

An Update on Methods Correlation Development
for Produced Water
Total Oil and Grease (TOG) Determination

SENAI Innovation Institute for Green Chemistry

Produced Water Workshop

INSTITUTO SENAI
DE INOVAÇÃO QUÍMICA VERDE

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INSTITUTO SENAI
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We are an applied research institute with the mission of contributing to the improvement of the Brazilian Industry through the exchange of research, development, innovation and technological transfer

An Update on Methods Correlation Development for Produced Water Total Oil and Grease (TOG) Determination

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PETROBRAS



Contextualization

- Total oil and grease (TOG) in production water is extremely important for companies in the oil sector;
- Guiding parameter for the destination that these waters will receive, which might cause irreversible environmental damage;
- In Brazil, the reference method for TOG determination is gravimetric, described by Standard Methods 5520B;

Contextualization

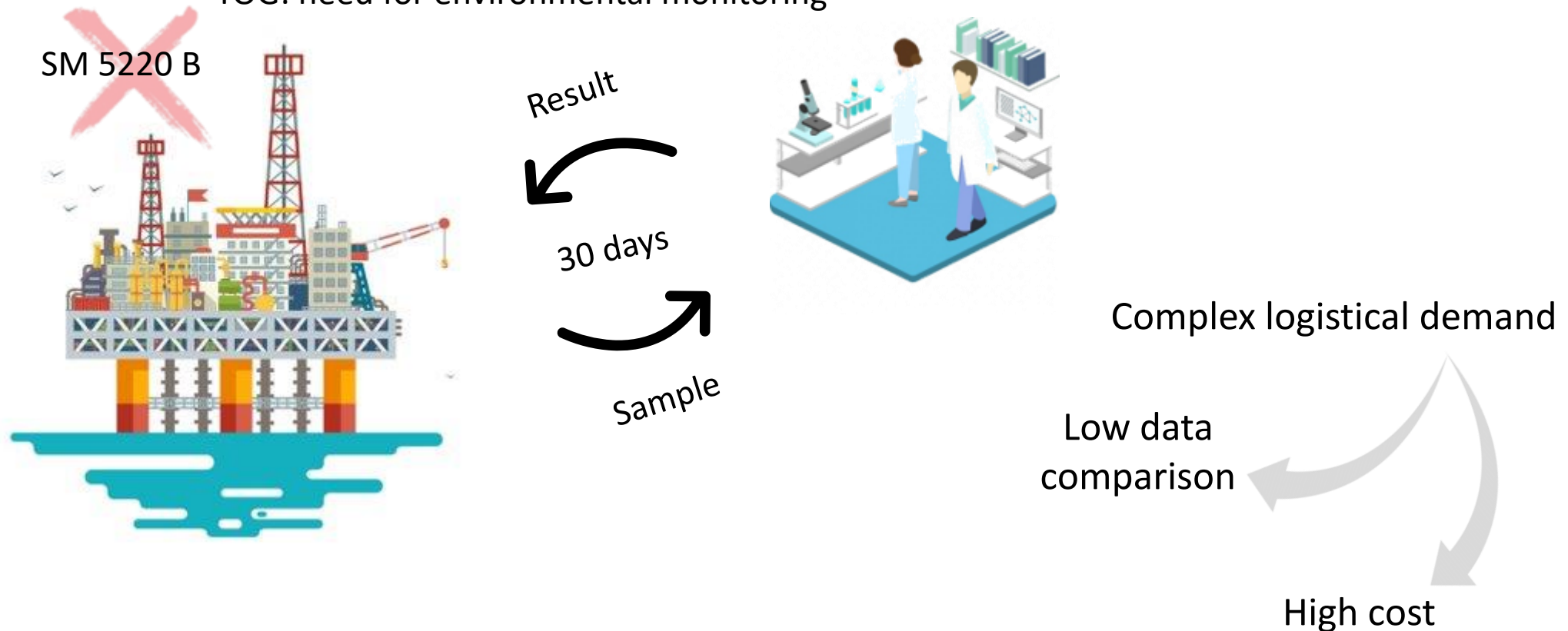
- ⊘ Large volume of organic solvent
- ⊘ Cannot be performed offshore
- ⊘ Not sustainable



Contextualization

Brazil: Legal requirements established by law CONAMA 393/2007

TOG: need for environmental monitoring



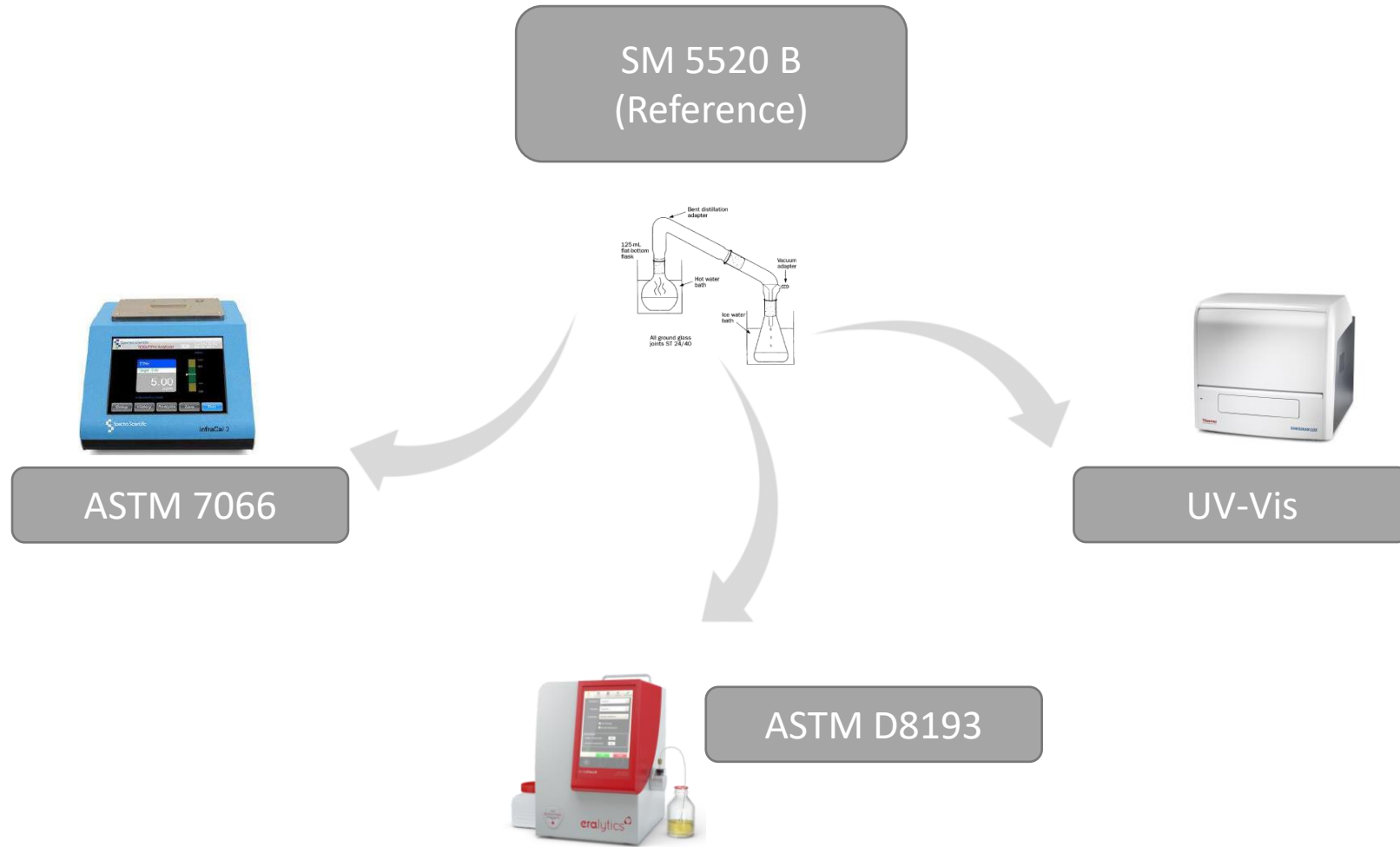


Objective

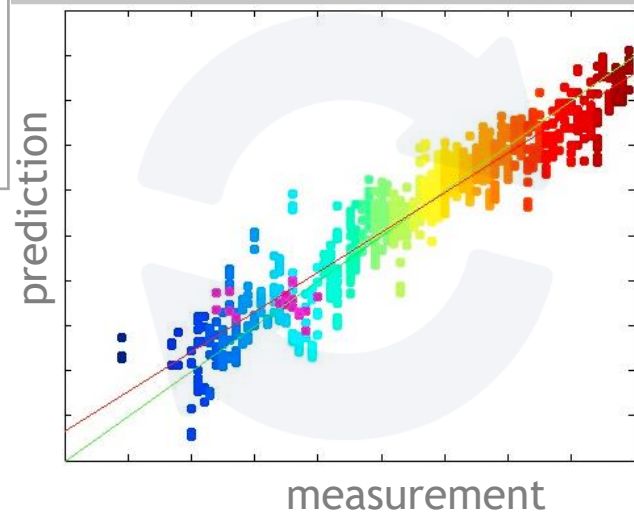
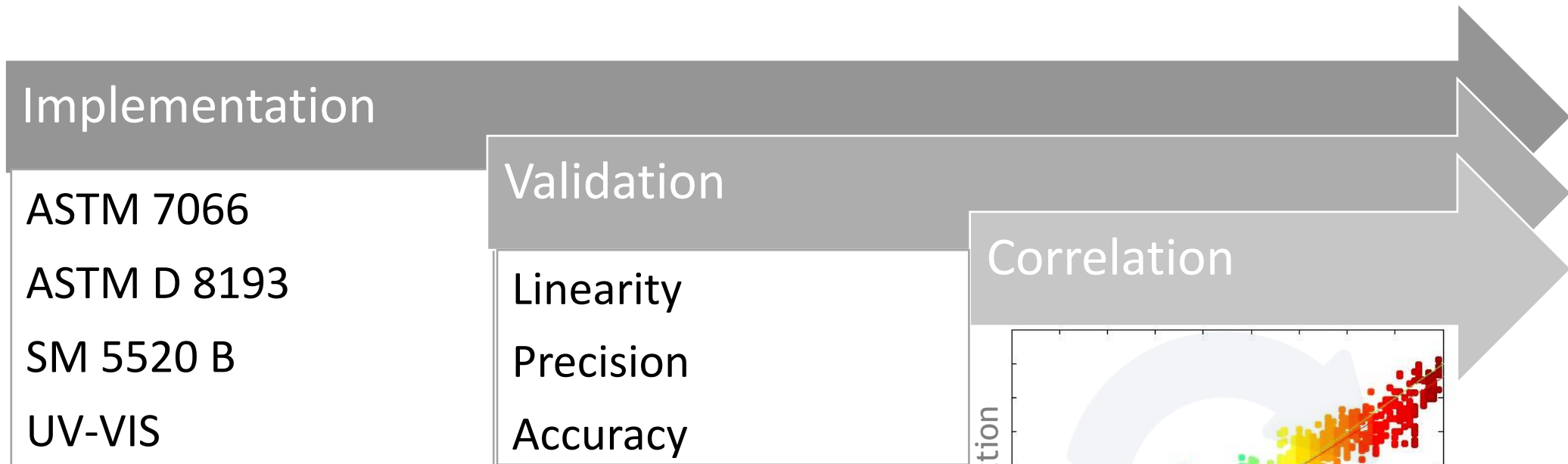


This research project aims to establish correlations between SM 5520B and alternative methods involving spectroscopic techniques, such as UV-Visible and Infrared Spectroscopy (ASTM D8193 and ASTM 7066).

Methodology



Methodology





Results - Implementation

- ✓ Methodologies focus on different chemical classes
- ✓ Standards indicate different substances for calibration
- ✓ Difficult correlation

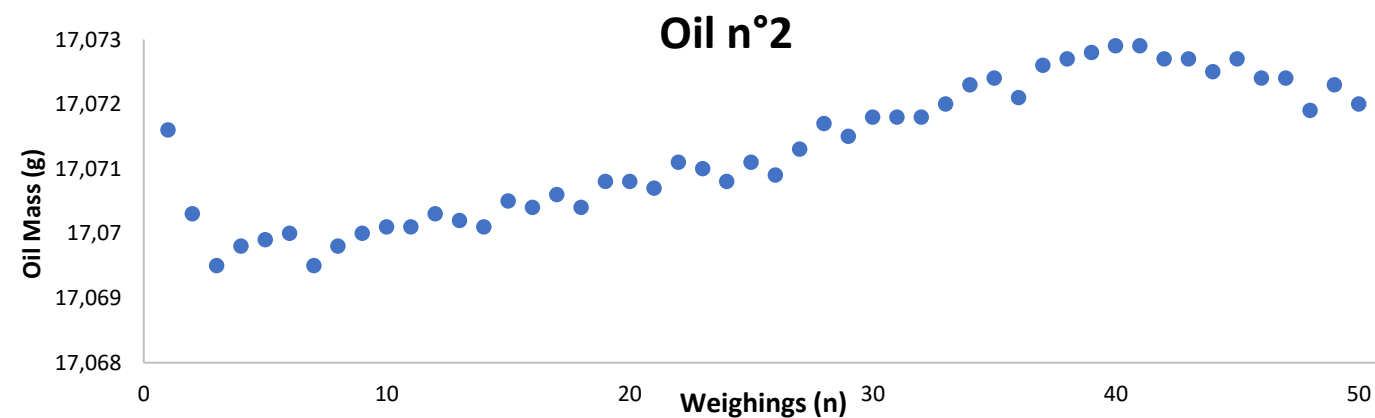
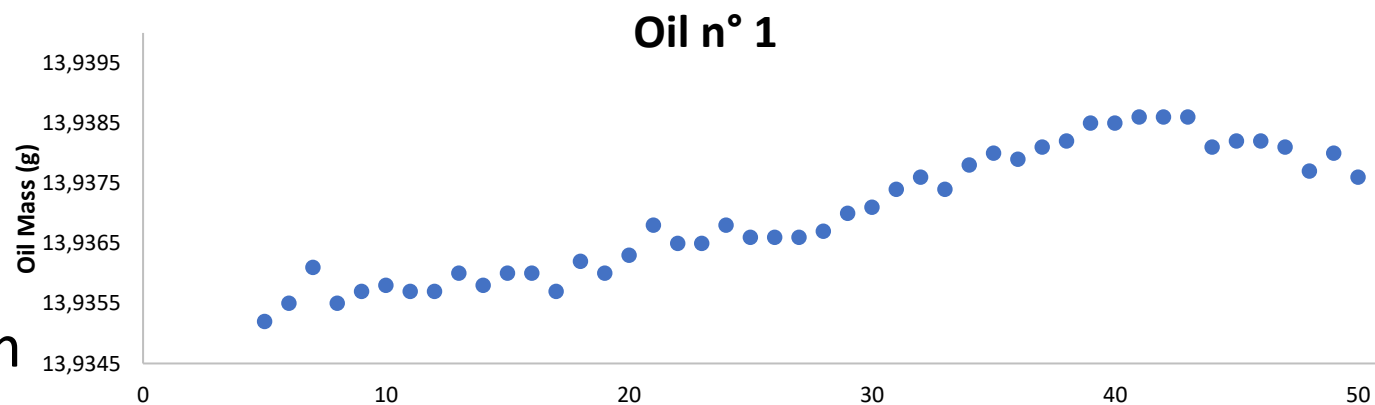
Results - Implementation

Oil-in-water standard development

🎯 Oil depletion

🎯 Elimination of volatiles with vacuum

🎯 Reproducibility in oil weighing



Results - Implementation

Oil-in-water standard development



Oil

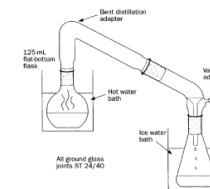


Depletion



Oil in water samples

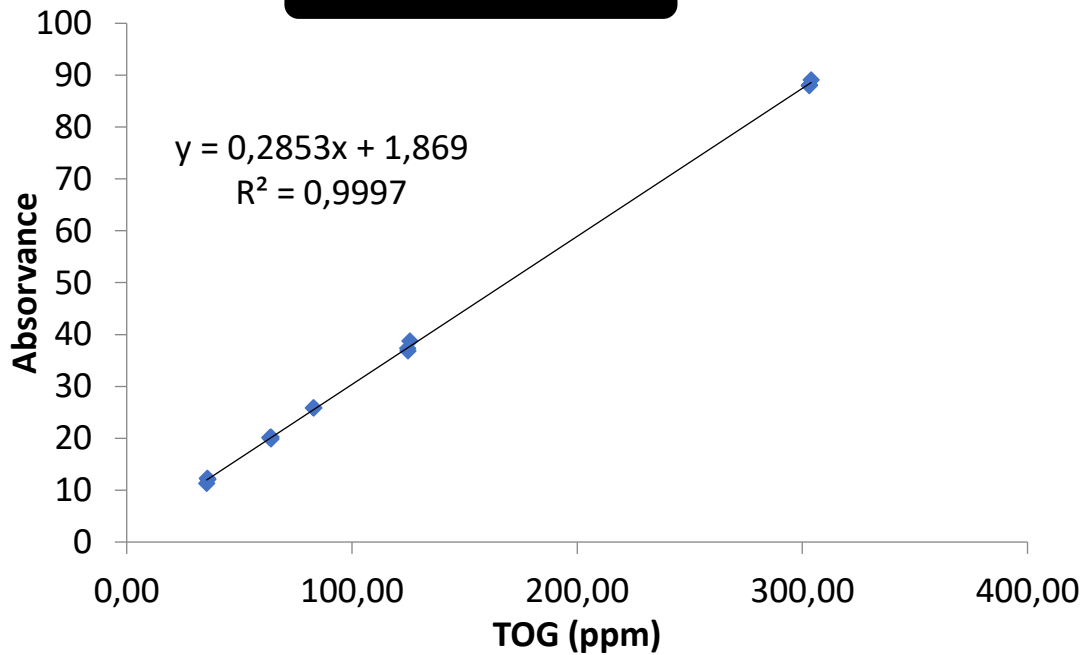
Salinity 3.5%; pH 2



Results - Validation

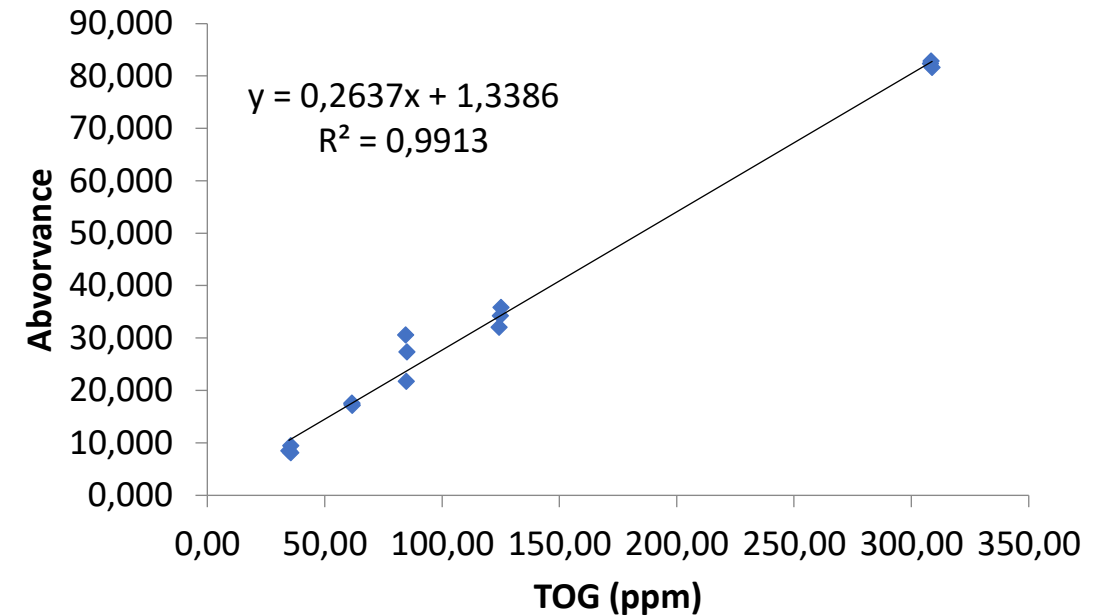
Linearity

Oil no. 1



ASTM D 8193

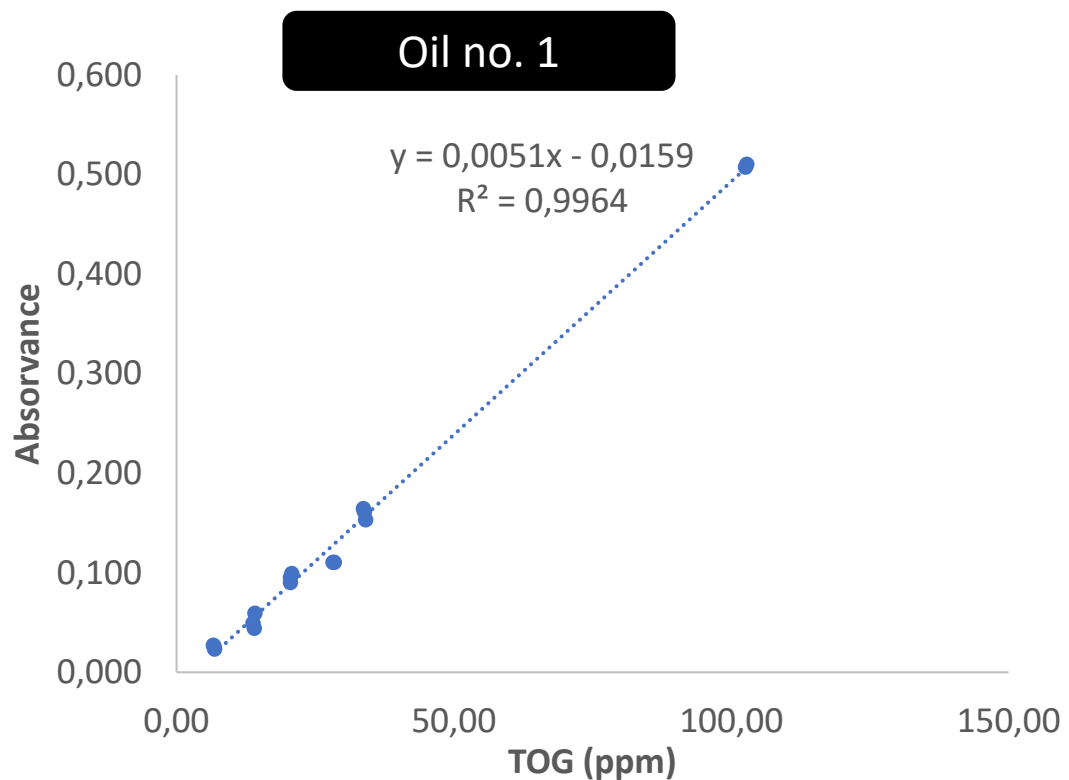
Oil no. 2



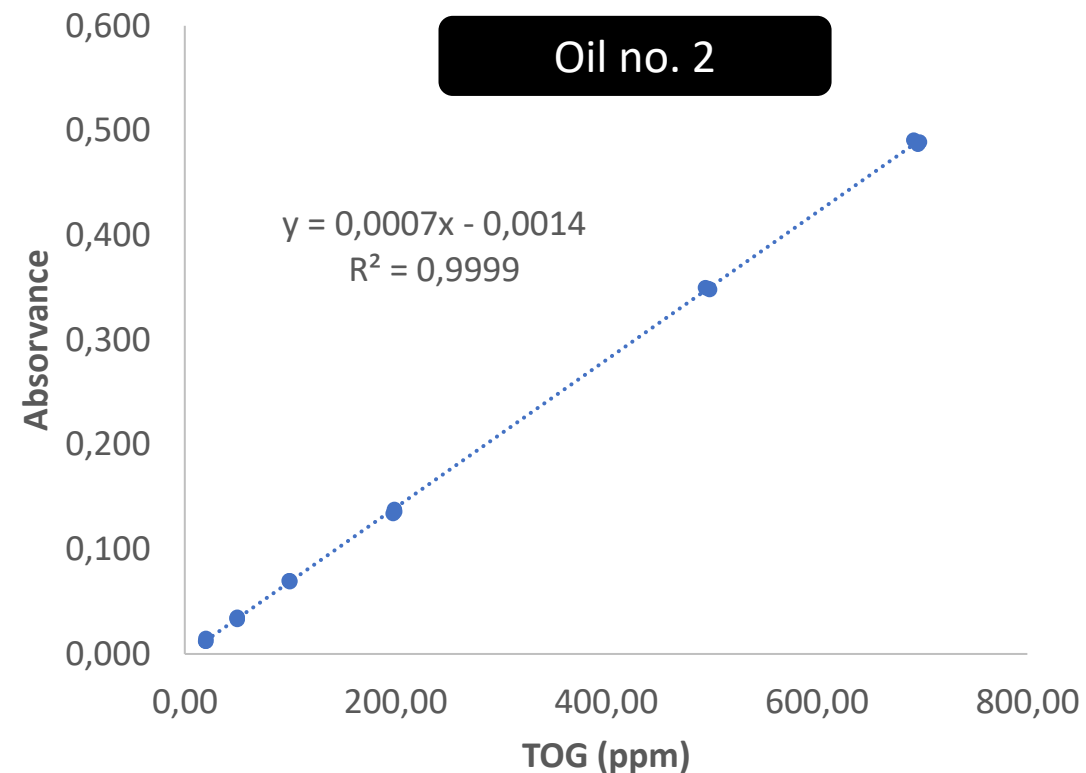
Normal, homoscedastic and independent distribution of the data
(Statistical evaluation: Tests: Shapiro-Wilk, Breusch-Pagan and Durbin-Watson)

Results - Validation

Linearity



UV-VIS



Normal, homoscedastic and independent distribution of the data
(Statistical evaluation: Tests: Shapiro-Wilk, Breusch-Pagan and Durbin-Watson)

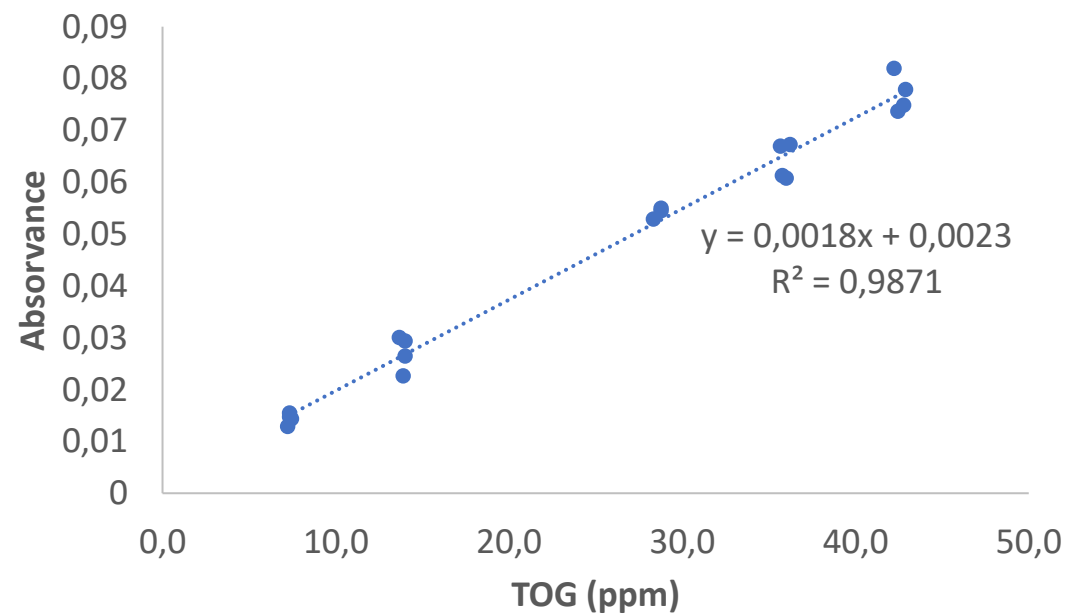
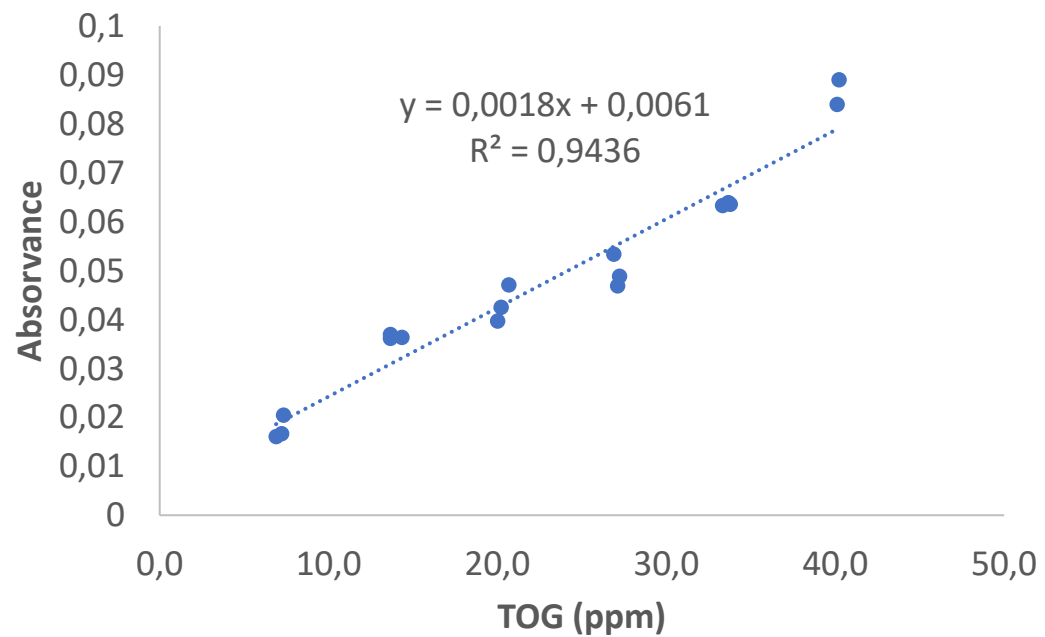
Results - Validation

Linearity

Oil no. 1

ASTM 7066

Oil no. 2



Results - Validation

Repeatability

Oil no. 1

SM 5520 B

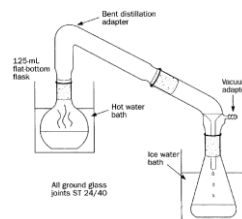
Oil no. 2

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	15	82.06	5.2
High	100	70.43	5.1

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	15	66.82	3.0
High	100	64.68	9.4



Results - Validation

Repeatability

Oil no. 1

ASTM D 8193

Oil no. 2

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	35	99.74	2.3
High	300	99.86	0.9

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	35	101.34	0.03
High	300	100.00	0.07



Results - Validation

Repeatability

Oil no. 1

ASTM 7066

Oil no. 2

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	7	114.59	9.80
High	30	126.48	8.05

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	7	100.9	18.11
High	30	110.6	22.22



Results - Validation

Repeatability

Oil no. 1

UV-VIS

Oil no. 2

Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	6	91.51	2.30
High	47	87.09	2.91

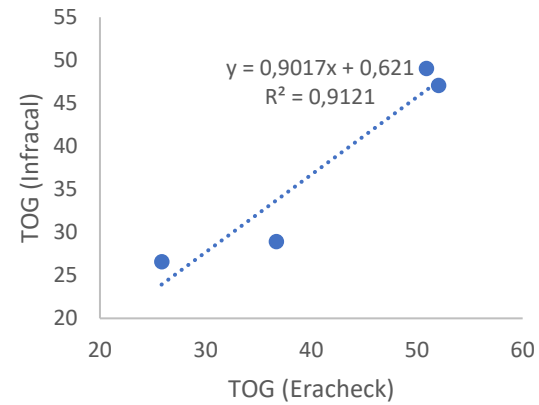
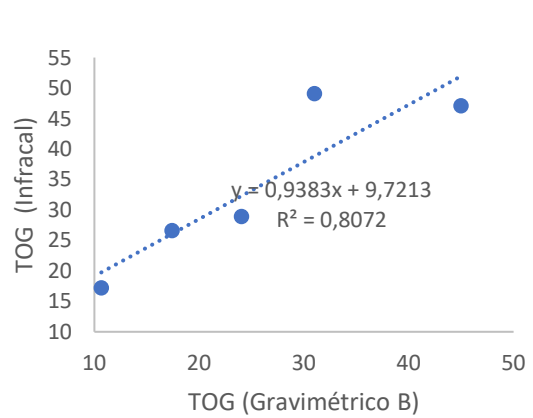
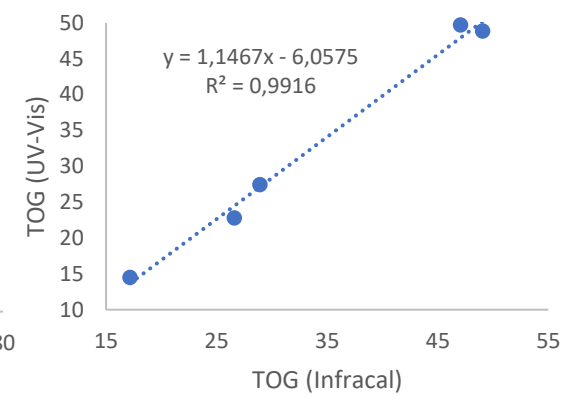
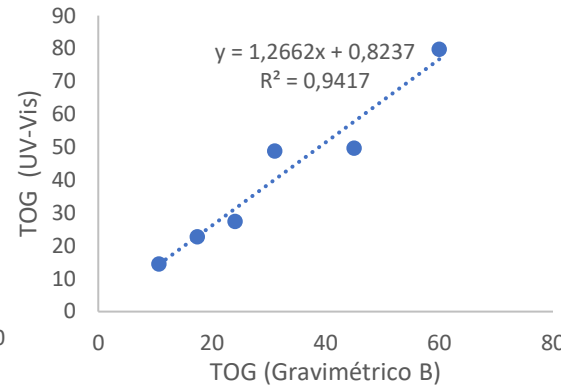
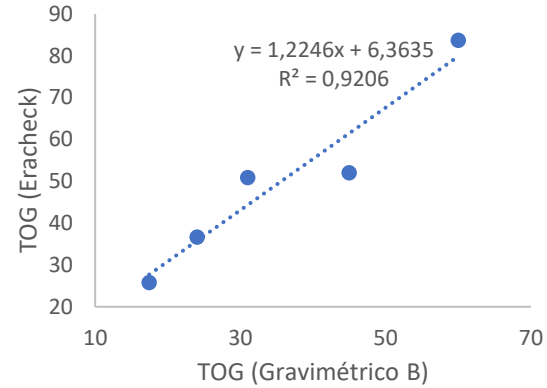
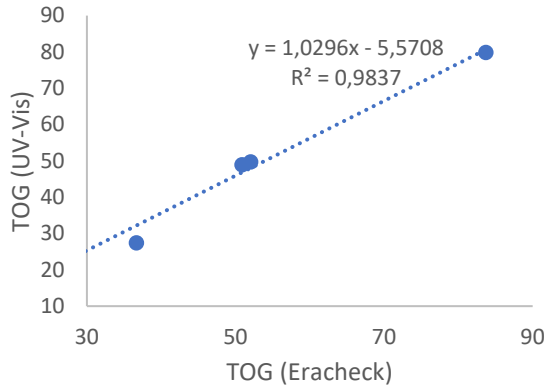
Repeatability Evaluation

Range	Concentration (mg.L ⁻¹)	Recovery(%)	Rel. Std. Dev (%)
Low	6	136.16	0.02
High	47	104.88	0.05



Correlation Models

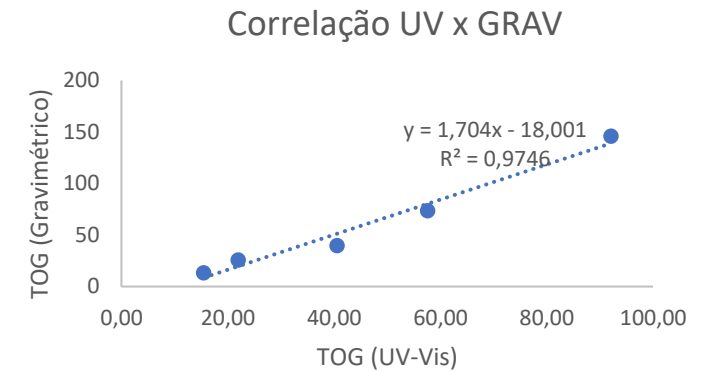
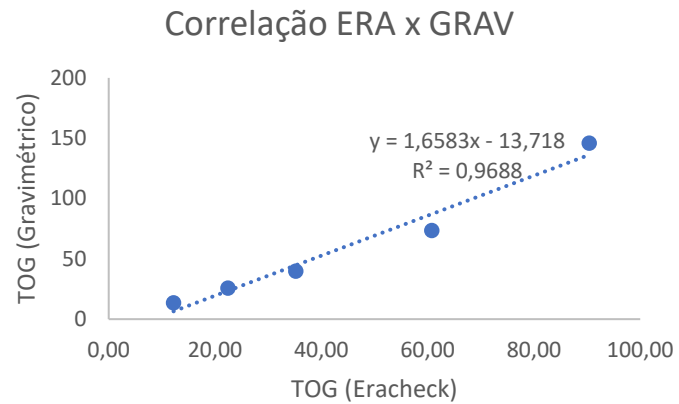
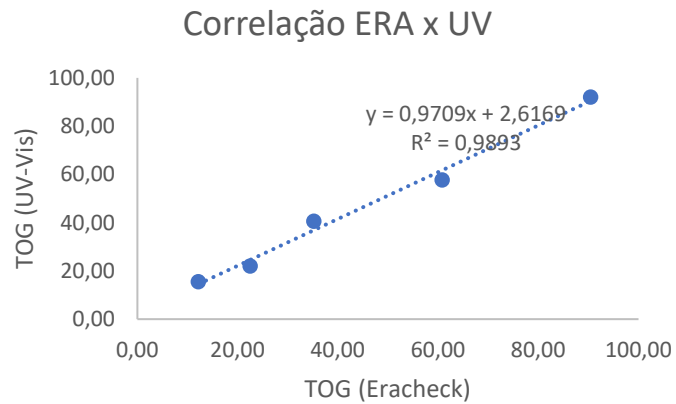
Oil no. 1



	SM 5520 B	ASTM D 8193	UV-VIS	ASTM 7066
SM 5520 B	1,000			
ASTM D 8193	0.921	1,000		
UV-VIS	0.942	0.984	1,000	
ASTM 7066	0.878	0.912	0.992	1,000

Correlation Models

Oil no. 2



	SM 5520 B	ASTM D 8193	UV-VIS
SM 5520 B	1,000		
ASTM D 8193	0.969	1,000	
UV-VIS	0.975	0.988	1,000



Conclusion and Next steps

- 🎯 Validation with depleted oil has been completed
- 🎯 The methods showed good correlations ($r = 0.9$)
- 🎯 Evaluation of the existence of correlations considering real samples
- 🎯 Evaluation of correlations considering oil-in-water samples without depletion procedure (presence of volatile compounds)
- 🎯 Final event for sharing the obtained data with Scientific and O&G Community .



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