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Rare Earth Element Determination in Heavy Crude Oil by USN-ICP-MS After Digestion Using Single Reaction Chamber (SCR) Technology

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INTRODUCTION

Elemental analysis of heavy crude oil is important for a variety of reasons including region of origin, maturity, migration, type, and identification of potential challenges that could arise during the refining process. There are numerous challenges with analysis of heavy crude oil with most being centered on the sample prep process due to the complexity of the sample matrix. Microwave digestion has been used to overcome some of these challenges, however, traditional rotor based systems still face problems with limits of quantification due to sample size limitations, temperature and pressure capabilities, high residual organic contact (ROC), and high residual acidity. The revolutionary Milestone's Single Reaction Chamber (SRC) microwave digestion incorporates all of the benefits of closed vessel microwave digestion while making sample preparation fast, easy, effective, and the highest quality. SRC operates at very high temperature and pressure (300 °C and 199 bar respectively), thus complete digestion of even the largest sample size and most reactive samples can be achieved. The sample can be weighed directly into a disposable glass vial, eliminating the need for acid cleaning and vessel assembly. The paper "*Rare earth element determination in heavy crude oil by USN-ICP-MS after digestion using a microwave-assisted single reaction chamber*", Druzian et al, *J. Anal. At. Spectrom*., 2016, 31, 1185, provide evidence of the digestion quality obtained with Milestone's ultraWAVE in comparison with a rotor based echnology.

ABSTRACT

In this work a method for rare earth element (REE) determination by inductively coupled plasma mass spectrometry (ICP-MS) with an ultrasonic nebulizer (USN) was proposed after heavy crude oil digestion by microwave-assisted wet digestion (MAWD) using a single reaction chamber (SRC) system. Operational conditions of the MAWD-SRC method, such as sample mass (from 250 to 1000 mg), type and volume of digestion solutions, temperature achieved during digestion (from 200 to 270 ºC) and microwave irradiation time (35, 40, 45 and 50 min) were investigated.

Using optimized digestion conditions, the carbon concentration and residual acidity in digests were 2345 mg L^{-1} and 14.6%, respectively.

Since the acidity was low and dilution was not required after digestion by the MAWD-SRC method, lower limits of quantification (LOQs) were obtained (0.1 up to 2 ng g^{-1}) and the determination of some analytes present in very low concentration was possible. It was possible to digest a relatively high crude oil mass (up to 1 g) using 8 mL of 14.4 mol L^{-1} HNO_3 in just 40 min, which can be considered as an important aspect taking into account the difficulties involved in heavy crude oil digestion for further REE determination by ICP-MS. The accuracy was evaluated by analyte spike and also by comparison of results obtained by MAWD-SRC with those using conventional MAWD and also by microwave-induced combustion. Suitable recoveries were obtained for all analytes (94 to 110%) and no statistical difference was observed between the results obtained by MAWD-SRC and those using other methods.

INSTRUMENTATION

Figure 1. Milestone's ultraWAVE

An ultraWAVE digestion system, based on a single reaction chamber (SRC) equipped with five quartz vessels (total volume of 40 mL) was used for crude oil digestion (MAWD-SRC method). The SRC features a large 1 Liter pressurized stainless steel reaction chamber, which also serves as the microwave cavity.

Samples are weighed into auto sampler-type vials with the appropriate digestion acid and loaded into a rack. The rack is loaded into the chamber, which is then sealed and pre-pressurized with nitrogen to 40 bar prior to microwave heating. Pre-pressurization prevents splashing or boiling of the sample solutions, which prevents cross contamination or loss of volatiles. Because the pressure in the chamber increases with sample temperature, boiling never occurs.

SRC can operate at very high temperature and pressure – up to 300 ºC and 199 bar, which enables the complete digestion of every sample-type. The higher pressure capability of a SRC allows higher sample weights to be digested. With SRC, different sample types can be run simultaneously – there is no need to "batch" digestion runs into identical sample types as with traditional microwave digestion. The SRC also requires less digestion acid, which lowers the reagent blank and the ultra-pure reagent costs.

On completion of the program, the chamber automatically vents and the rack is removed. Samples are diluted to volume in the vials, ready for aliquoting and measurement.

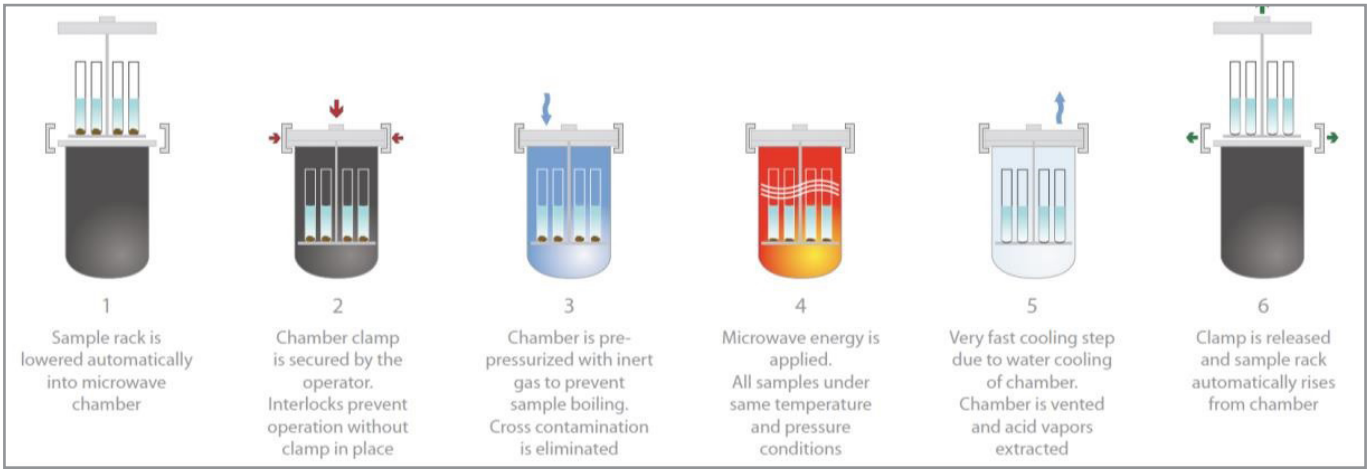

Figure 2. – Workflow of the ultraWAVE

RESULTS AND CONCLUSIONS

To the best of the authors' knowledge, this is the first method able to digest 1 g of crude oil using wet digestion in closed vessels. This fact allowed the determination of those elements present at very low concentration in crude oil. This article is another clear evidence on the reliability of SRC technology.

The ultraWAVE ensures complete digestion of all sample sizes, while the rotor-based technology shown a much higher carbon concentration due to the low digestion temperatures attained. Digestion efficiency using the ultraWAVE was higher, providing low residual carbon and acidity in digests, which are important parameters to avoid interferences during the determination of REE by ICP-MS.

The ultraWAVE combined good performance, safety and relatively high sample throughput since the digestion could be performed in multiple positions in as little as 40 minutes.

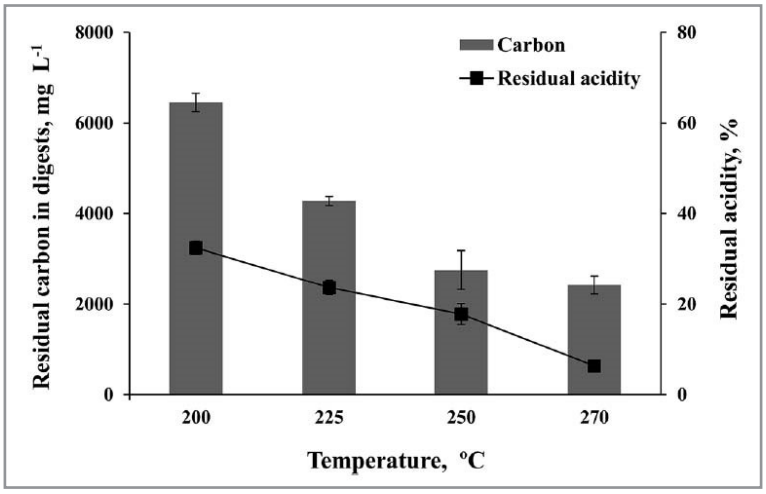

Figure 3. Effect of temperature in the digestion of crude oil "A" by MAWD-SRC (mean and standard deviation, n=3). Digestion was performed using 750 mg, 6 mL of 14.4 mol L^{-1} HNO_3 as the digestion solution and a microwave irradiation time of 40 min.

Finally, LOQs (limit of quantitation) were lower than those obtained using the conventional microwave system, as well as residual acidity.

REFERENCES

Abstract, graph and main part of the text of this report is taken from: "Rare earth element determination in heavy crude oil by USN-ICP-MS after digestion using a microwave-assisted single reaction chamber" Druzian et al, *J. Anal. At. Spectrom*., 2016, *31*, 1185 (https://doi.org/10.1039/C6JA00050A).

FURTHER READINGS

To learn more about ultraWAVE and other related topics, feel free to visit these websites:
Milestone ultraWAVE – SRC Technology: https://www.milestonesrl.com/products/microwave-digestion/ultrawave
Milestone srl: http://www.milestonesrl.com

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