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Sulfur Analysis in Diesel Fuel

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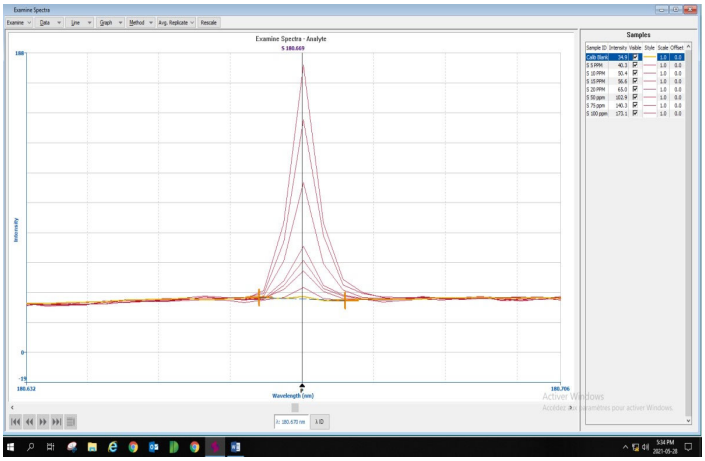
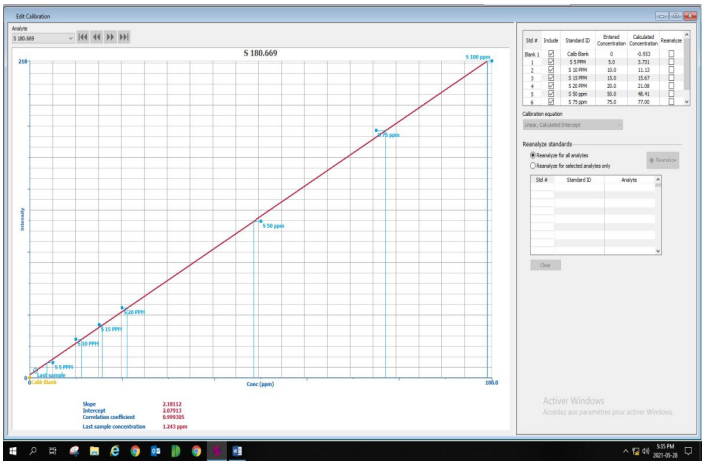
Continuous improvement for Sulfur measurement in Engine Fuel by ICP

In addition to the limitation of Canada on Sulfur content in diesel fuel, sulfur forms particulates that are a primary contributor to air pollution and the cause of harmful corrosion in the engine. Because of the criticality of Sulfur content in low concentration, it becomes necessary to seek more precision for this measurement.



Method improvement has different steps, and some parameters of ICP (Inductive Coupled Plasma) such as pump speed and gas flow must change to make the method suitable for the application. Choosing the best wavelength for Sulfur is one of the most critical parameter for accuracy.

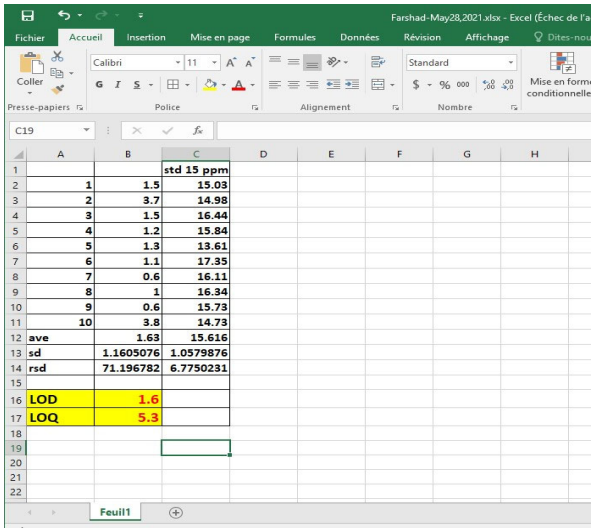


The best wavelength means low noise in the blank, clear, and sharp intensity in Standard solutions. Sulfur at 181.975 nm, at 182.563 nm and at 189.965 nm, had been tested with blank, 5 and 10-ppm standards. We

found that intensity is too close each other and gave less precision.

However, the pick of the Sulfur at **180.669 nm** wavelength in Standard solutions 5 and 10 ppm has a good intensity and noise in the blank is very low. We choose this wavelength as the best one for low concentration Sulfur in Diesel Fuel.

1. Standards and calibration curve: To enabling us to measuring the lowest Sulfur concentration in the fuel samples; 5, 10, 15, 25, 50, 100 Standards Sulfur solutions in fuel (same matrice) are used. We prepared all the standards at the same day, same solvent.
2. Eliminate all cross contamination: Using Premisolv to wash the mixer and prob rejected to the waste container.
3. Sample measurement: Used 4 times reading to get high accuracy in measurement.
4. Washing time, and delay time before and after measurement: The times increased to eliminating any carry over



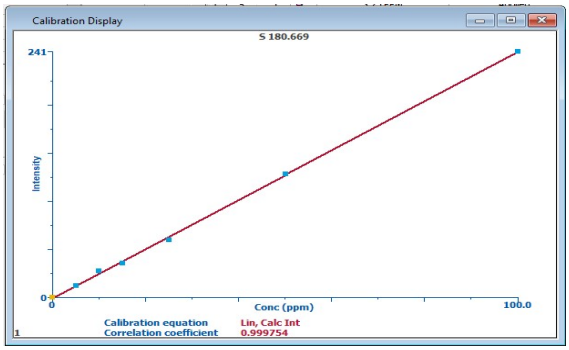
	A	B	C
1			std 15 ppm
2	1	1.5	15.03
3	2	3.7	14.98
4	3	1.5	16.44
5	4	1.2	15.84
6	5	1.3	13.61
7	6	1.1	17.35
8	7	0.6	16.11
9	8	1	16.34
10	9	0.6	15.73
11	10	3.8	14.73
12	ave	1.63	15.616
13	sd	1.1605076	1.0579876
14	rsd	71.196782	6.7750231
15			
16	LOD	1.6	
17	LOQ	5.3	

1. LOD and LOQ evaluation:

As we have base line noise so, Signal-to-Noise applied to calculate the LOD (Limit of detection) and LOQ (Limit of quantification); by using the slope of the calibration curve, and injecting 10 times Blank solution.

LOD = 1.6 ppm LOQ = 5.3 ppm

We suggested to considering our **LOQ about 6 ppm**.



2- Linearity

In different days Min. six Std in different concentration from 5 too 100 ppm used for calibration Curve (by focusing on low std concentration such as 5, 10, 15, 25 ppm and considering the 15 ppm as standards limit), and the Correlation coefficient was bigger than 0.999.

The results more than **100 ppm** for the sample will be less accurate; we keep focussing on low concentrations.

3- Precision and reproducibility

On May 28, 2021, Std 15 ppm, which it is considered as limit by government, injected 10 times and RSD calculate, RSD%=6.78

In different days, same samples (different days, different std prep, different samples prep) was measured and differences are acceptable, less than 10%.

4- Accuracy

To validate our results, we Used Std 10 ppm, of VHG, as a second standard supplier as QC1 .

	VHG Std 10 ppm
1	10.5035
2	10.641
3	9.686
4	10
5	10.781
6	10.483
7	10.429
8	11.133
9	10.24
10	10.257
ave	10.41535
sd	0.404483488
RSD%	3.88

First QC 1 (Std VHG 10 ppm) result:
Concentration= 9.615 ppm, Recovery= 96.15%
QC2 Std SCP 15 ppm passed.
Ave of 10 consecutive injection of Std 10 ppm VHG (as sample) =
Concentration= 10.42 ppm
RSD%= 3.88

Conclusion:

Sulfur at the wavelength of 180,669nm gives an RSD of 3.88%, a linearity of 0,999% and a LOQ of 6ppm. Results with concentrations of 10 to 25 ppm are more accurate. Moreover, the new method has been validated with standards from two different supplier to confirm Precision, Reproducibility and Accuracy

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