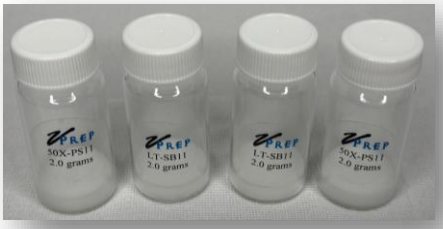
# OIL SOLIDIFICATION:
# Analysis with WD-XRF under Vacuum

Traditional oil analysis methods, although seemingly simple and routine, have several inherent challenges. Leakage can occur while the sample is in the autosampler or during analysis, leading to potential contamination, expensive repairs, and downtime. Settling of particulates may cause inconsistent results or deviations from nominal values, affecting measurement reliability. Some XRF instruments configured with only vacuum atmosphere, lacking helium capability, limit analyses to solid samples. However, the optional configuration for helium analysis needed to measure liquid samples introduces additional costs and sourcing challenges. Furthermore, the sensitivity for light elements such as Na, Mg, and Al is often reduced in comparison to vacuum, potentially compromising detection levels. These drawbacks highlight the need for alternative preparation techniques for oil sample analyses by XRF.

## STANDARDS AND INSTRUMENTATION

The calibration for oil solidification analysis was established using oil standards (SFNVM012) provided from ASI Standards (www.asistandards.com) and wax blends from VPrep Corp (www.vprepcorp.com). A 1:1 ratio of oil-to-wax was prepared and solidified. Procedure on oil solidification are demonstrated here: XRF Videos | VPrep Corp

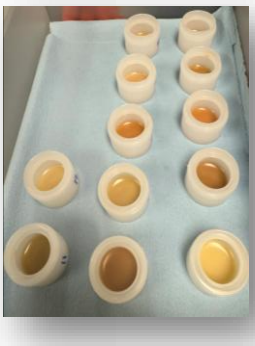




Measurements were conducted using the benchtop Supermini200, featuring a 200W Pd target X-ray tube. The Kα line was analyzed for all elements, with a total measurement time of under 5 minutes. Heavy elements—V, Fe, and Ni—were measured using a LiF(200) analyzing crystal and a scintillation counter, while lighter elements, such as S, were analyzed with a PET crystal and a proportional counter.

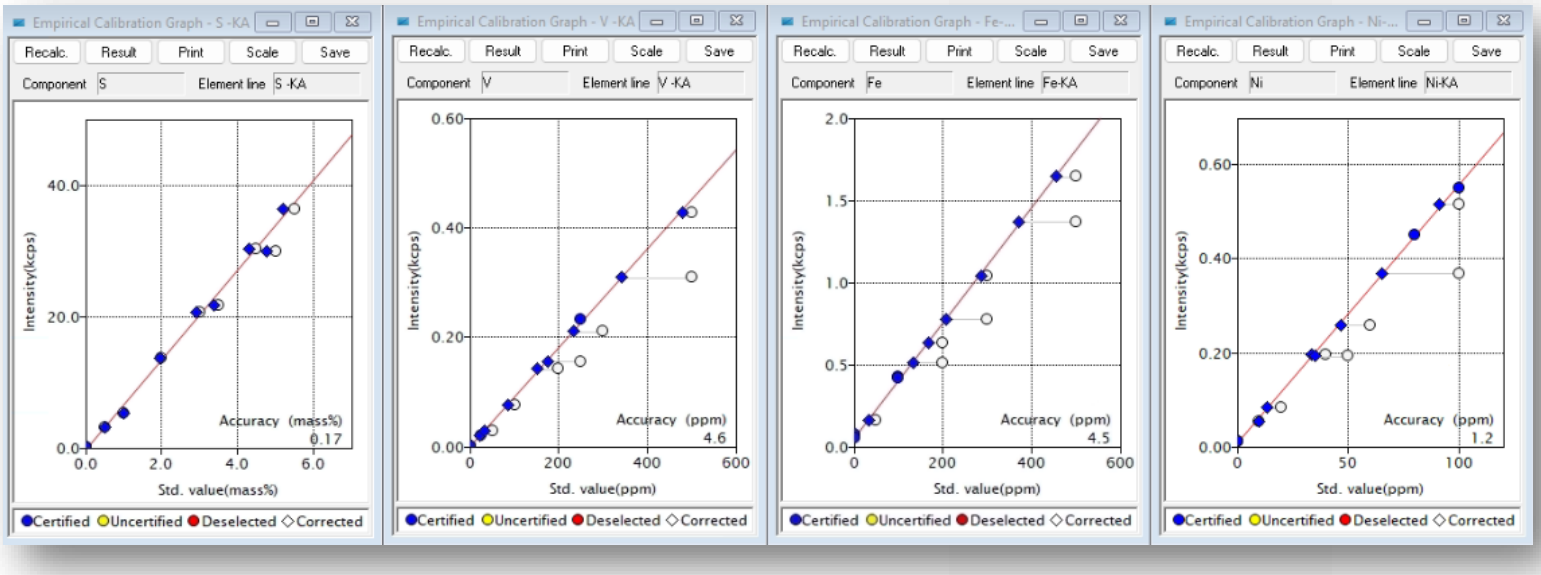
## CALIBRATION CURVES

Representative calibration curves (shown below) illustrate the accuracy of the elemental analysis. A simple absorption/enhancement correction was applied to all calibrations to compensate for the drastic variation in S content of the oil matrix. Theoretical alphas, or a full fundamental parameters method could be applied for more complicated sample matrices but were not necessary for this application.

| | Name | Note | S | V | Fe | Ni | CH2 | C6H1005 |
|---|---|---|---|---|---|---|---|---|
| | | | mass% | ppm | ppm | ppm | mass% | Additive ratio |
| 1 | 1 | | 0 | 0 | 0 | 0 | Balance | 1.00000 |
| 2 | 2 | | 0.5 | 501 | 300 | 10 | Balance | 1.00000 |
| 3 | 3 | | 1 | 25 | 500 | 100 | Balance | 1.00000 |
| 4 | 4 | | 0 | 250 | 100 | 80 | Balance | 1.00000 |
| 5 | 5 | | 2 | 101 | 200 | 40 | Balance | 1.00000 |
| 6 | 6 | | 2.5 | 400 | 400 | 5 | Balance | 1.00000 |
| 7 | 7 | | 3 | 300 | 0 | 60 | Balance | 1.00000 |
| 8 | 8 | | 3.5 | 200 | 500 | 0 | Balance | 1.00000 |
| 9 | 9 | | 0 | 0 | 100 | 100 | Balance | 1.00000 |
| 10 | 10 | | 4.5 | 250 | 300 | 50 | Balance | 1.00000 |
| 11 | 11 | | 5 | 500 | 200 | 20 | Balance | 1.00000 |
| 12 | 12 | | 5.5 | 51 | 50 | 100 | Balance | 1.00000 |



Representative solidified oil calibration standards prepared simultaneously



## REPRODUCIBILITY

To assess reproducibility, standards 150 and 152 were analyzed in six (6) replicates using the calibrated method. The relative standard deviation (%RSD) for all measured elements was below 5%, confirming the high precision and stability of the SM200 system. This level of reproducibility ensures consistent results across multiple analyses, which is critical for quality control in steel production.


**SUPERMINI200**

## ELEMENTAL ANALYSIS TESTING SERVICES

VPrep Corp offers fast elemental analysis on a variety of materials by non-destructive direct wavelength-dispersive X-ray fluorescence (WD-XRF) for a wide elemental range. The Rigaku Supermini200 system is our choice for XRF analysis, offering precise elemental identification and quantification for materials such as cement, metal alloys, geological, plastics, pharmaceuticals, oils, and many more. We offer methods for full scans, semi-quantitative, and quantitative to cover all material types.

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