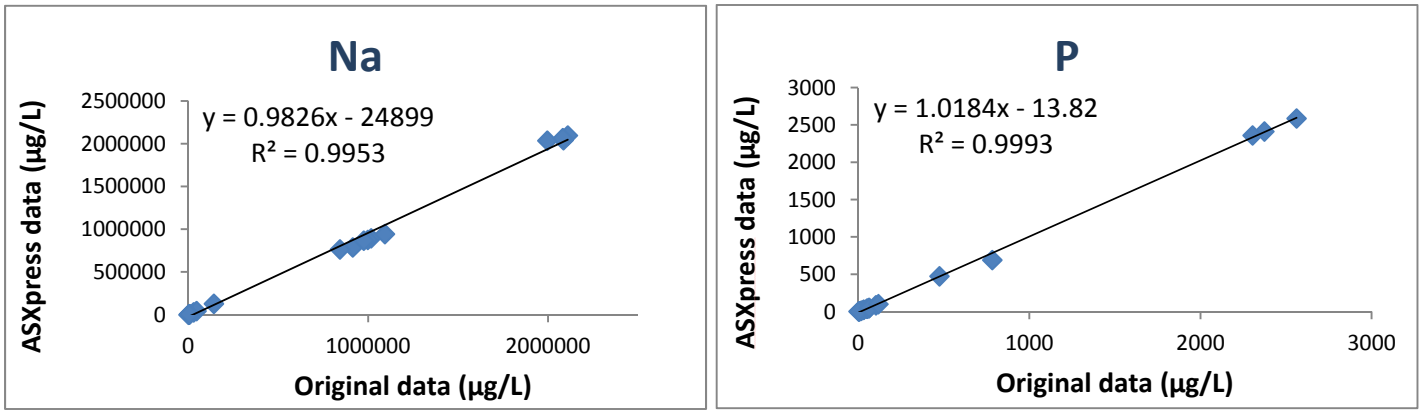

Figure 5: Validation of the original and the *ASXPRESS* accelerated method for selected elements of interest.

CONCLUSION

The original method total analysis time of 4 min 27 s could be reduced to 2 min 45 s using the newly developed method resulting in an efficiency increase of 38% without changes to the original method (0 s plasma stabilization time, 0 s rinse time, only 20 s flush time).

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