Chemical Tracers for Near-Well SOR-monitoring

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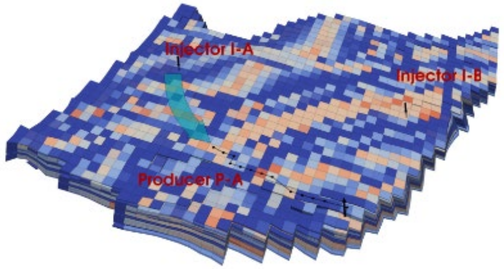
Project Manager O&G Application Lab. SNF France

Ex-Research Secientist IFE/National IOR Centre of Norway [2016-2020]

Project Organization

IOR Centre of Norway Theme 2/Task 5

“We put emphasis on real fields and aim to develop methodologies that ease the decision making and the reservoir management.”



THEME 2: MOBILE OIL – RESERVOIR CHARACTERISATION TO IMPROVE VOLUMETRIC SWEEP

TASK 5: TRACER TECHNOLOGY
Development tracer technology to measure reservoir properties and (changing) conditions during production.

https://www.uis.no/en/research/national-ior-centre-norway

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Arun Selvam

Workflow

2014-2016

Post-doctoral Fellow T. Brichart

- Introduction of new prospects in Tracer Technology for SWCT
- Establish chemical tracer synthesis strategies

2016-2018

Post-doctoral Fellow M. Ould Metidji

- Pursue exploring synthesis routes
- Develop characterization tools adapted for studying the new chemical structures

2018-2020

Research Scientist M. Ould Metidji

- First static test in reservoir conditions
- First coreflood experiments
- Identify key –parameters for potential up-scaling

Why use single-well tracer test ?

- Tracer test measuring the oil-contactable **Remaining Oil Saturation (S_{OR})**
- Test done on one well → results extrapolated to a larger area
- Preferential test used to determine S_{OR} by the industry: >500 tests! (shorter path to valuable results)

Estimation of conventional oil-reserves

Evaluate IOR/EOR operations efficiency

Fig. 13 Operational schedule for Well-X chemical flooding efficiency mini-pilot

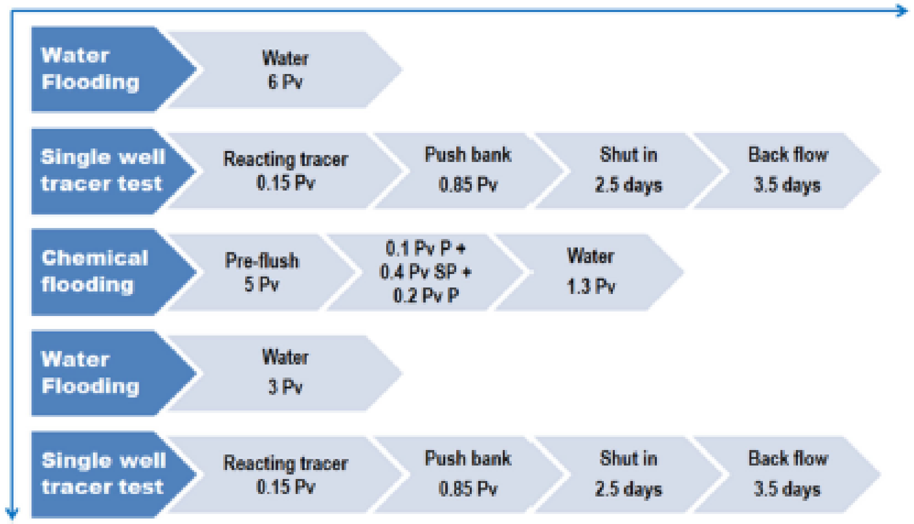


Table 5 Operational parameters for Well-X SWTTs

Reacting tracer concentration (ppm)	Volume of investigation (ft^3)	Injection rate (ft^3)	Production rate (ft^3)	Reaction rate (day^{-1})	Partition coefficient	Shut-in time (days)
10,000	8,992	8,992	7,194	0.05	3	2.5

Bu, Peter & Alsofi, Abdulkareem & Liu, Jim & Benedek, Lajos & Han, Ming. (2014). **Simulation of Single Well Tracer Tests for Surfactant-Polymer Flooding**. Journal of Petroleum Exploration and Production Technology.

History of Chemical Tracers for SWCTT

- 70's Firstly introduced
- 2013 Patent on Fluorescent tracers for O&G
- 2015 Patent on New Organic Tracers
- 2016 First Field Test in Saudia Arabia
- 2014 R&D on Fluorescent Tracers

1971 – Deans, H.
US3623842

2013 – Agenet, N. *et al.*
WO2014091144A3

2016 – Al Abbad, M. *et al.*
SPE-181408-MS

2014 – R&D Task National IOR Centre of Norway

Very few developments within Half a Century !

The National IOR Centre of Norway

Key Challenges

At the field scale

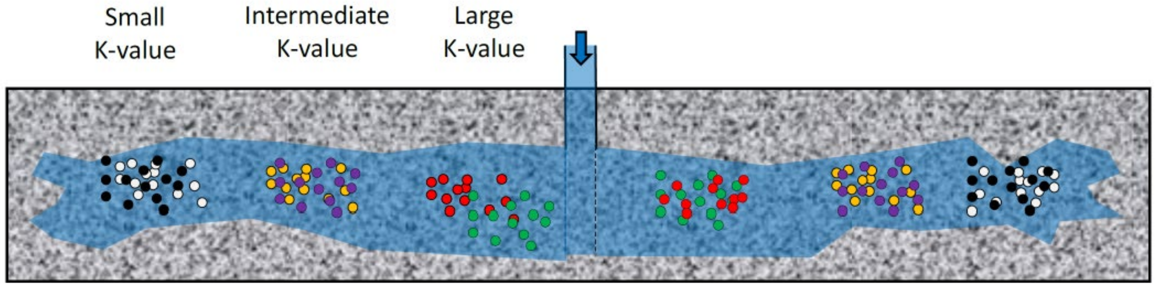
- Traditional Single-Well Technology: mg/L — **ppm** → ~ 0,5 mT / test
- Latest developments: ng/L — **ppt** → few grams / test
- **Our ambition with fluorescent tracers** — **ppb**→ hundreds of grams / test

Mass spectrometry (ppm, ppt)

Spectrophotometry (ppb)

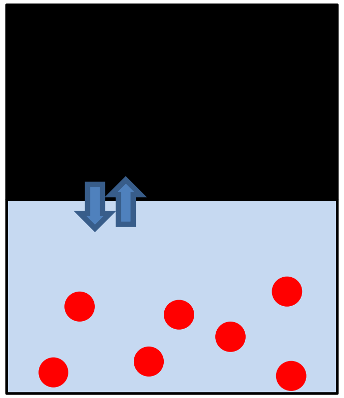
Main focus of the study

- Decrease concentrations / detection limits
- Design of tracer derivatives with different K-values and controlled hydrolysis rate
- To ultimately target different zones & cover a large range of temperature
- Real-time monitoring of tracers (fluorescence)



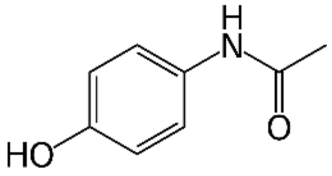
Al-Abbad, M., M. Sanni, S. Kokal, **A. Krivokapic, C. Dye**, Ø. Dugstad, S. Hartvig and **O. Huseby** (2016). A Step-Change for Single Well Chemical Tracer Tests (SWCTT): **Field Pilot Testing of New Sets of Novel Tracers**. UAE SPE-181408

Partitionning tracers

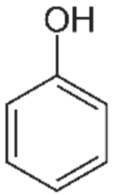


- Oil composition
- Temperature
- Water salinity

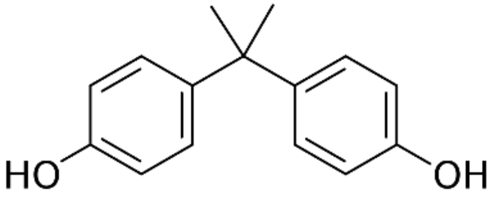
$$K_{part.} = \frac{[Tracer]_{Oil}\ (eq)}{[Tracer]_{aq.}\ (eq)}$$



paracetamol

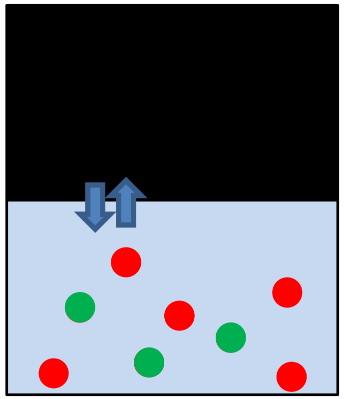


phenol



Bisphenol-A

Reactive partitionning tracers

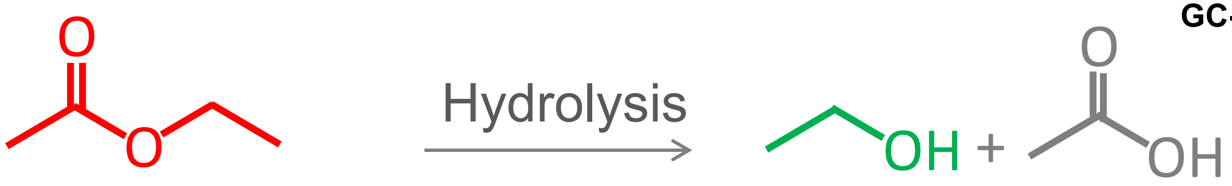


● Partitioning reactive tracer

● Passive tracer

Traditional tracer used since the 70s’

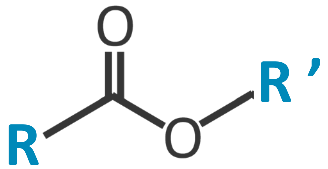
Detection limit mg/L
500 kg
GC-MS



ethylacetate

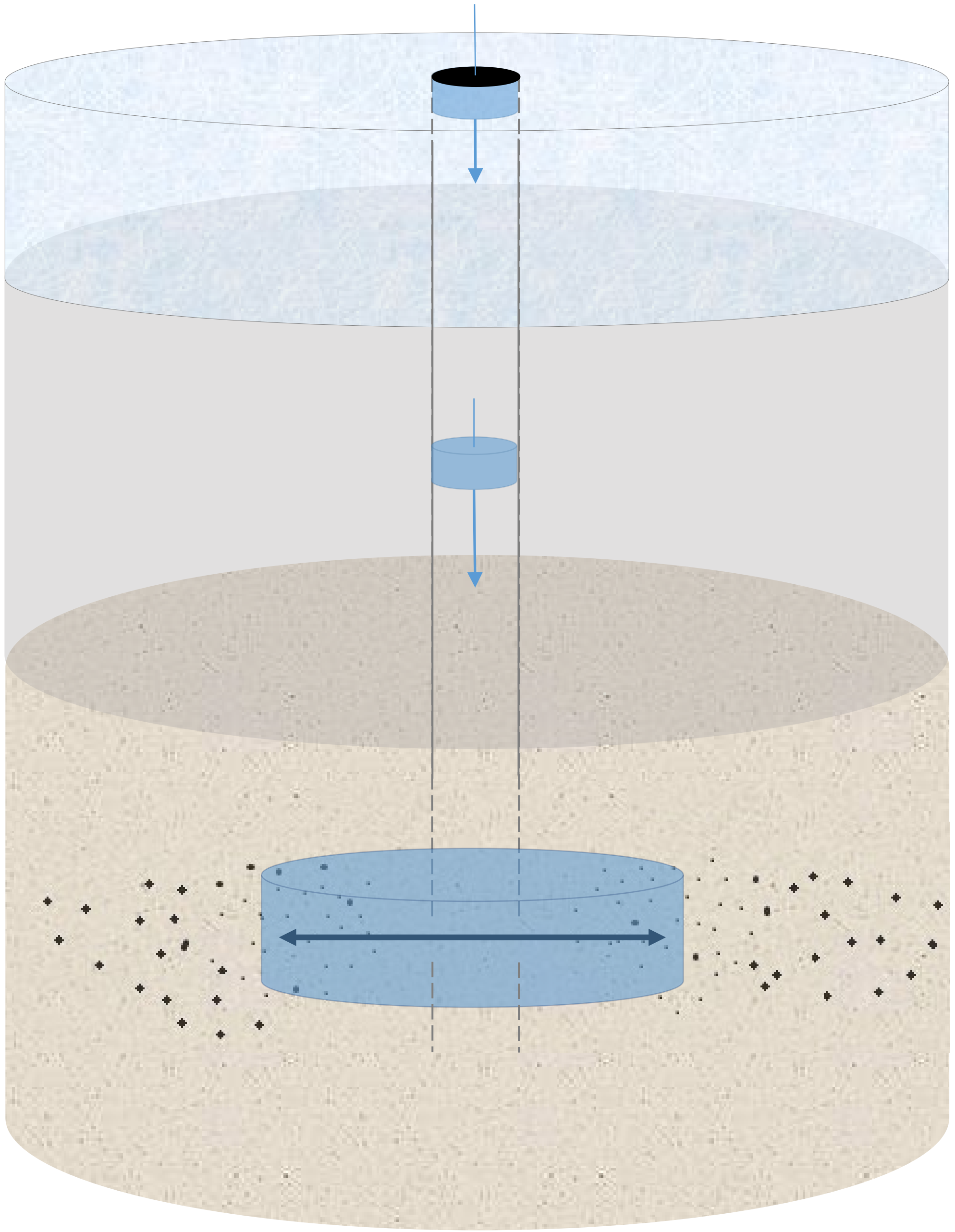
Tracers Implemented since 2016

Detection limit ng/L
50 g
GC-MS



new esters

Single-Well Chemical Tracer Test

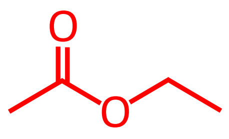


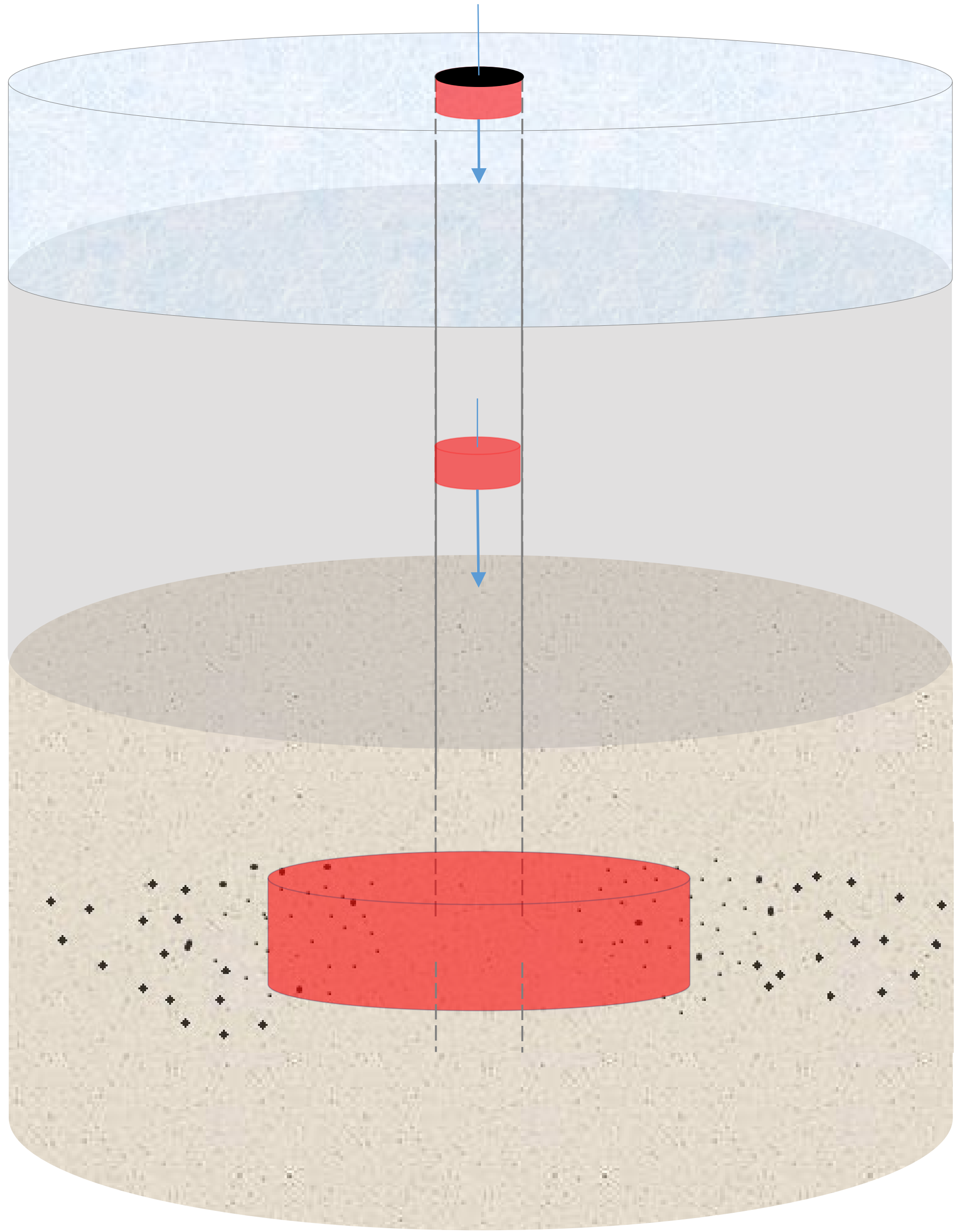
Step 1:
water-flooding to displace mobile oil from the tested area

S_{OR} = immobile oil !

- 5-10 meter away from the well-bore
- Reservoir cooled-down (Middle-East conditions)

Single-Well Chemical Tracer Test





Step 2:
Reactive tracer injection

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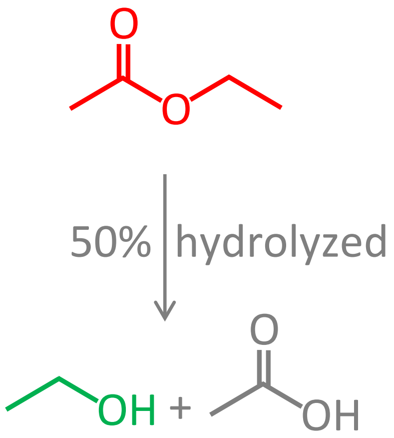
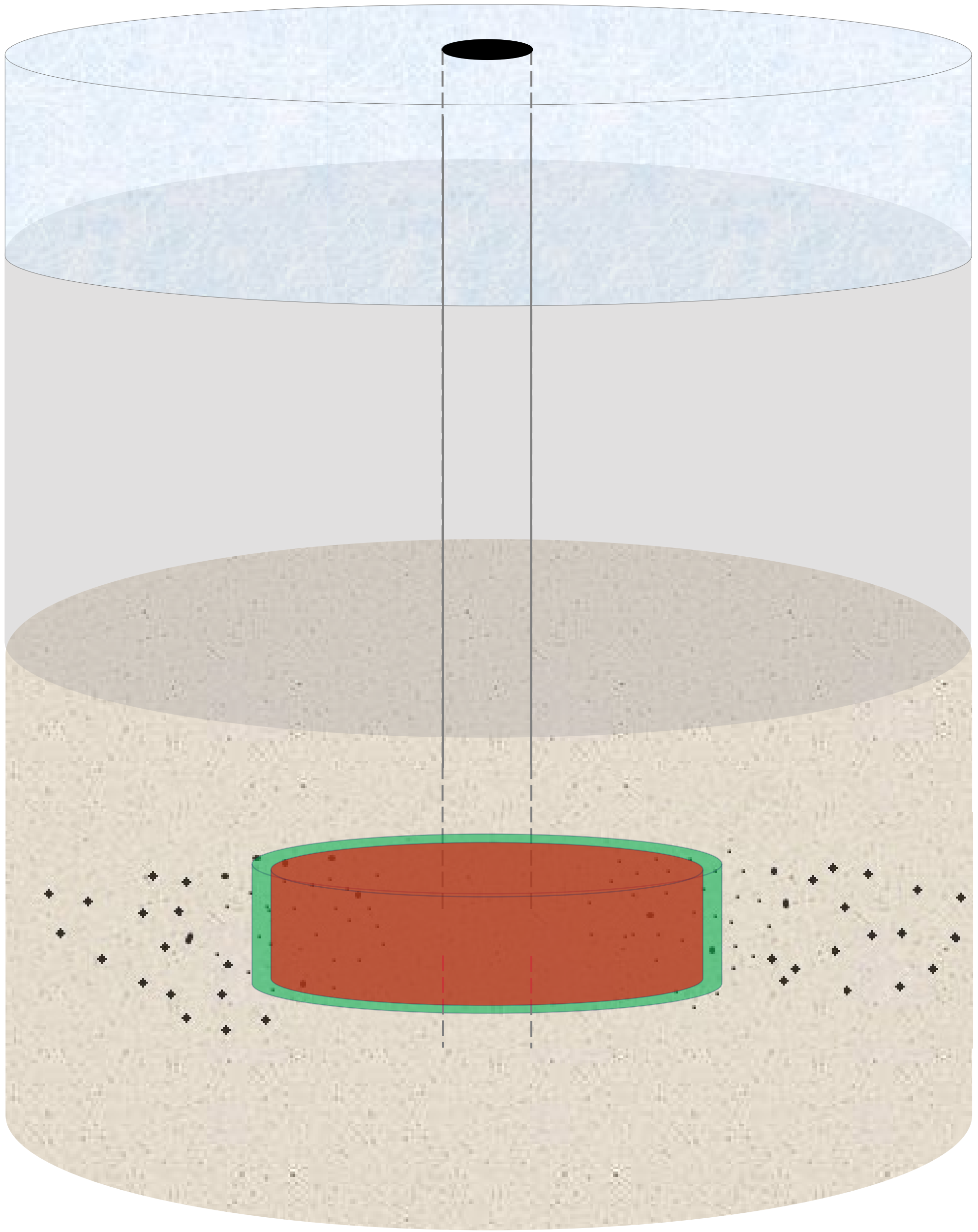
Single-Well Chemical Tracer Test



Passive tracer generated in-situ



Step 3:
Well shut-in until 50% ester hydrolyzed
Takes 3 to 10 days

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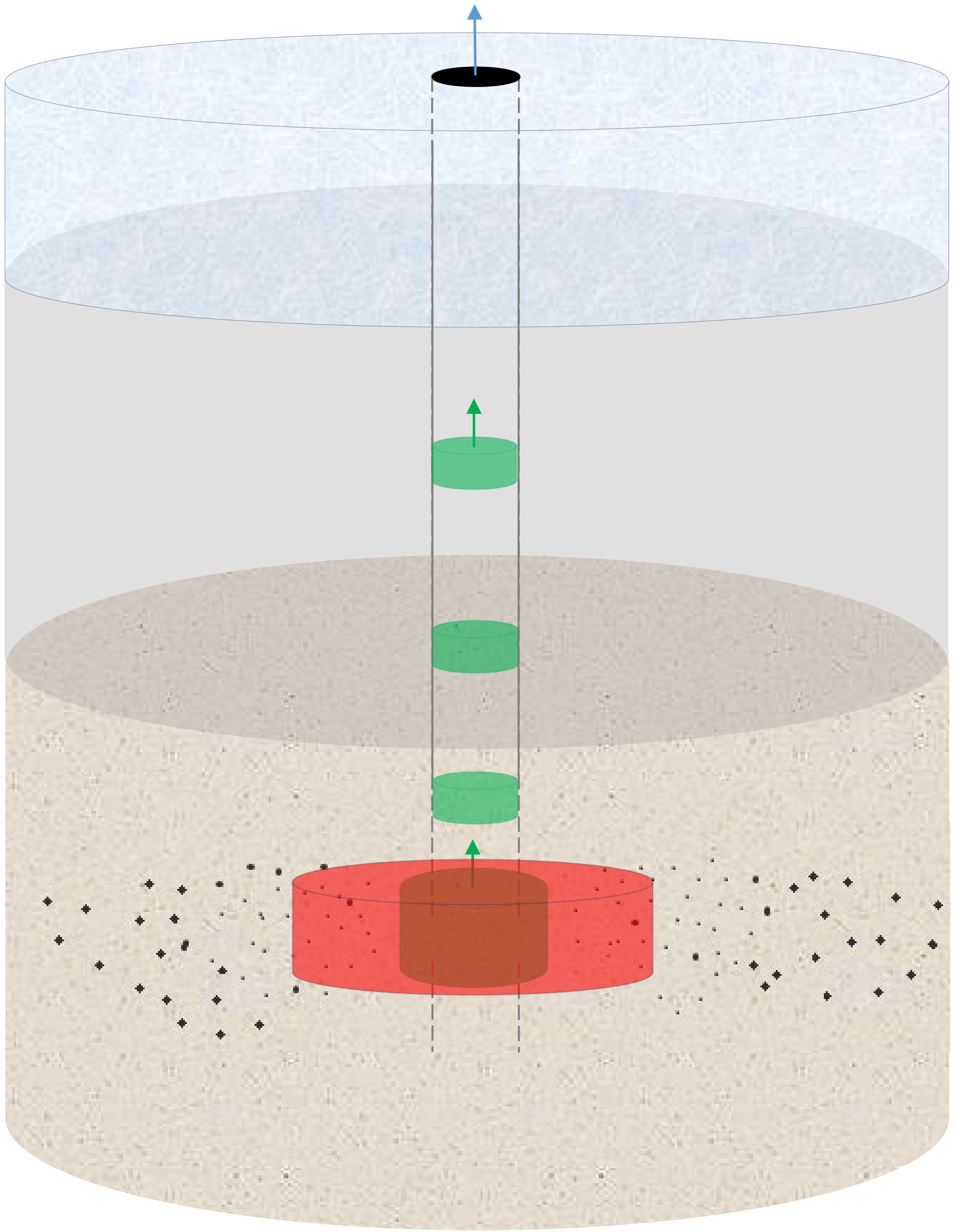
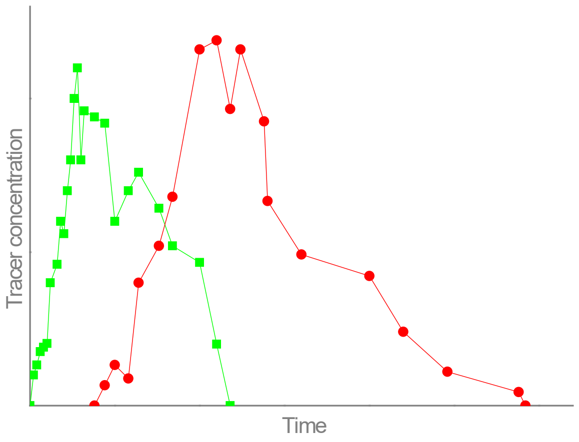
Single-Well Chemical Tracer Test



Step 4:
Back-production: chromatographic separation



$$S_{OR} = \frac{Tr - Tw}{Tr + Tw\,(K_{part.} - 1)}$$

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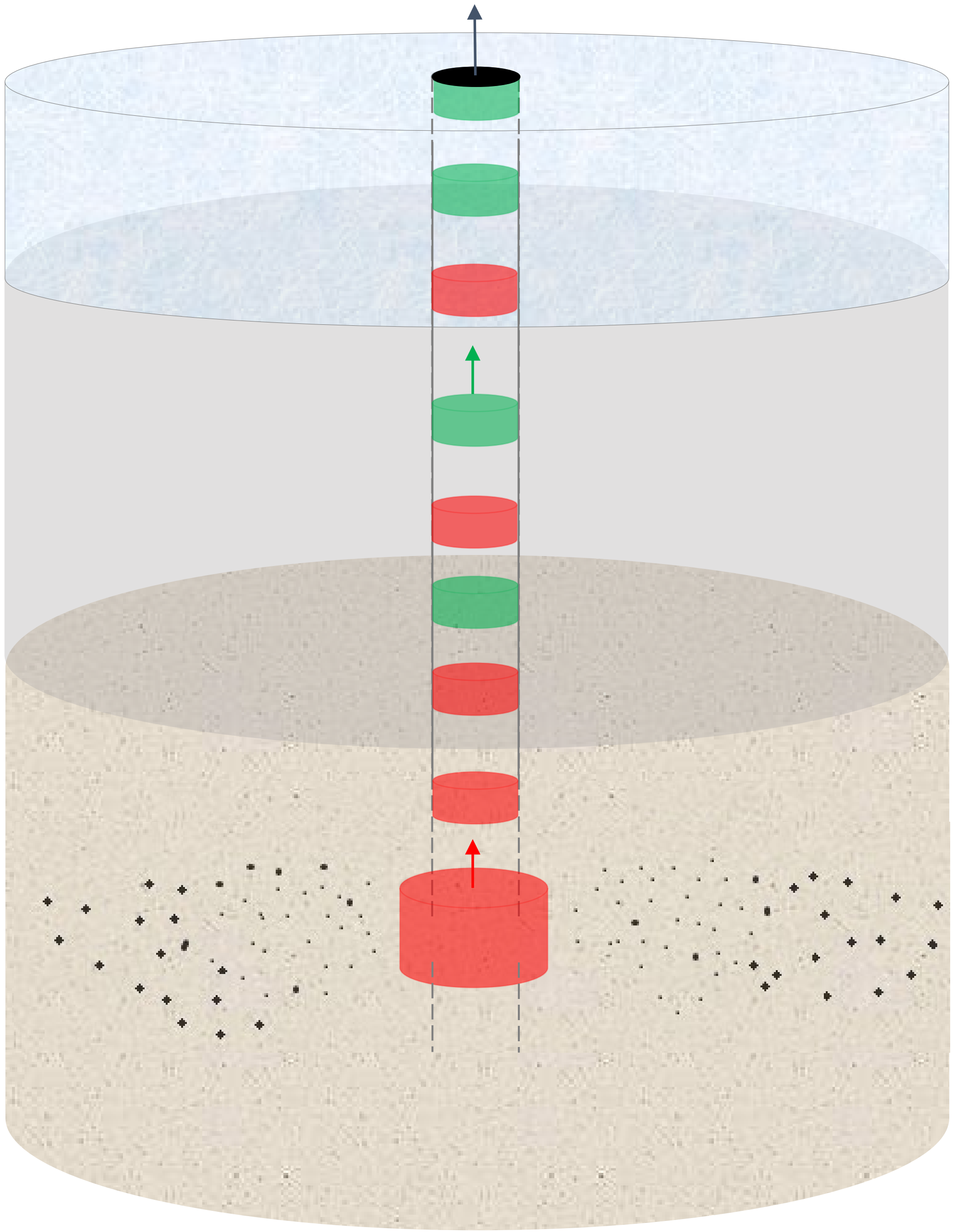
Single-Well Chemical Tracer Test



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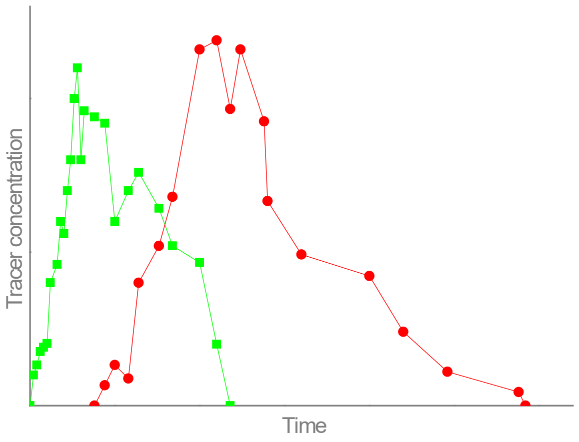

$$S_{OR} = \frac{Tr - Tw}{Tr + Tw\,(K_{part.} - 1)}$$

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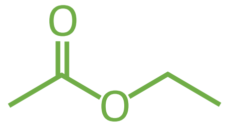
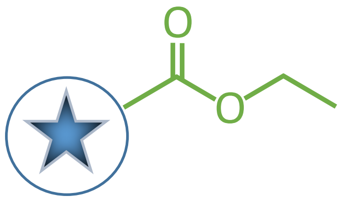
Targets

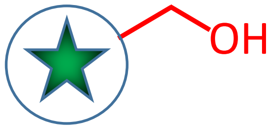
- X Injection in the kg range ! → poor detection limit (**ppm**)
- X Monitoring by GCMS : qualified workers
- X Secondary products : large ammount of acid *in-situ*



Fluorescents reactive esters

- ✓ Lower detection limit (**ppb**)
- ✓ Easier / faster handling !
- ✓ Possibility to use *on-site* or even *on-line*

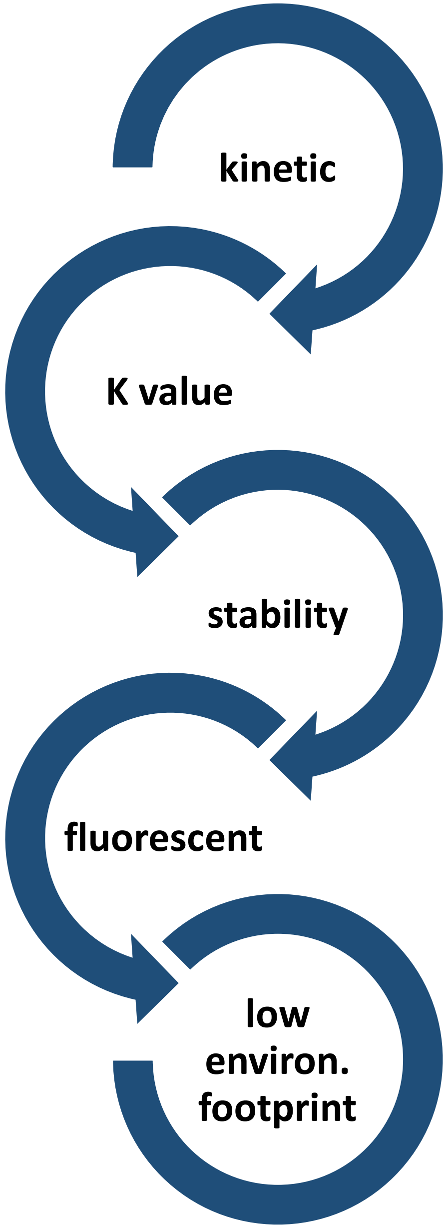




Key Challenges

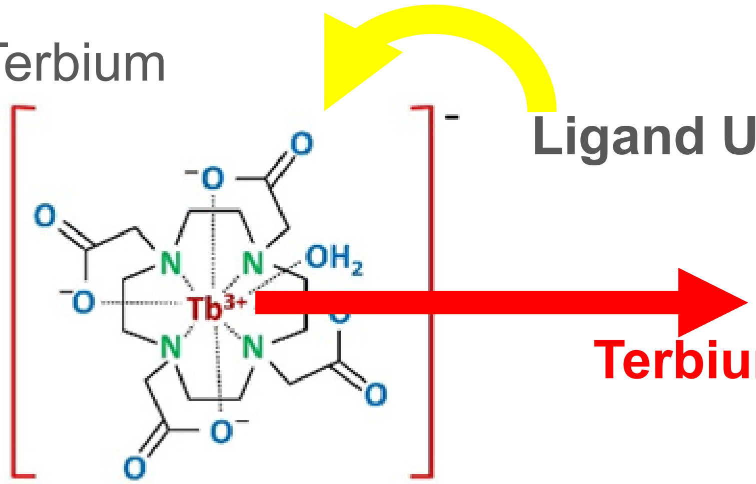
Main requirements to fulfill

- Reactive tracer with controlled kinetic
- Partitionning tracer with adapted k value
- Stable in reservoir conditions
- Detectable by TRF down to ppb (/ppt)



New Tracer Candidates

DOTA-Terbium

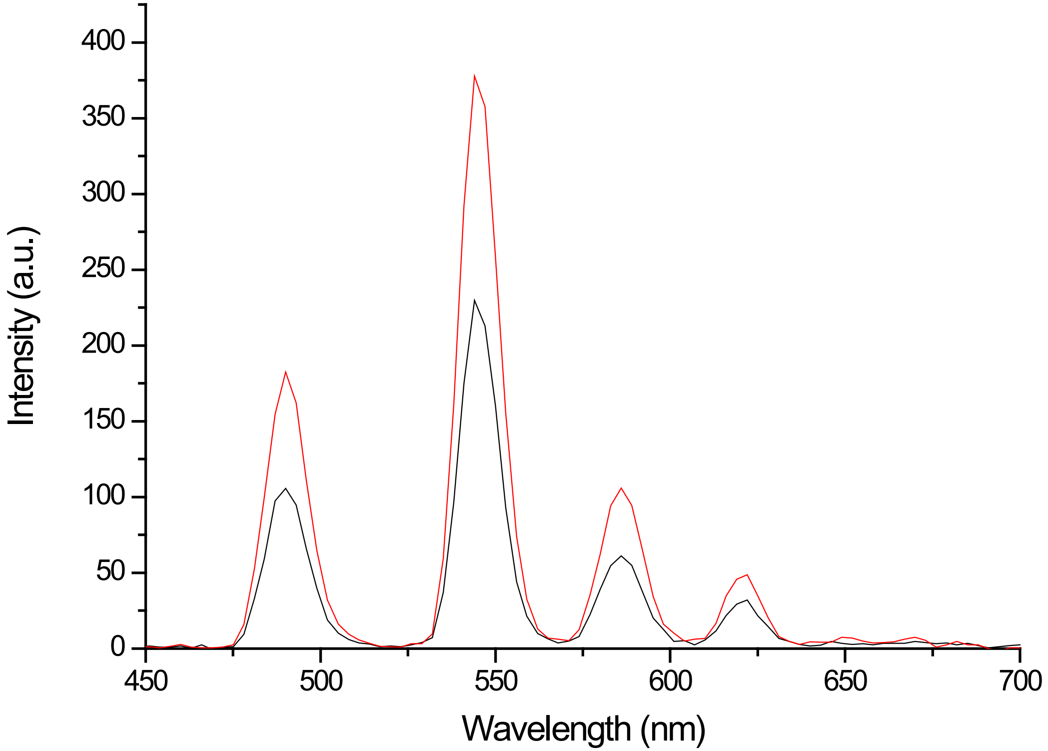


Ligand UV exc.

Terbium emission

Perriat, P., Crowther, N., Martini, M., Tillement, O., Brichart, T., University Claude Bernard Lyon 1 & Agenet, N. TOTAL S.A. (2014). U.S. Patent Application No. 14/128,194.

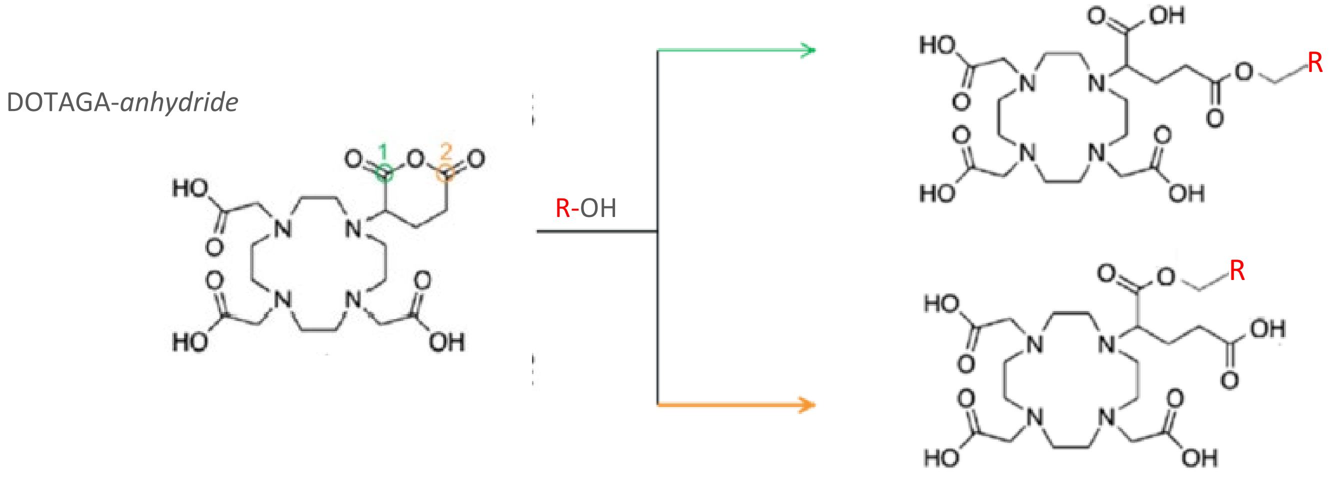
Fluorescence emission spectra



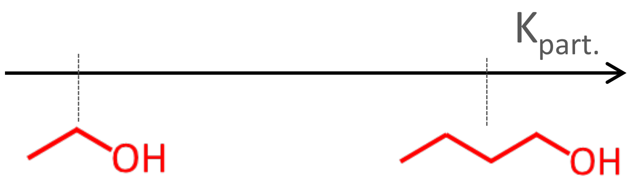
- ✓ Cyclic ligand: high stability constant
- ✓ **Long-lived fluorescent emission (ms)**
- ✓ Low detection: **ppt** to **ppb** depend on:

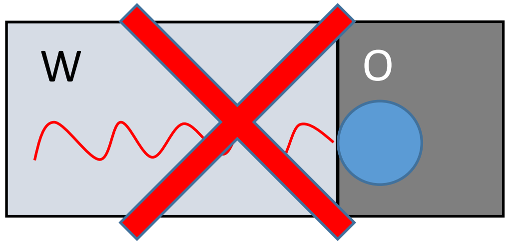
✓ Unique spectral fingerprints! not « significantly » present in oil-reservoirs!

Synthesis strategy: Step 1 Ligand Design

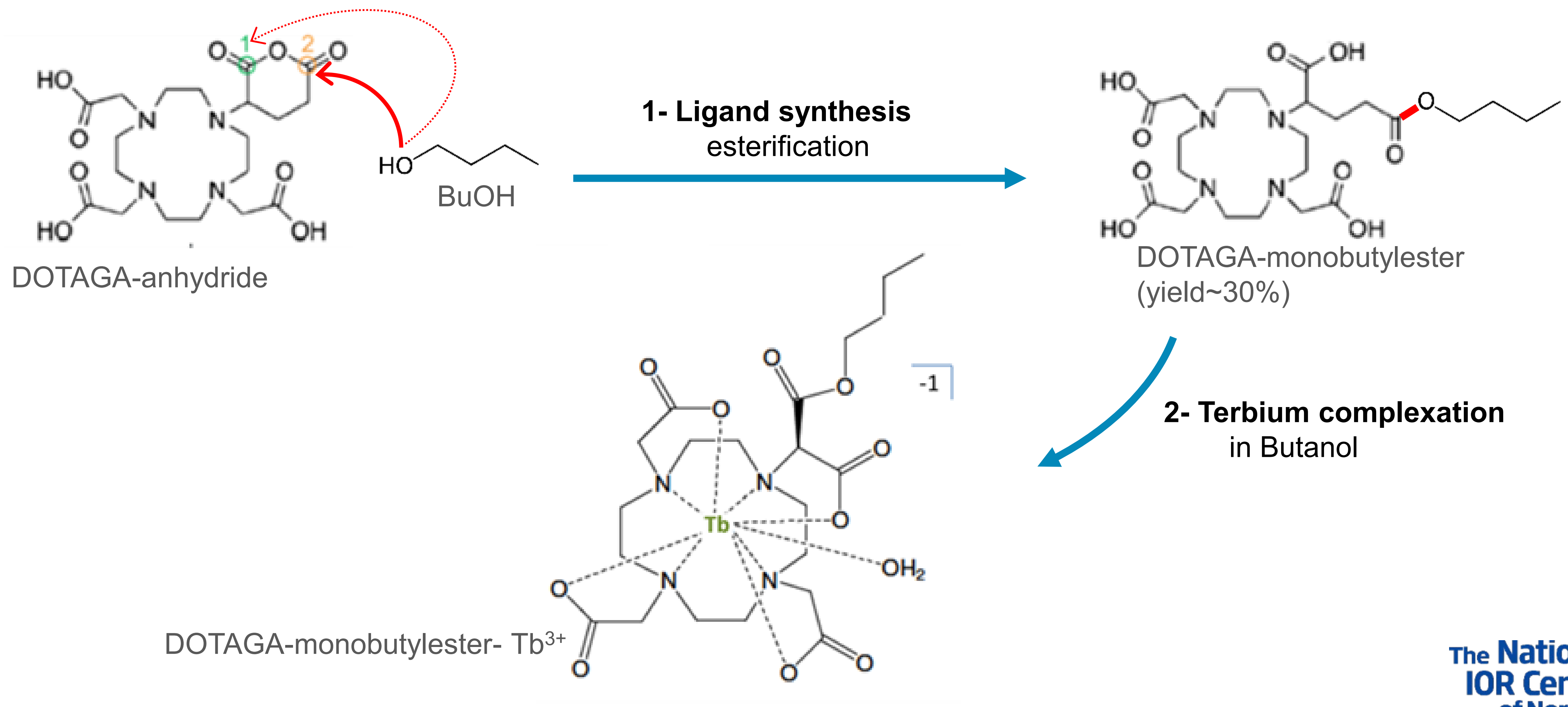


By varying R length → partitionning coefficient adjustement ($K_{part.}$)



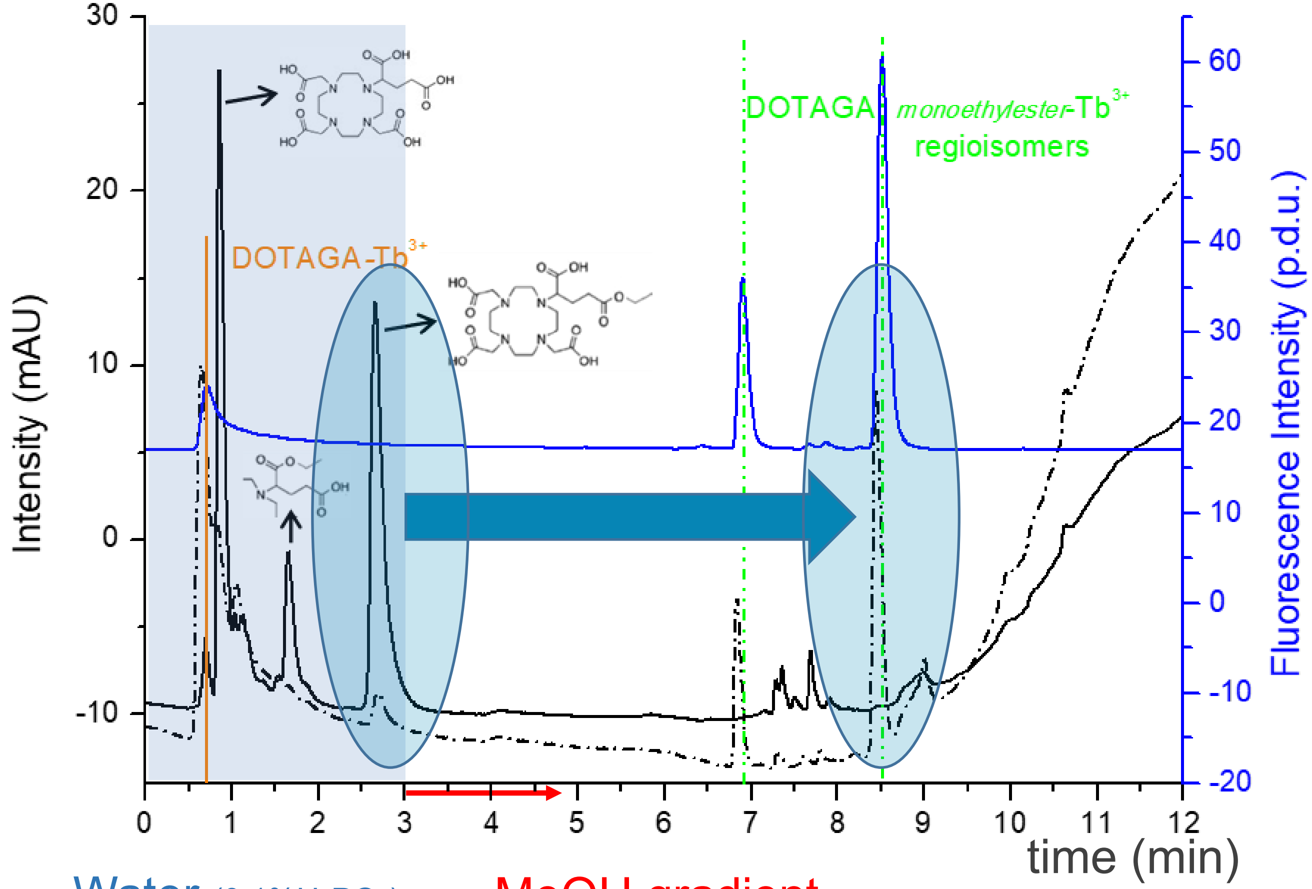


Synthesis strategy: Step 2 Metal Ion Complex



New tracer characterization: HPLC-TRF/UV

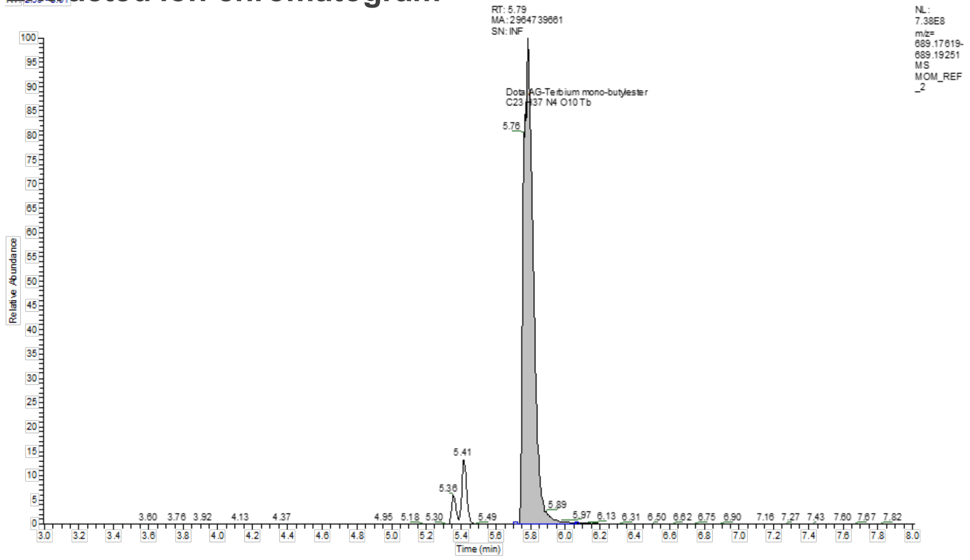
HPLC-TRF/UV



Water (0.1%H_3PO_4) MeOH gradient

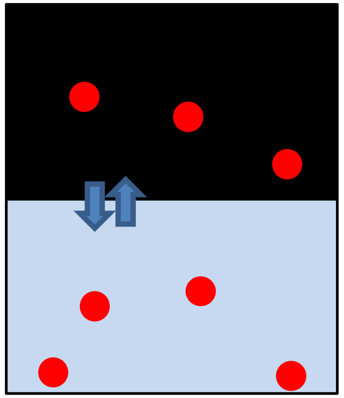
LC-MS

Extracted ion chromatogram



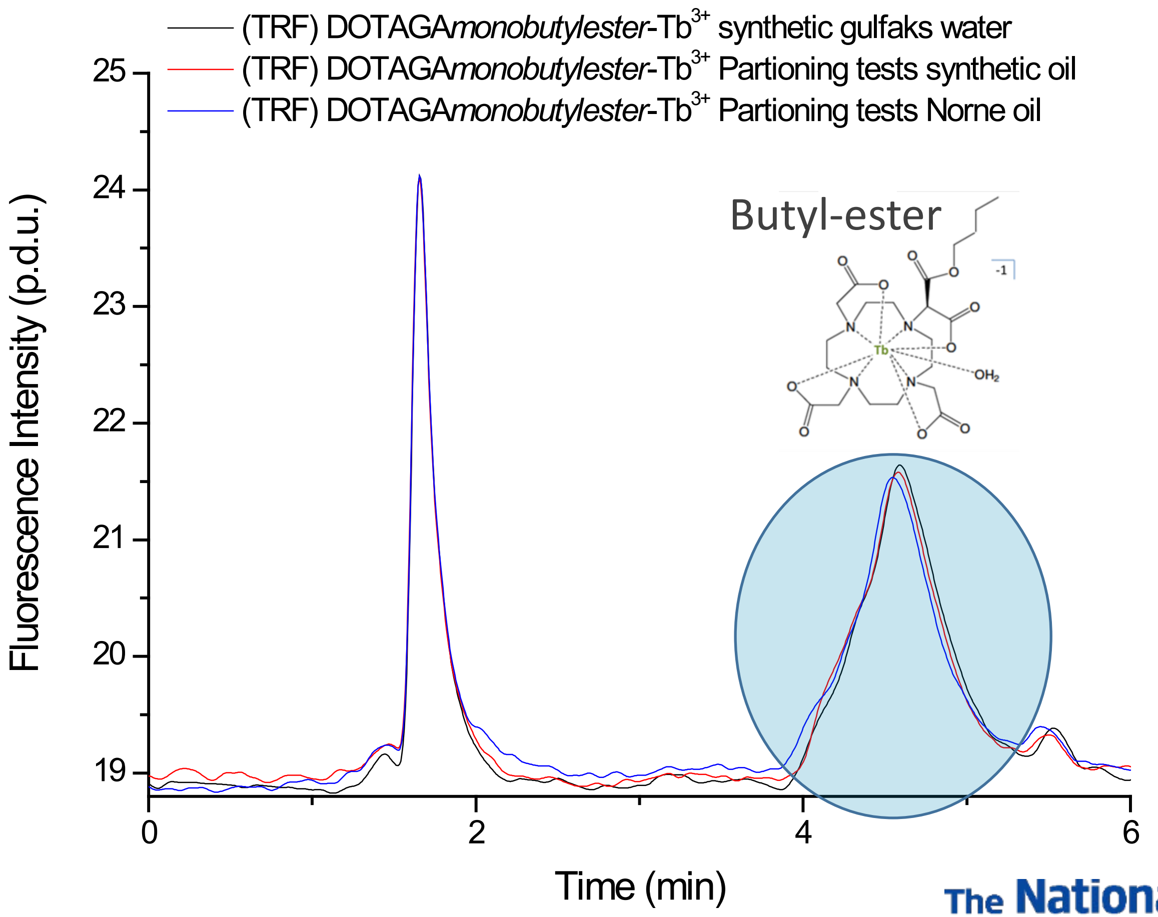
- Retention on C_{18} increases after complexation of metal ions
- TRF chromatograms → unique combination of metal with the ligand!

Partitioning Test (static experiments)



Synthetic oil
(Iso-octane/toluene/octanol)
or crude-oil (NCS: Norne)

Synthetic production water



Tracer exclusively hydrophilic !
Despite of the retention on the C_{18} column

→ Results confirmed by LC-MS

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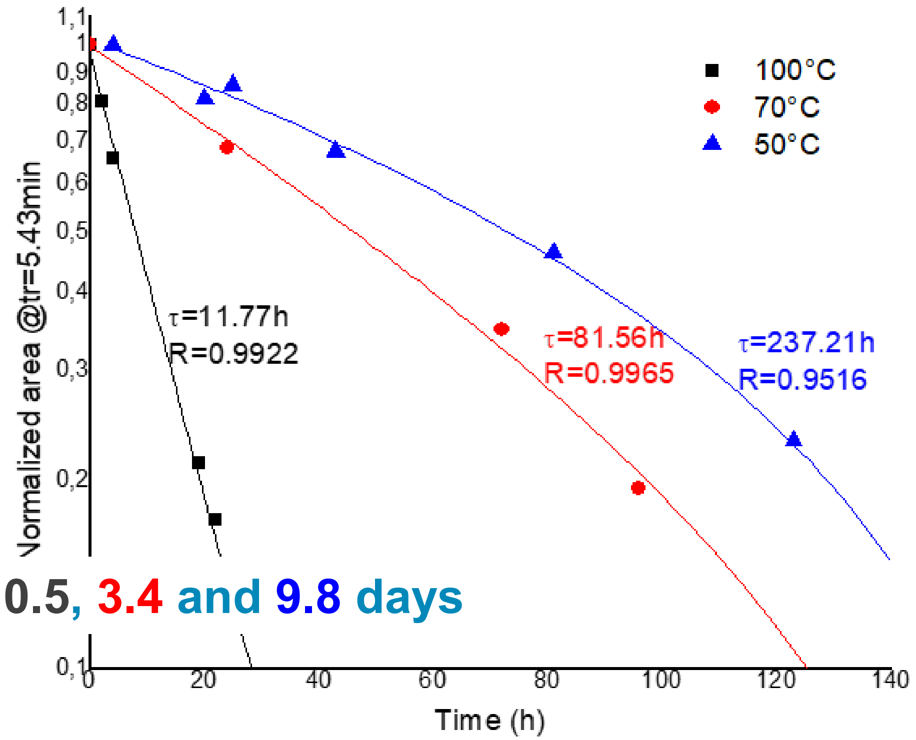
Temperature History Tracer / Stability test

Tracer in synthetic production water

50, 70 and 100 ° C (pH 6.5)

40,0g/L NaCl
0,63g/L KCl
2,54g/L $MgCl_2$
0,21g/L $NaHCO_3$
0,04g/L Na_2SO_4

Shut-in (days)	Salinity (ppm)	*T* (°F)	Reference
12	120,000	170	Tomich et al. (1973)
2.5	5,000	115	De Zwart et al. (2011)
4			De Zabala et al. (2011)
1.7	4,200	194	Hernandez et al. (2002)
4.5	200,000	100	Deans and Carlisle (1988)
5	43,000	234	Deans and Carlisle (1988)



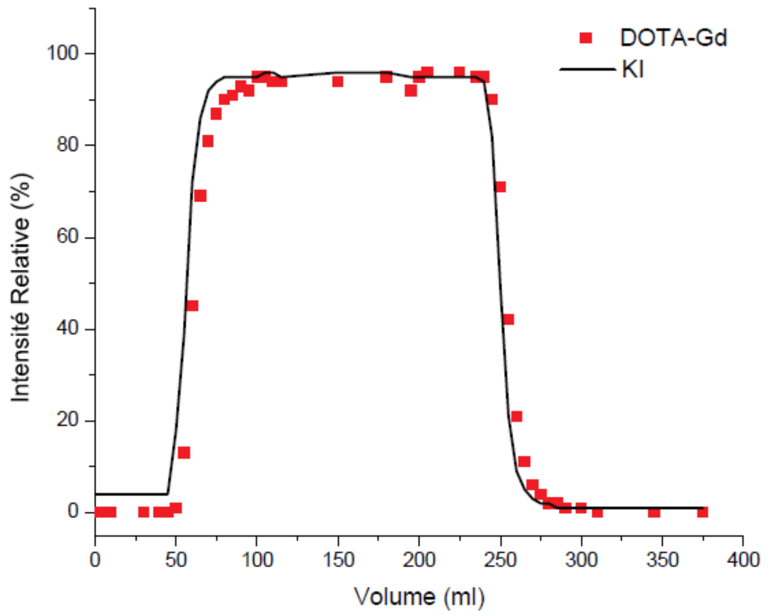
Half-lives: 0.5, 3.4 and 9.8 days

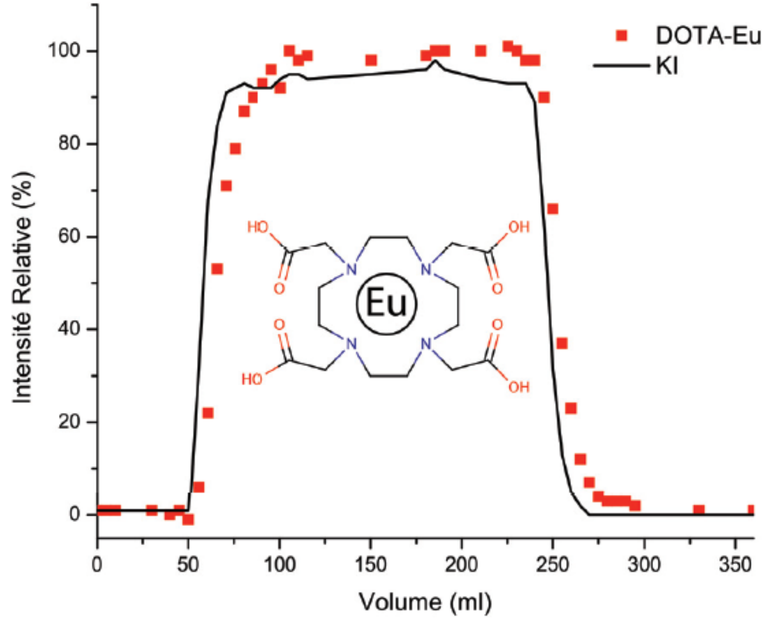
Bu, Peter & Alsofi, Abdulkareem & Liu, Jim & Benedek, Lajos & Han, Ming. (2014). **Simulation of Single Well Tracer Tests for Surfactant-Polymer Flooding**. Journal of Petroleum Exploration and Production Technology.

Coreflooding evaluation

Experimental Conditions

- Bentheimer Sandstone (1.5 Darcy)
- Passive form
- Test different fluorescent metal ions
- Next step → partitioning forms evaluation





Main requirements to fulfill

- Reactive tracer with controlled kinetic
- Adapted partitioning coefficient
- Stable in reservoir conditions
- Detectable by TRF down to ppb (/ppt)
- Validate the tracers via coreflooding evaluations
- No environmental issues (used in biomedicine + <ppb)

Key takeaways

Lanthanide complex

- Try more hydrophobic structures (>pentylester)
- Start coreflood evaluation on new partitioning compounds
- Identify other fluorescent structures with simpler chemistries

Potential Up-Scaling

Case	h (ft)	EtAc		Push bank		Shut-in (days)	Salinity (ppm)	*T* (°F)	Reference
		Con.	(bbl)	Con.	(bbl)				
1	20	1.5 % V	550	0.5 % V	1,370	12	120,000	170	Tomich et al. (1973)
2	65.6	10,000	189	2,500	755	2.5	5,000	115	De Zwart et al. (2011)
3	160.7	9,000	1,000	2,400	4,044	4			De Zabala et al. (2011)
4	22	15,245	75	4,300	525	1.7	4,200	194	Hernandez et al. (2002)
5	32	9,000	135	1,700	570	4.5	200,000	100	Deans and Carlisle (1988)
6	45	7,000	135	12,700	550	5	43,000	234	Deans and Carlisle (1988)

Potential Up-Scaling

Considering 500g / SWCT test

- Conversion yield step 1 (esterification): ~50%
- Conversion yield step 2 (complexation): 100%

Raw Materials

Step1

- DOTA-GA anhydride Mw=458,46 $g.mol^{-1}$ (purity >99%) …………………60mg
- Butanol Mw=74,121 $g.mol^{-1}$ (purity >99%) …………………30mg

Step2

- DOTA-GA ester Mw=689.17 $g.mol^{-1}$ (purity >99%) …………………30mg
- $TbCl_3.6H_2O$ Mw=373.38 $g.mol^{-1}$ (purity >99%) …………………80mg
- Butanol Mw=74,121 $g.mol^{-1}$ (purity >99%) …………………17mg

Final balance for 1 test:

215g of DOTAGA-A + 285g of Terbium salt + 168g Butanol

Acknowledgement

SNF S.A. (Permission to present)

Tor Bjørnstad

Tracer Department, IFE

The National IOR Centre of Norway

The 2020 user partners and observers:

NEPTUNE ENERGY

wintershall dea

vår energi

HALLIBURTON

Schlumberger

ConocoPhillips

Lundin Norway

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AkerBP

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Deliverables

PUBLICATIONS

Ould Metidji M., Silva M., Krivokapic A. and Bjørnstad T., (**2019**) Synthesis and Characterization of a Reactive Fluorescent Tracer and its Possible Use for Reservoir Temperature's Data Collection, 20th European Symposium on Improved Oil Recovery in Pau, France, 8-11 April 2019.

Brichart T., Ould Metidji M., Ferrando-Climent L., and Bjørnstad T., (**2017**) New Fluorescent Tracers for SWCTT, EAGE 19th European Symposium on Improved Oil Recovery held in Stavanger, Norway, 24-27 April 2017.

PRESENTATIONS

Ould Metidji M., (**2020**), Novel route of synthesis of partitioning fluorescent tracers adapted for SWCTT, IOR NORWAY 2020 & 14th Symposium on Wettability, 27-29 April 2020, Stavanger, Norway, Oral Presentation.

Ould Metidji M., (**2019**), Synthesis and Characterization of a Reactive Fluorescent Tracer and its Possible Use for Reservoir Temperature's Data Collection, EAGE 20th European Symposium on Improved Oil Recovery in Pau, France, 8-11 April 2019, Oral Presentation.

Ould Metidji M., Krivokapic A., Silva M., Cathles L., Selvam A. and Bjørnstad T., (**2019**), Contribution to the Study on Nanoparticles as Oil-Reservoir' Tracers, Conference IOR Norway "All for IOR, IOR for all", Stavanger, Norway, 19, 20 March 2019, Poster Presentation.

Ould Metidji M., Nanotracers intended for EOR operations, (**2018**), SPE Workshop: Improved Decision-Making Through Tracer Technology, Abu Dhabi, UAE, 14,15 March 2018, Poster Presentation.

Pr. Lawrence Cathles III and Ould Metidji, M., (**2018**), Multicomponent tracer methods to assess fracture-controlled flow tens of meters from the wellbore and a proposed field test, co-presentation at the workshop preceding the Conference IOR Norway, Stavanger, Norway, 23-25 April 2018, Oral Presentation.

Ould Metidji M., (**2017**), New Fluorescent Tracers for SWCTT, EAGE 19th European Symposium on Improved Oil Recovery, Stavanger, Norway, 24-27 April 2017, Oral Presentation.

Ould Metidji M., (**2017**), Carbon-based nanoparticles for Inter-Well Tracer Test, Internal Workshop at Cornell University: Earth and Atmospheric Sciences department, Ithaca, USA, March 2017, Oral Presentation.

M. Ould Metidji, (2017), Laboratory assessment of nanotracers for oil reservoir characterization, C2E – Colloids and Complex fluids for Energies IFPEN, Rueil-Malmaison - 4-6 December 2017, Poster Presentation

Nanoparticle Tracers for Petroleum Reservoir Studies

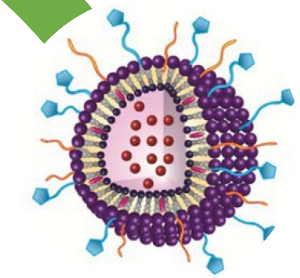
Arun Kumar Panneer Selvam

PhD Candidate

National IOR Center of Norway

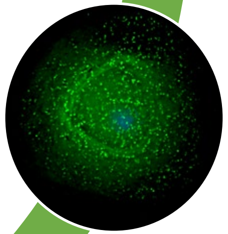
University of Stavanger (UiS), Institute for Energy Technology (IFE)

PhD Project



Nanocarriers

– *Residual Oil Saturation*



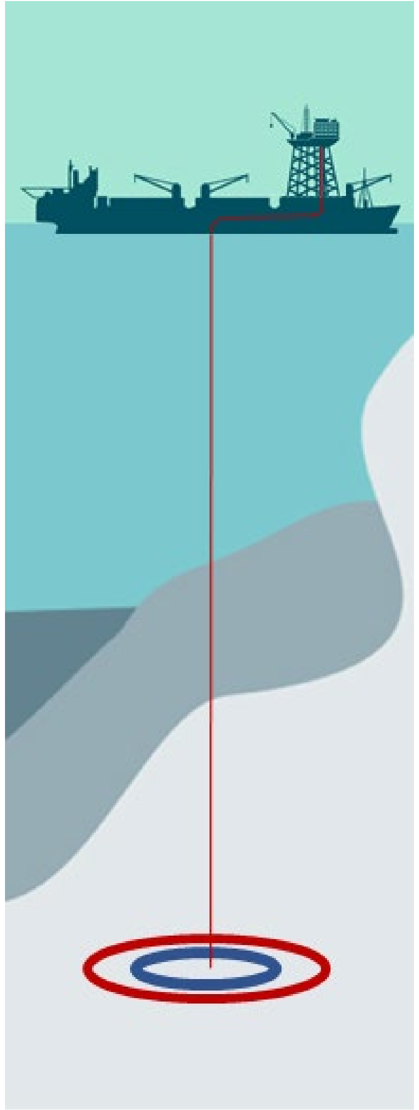
Quantum dot nanoparticles

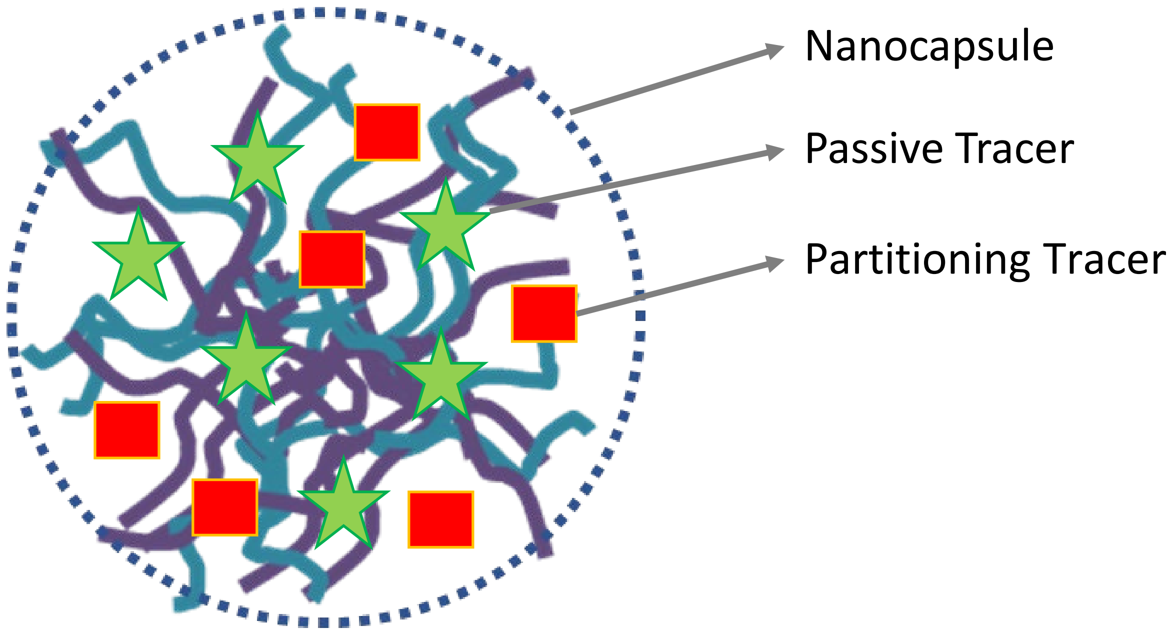
– *Fracture detection*

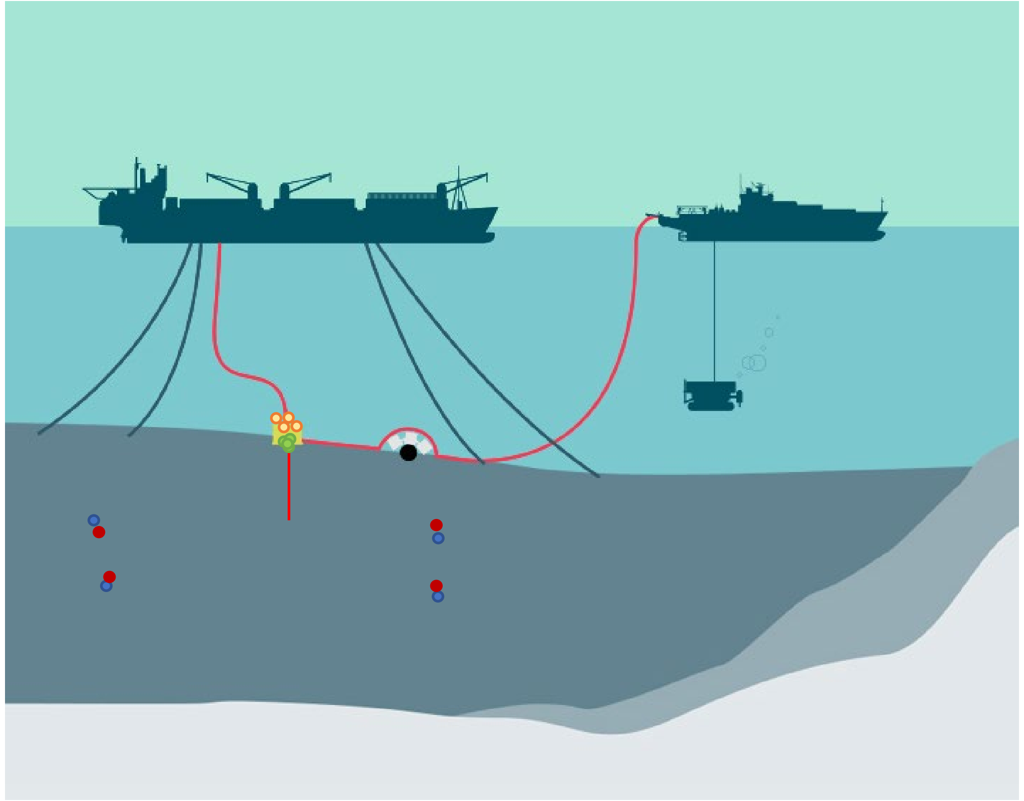
Single well Chemical Tracer Tests

- Challenges
  - Amount of Tracer required
  - Long well shut-off period

- Use of Stimuli-Sensitive load carriers for tracer release

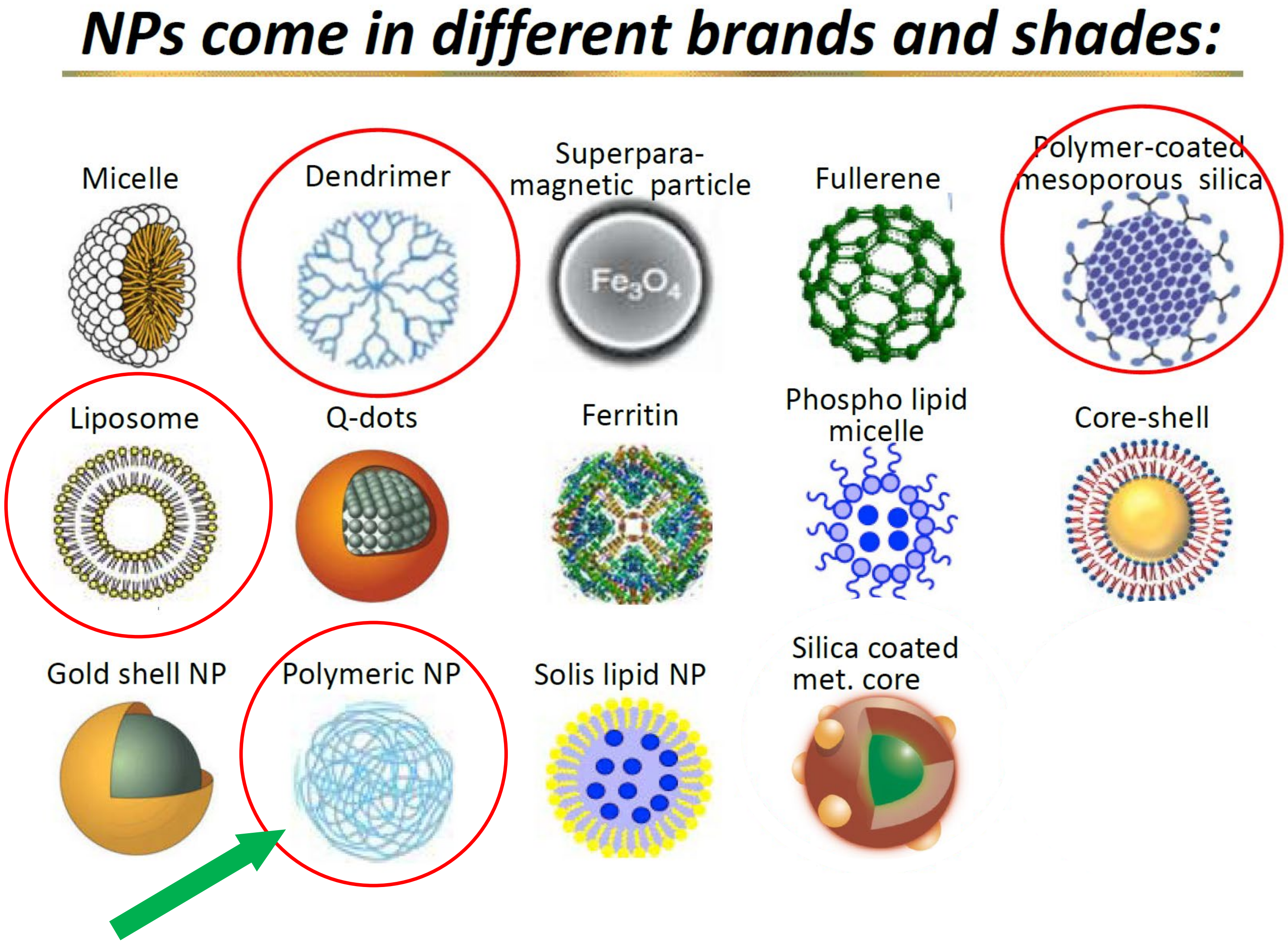



Nanocapsule
Passive Tracer
Partitioning Tracer



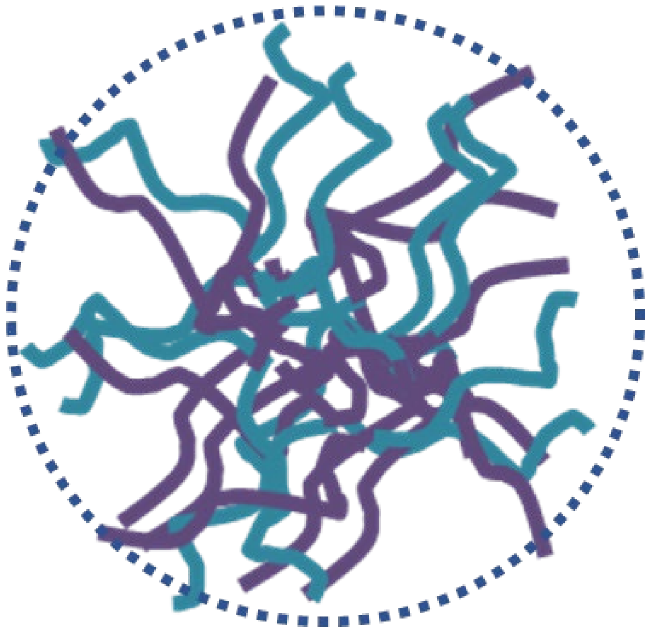
Nanocarriers $\xrightarrow{\text{Trigger}}$ Passive Tracer + Partitioning Tracer

Stimuli Sensitive Nanocarriers



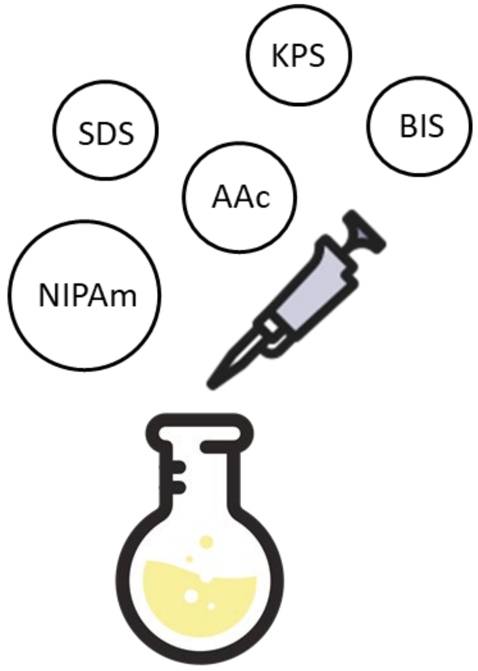
Nanogels

- ➢Colloidal Gels in nano-regime
- ➢Highly cross-linked polymeric network
- ➢Highly porous structure
- ➢Can be tuned towards different external stimuli (eg: Temperature, pH etc)
- ➢Sensitivity depends on the type of polymer used



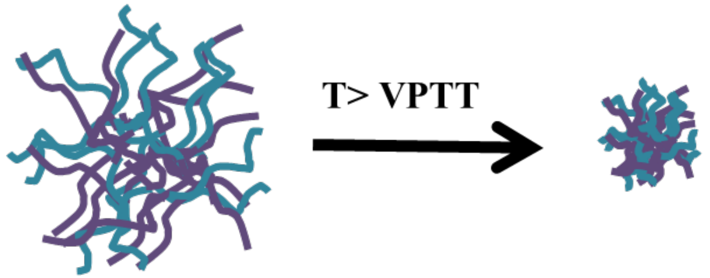
Synthesis of Nanogels

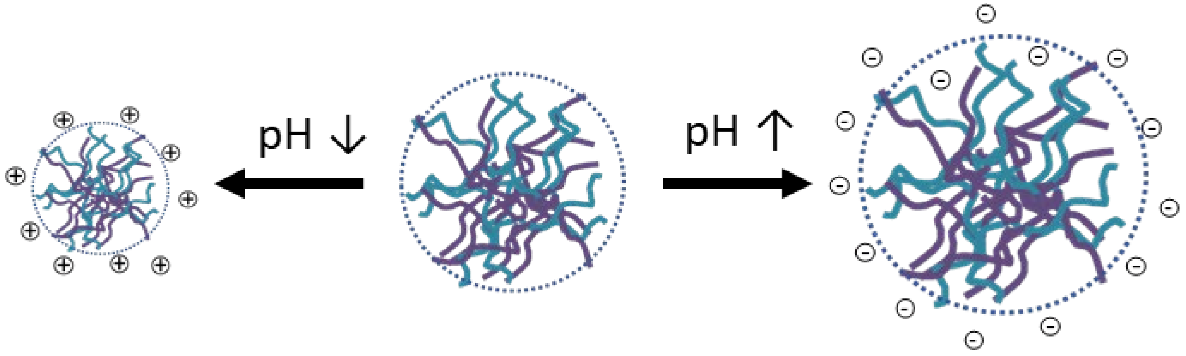
- ➢Emulsion Polymerization
- ➢Reactants:
  - ➢Monomers: N-isopropylacrylamide (NIPAm), Acrylic Acid (AAc)
  - ➢Cross-linker: N,N'-methylenebis(acrylamide) (BIS)
  - ➢Surfactant: Sodium dodecyl sulfate (SDS)
  - ➢Initiator: Potassium Persulphate (KPS)
- ➢Purification - Dialysis



Temperature and pH responsiveness

- Lower Critical Solution Temperature (LCST) describes the miscibility of polymer in solvent.
- The analogous term for 3D nanogel network is called volumetric phase transition temperature (VPTT).
- Influence of pH – dominated by behavior of AAc-segments





pNIPAm-AAc Nanogels

Sample	NIPAm (Mole %)	AAc (Mole %)	BIS (Mole %)
NG01	85	10	5
NG02	75	20	5
NG03	65	30	5
NG04	55	40	5
NG05	47.5	47.5	5

Table: Mole ratios of the monomer, co-monomer and cross linker used for the sample.

pNIPAm-AAc Nanogels

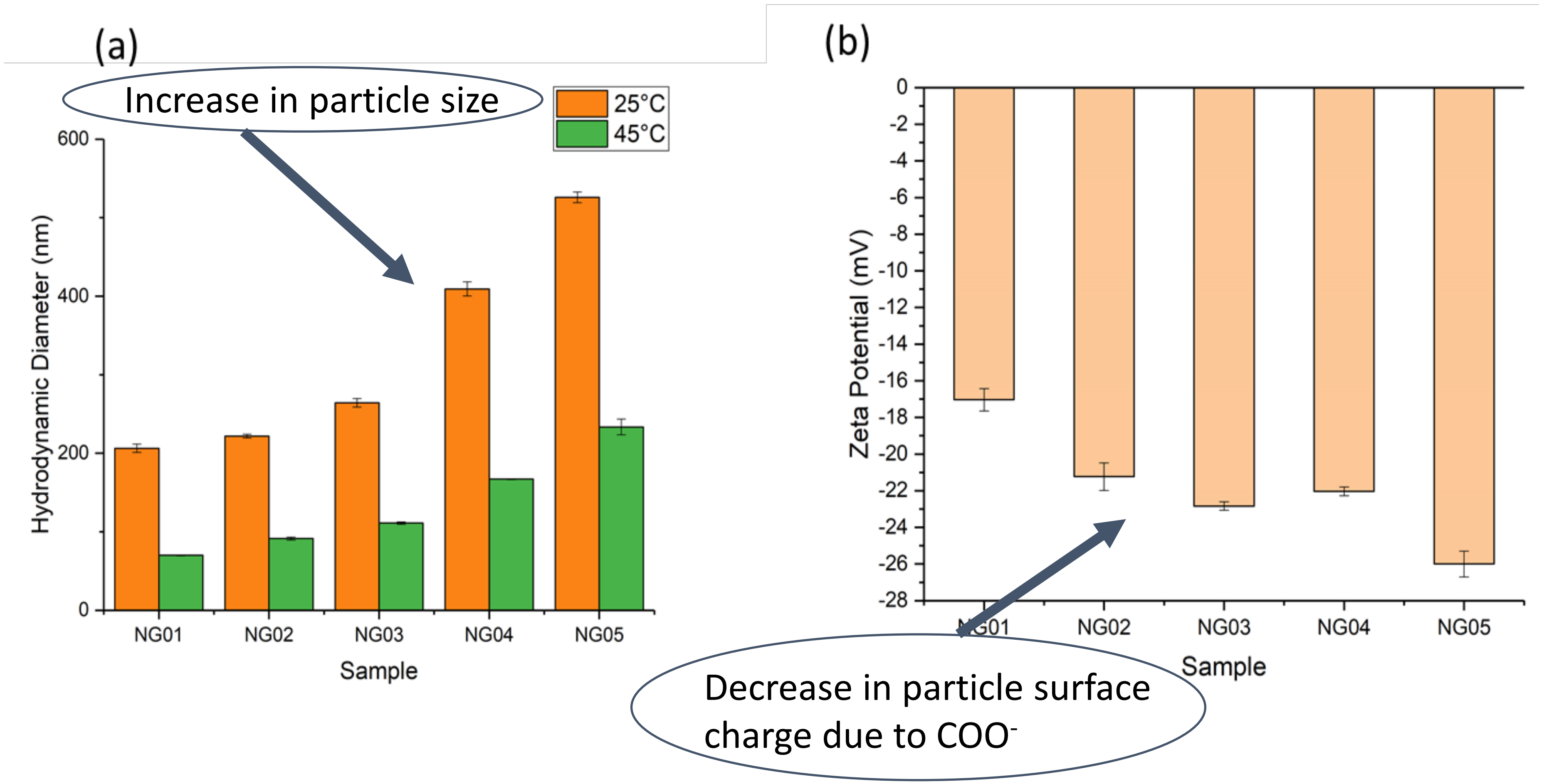


Figure: (a) Hydrodynamic diameter at swelled and collapsed state of different nanogel samples of varying mole ratios of NIPAm and AAc. (b) Zeta potential measurements of various nanogel samples.

Reversibility

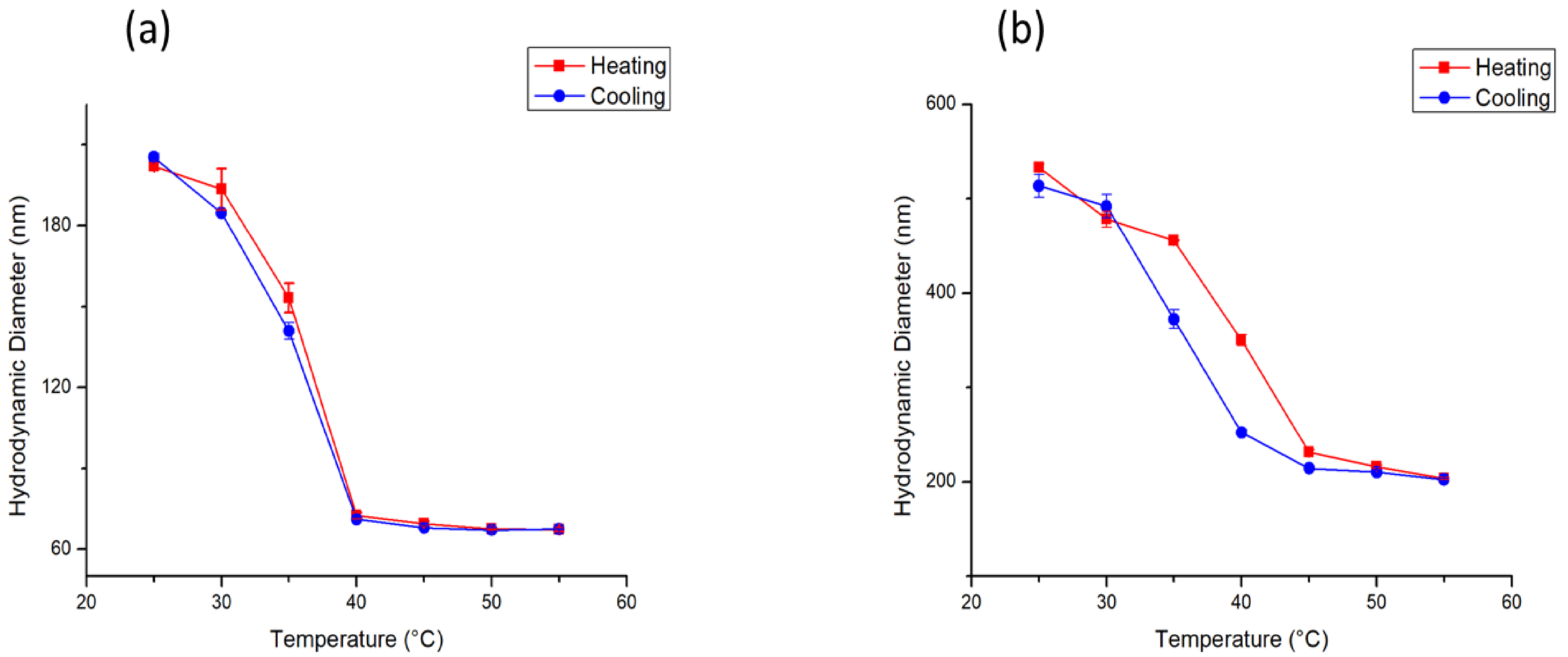
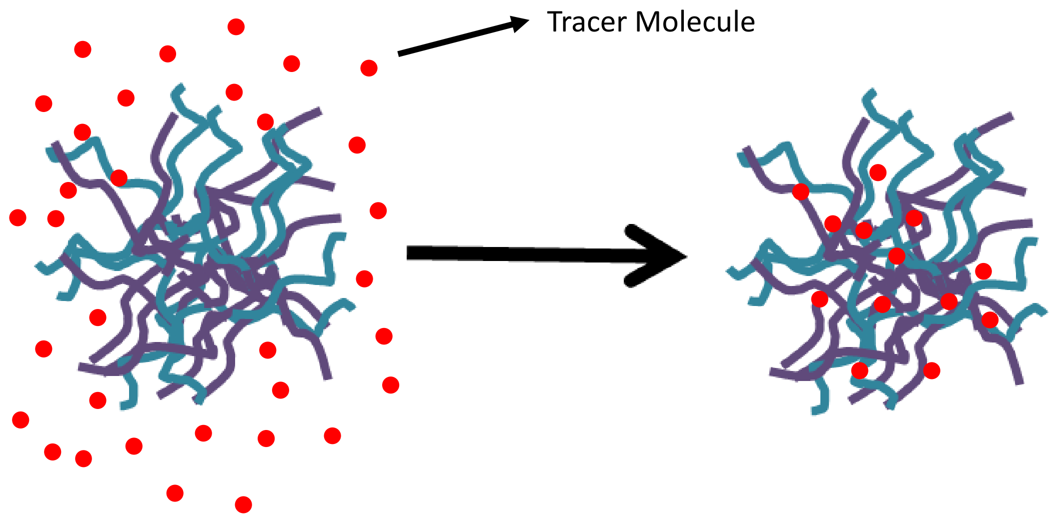
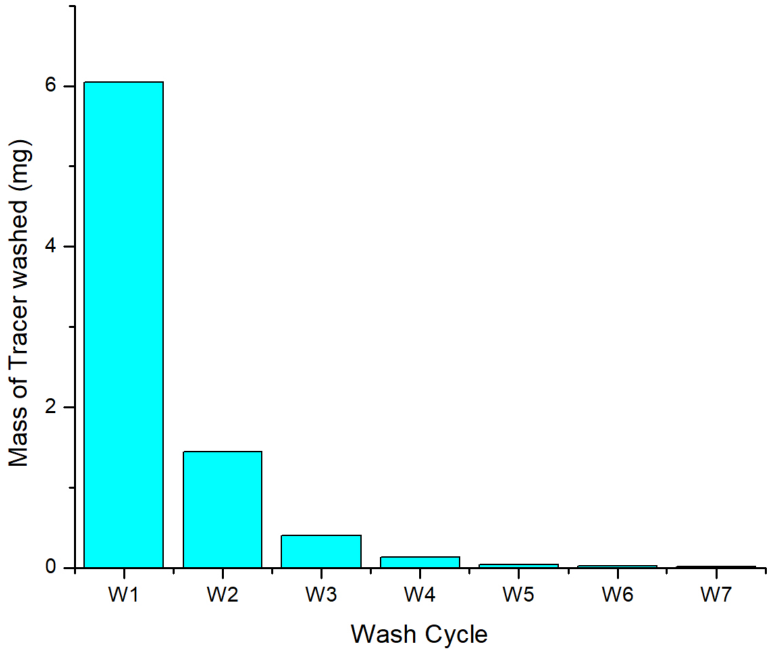


Figure: Reversible swelling and collapse of the nanogels as function of the temperature: (a) NG01 and (b) NG05

Tracer Loading and Release Mechanism

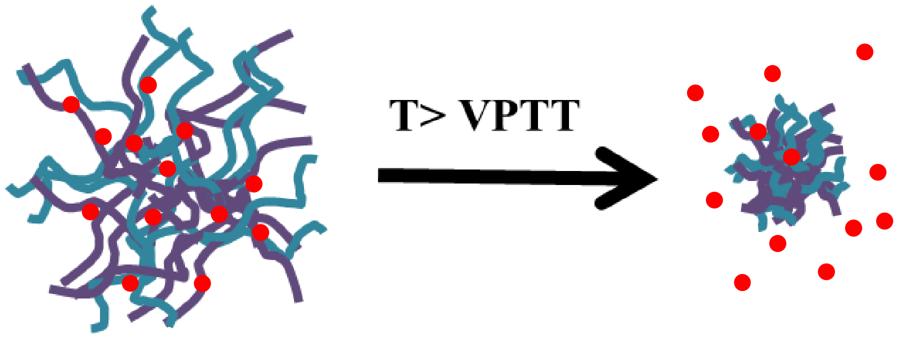
- Loading
  - Freeze-dried Nanogels introduced to high concentration tracer solution

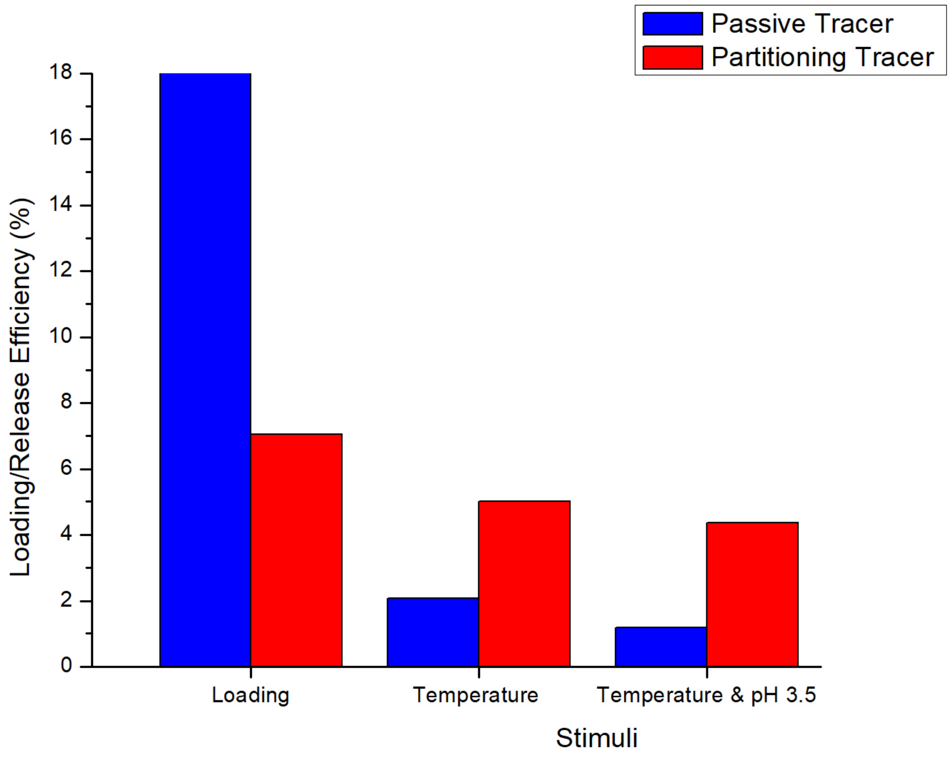




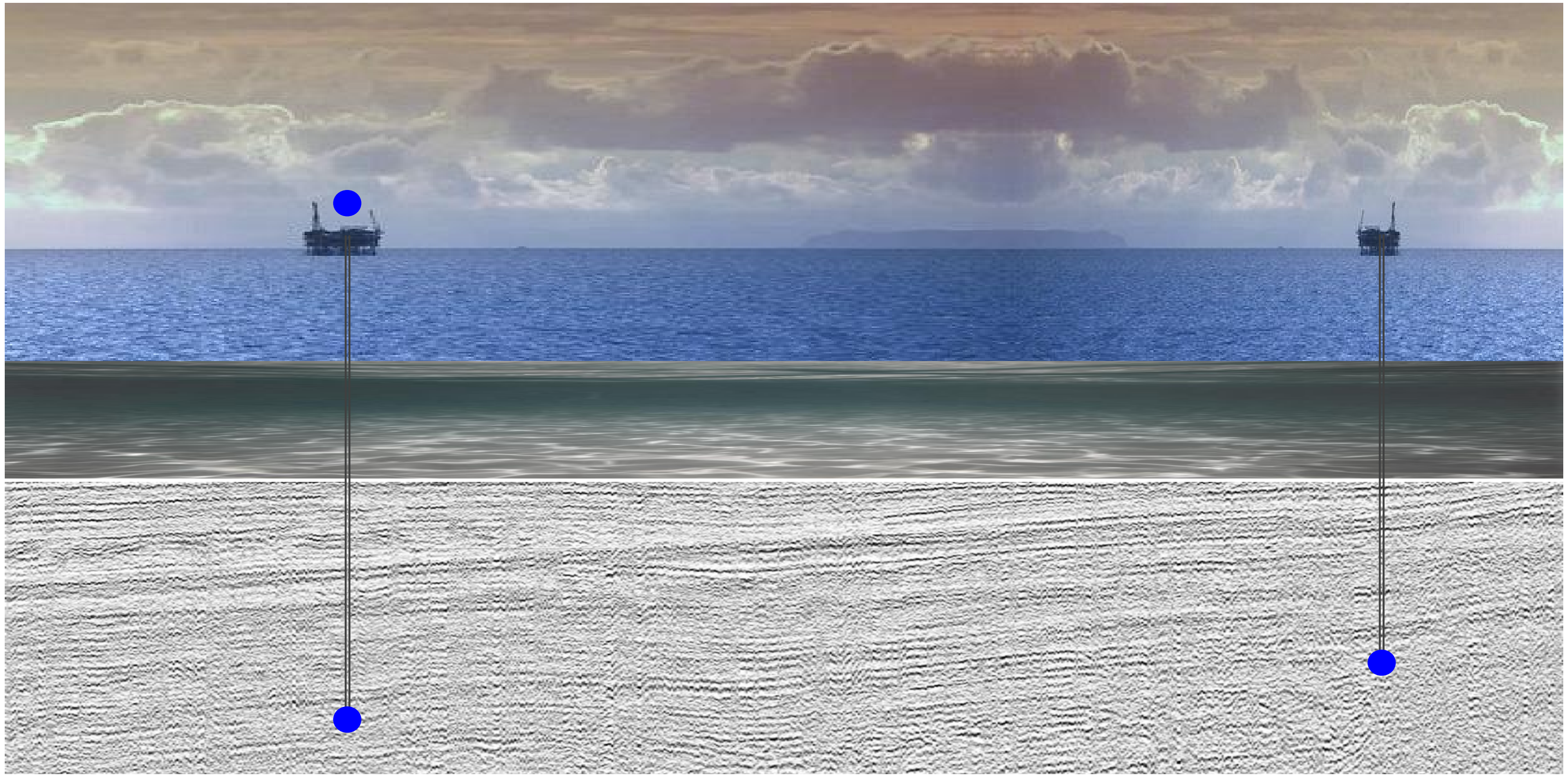
Tracer Loading and Release Mechanism

- Release
  - Release stimuli can be Temperature, pH, etc
  - Nanogel placed under release conditions





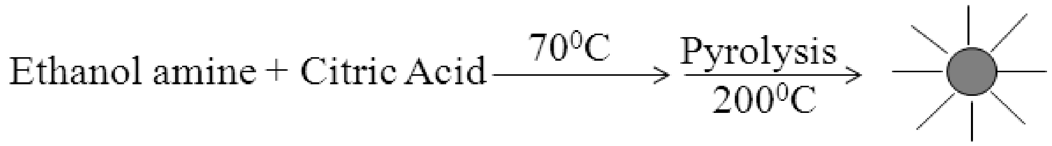
Inter-well Chemical Tracer Tests

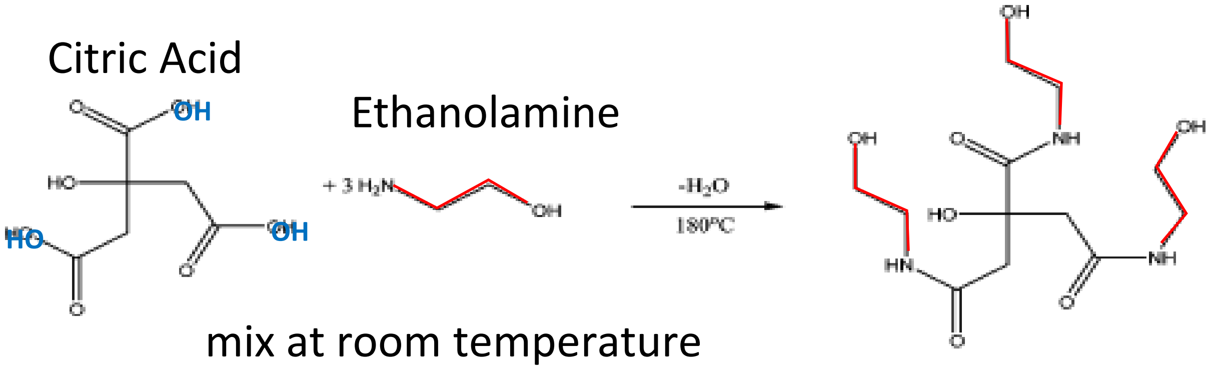


- Flow Directions
- Pathways
- Connectivity
- Heterogeneity
- Velocities
- Swept volumes
- Permeability

C-Dots

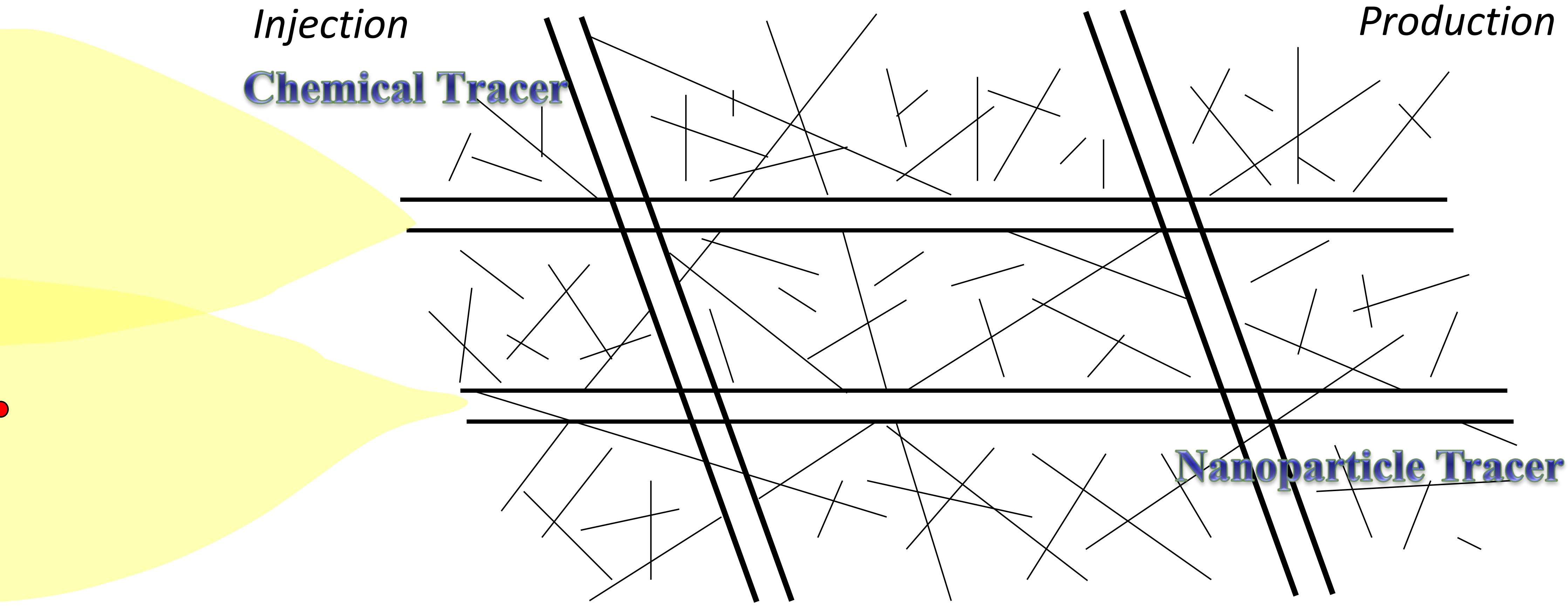
- Carbogenic (C) nanoparticles
- Fluorescent nature
- Particle Size ~ 5nm
- Simple synthesis protocol





Concept

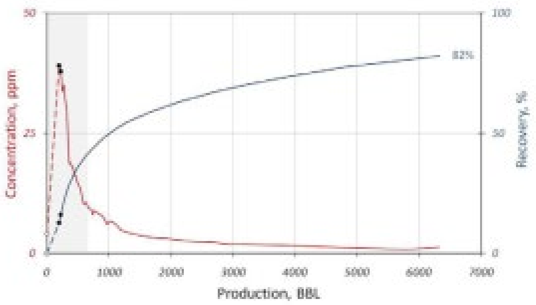
Fast-diffusing inert chemical tracers (**He/HTO**) **+** slow-diffusing inert particle tracers (**C-dots**)
2-well tracer tests degree of fracture control on fluid flow.



CDots – Field Test

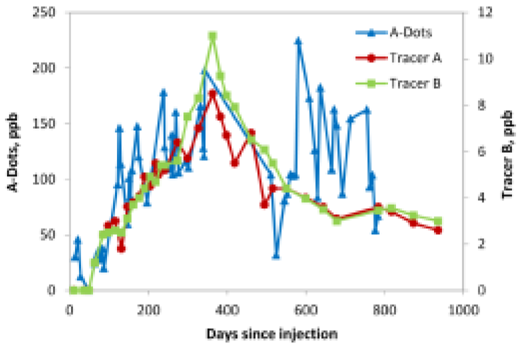
Single-Well Tracer Test : *push - hold – pull* 2011:

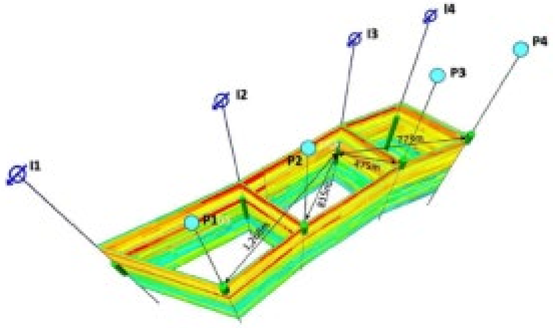
Kanj, M.Y., M.H. Rashid, and E. Giannelis, ***Industry First Field Trial of Reservoir Nanoagents***. Society of Petroleum Engineers.



Inter-Well Tracer Test 2016:

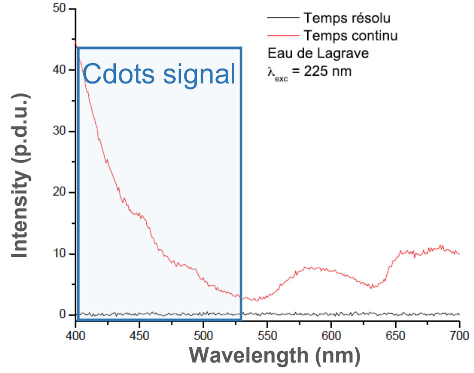
Kosynkin, D. and M. Alaskar, ***Oil Industry First Interwell Trial of Reservoir Nanoagent Tracers***. Society of Petroleum Engineers.

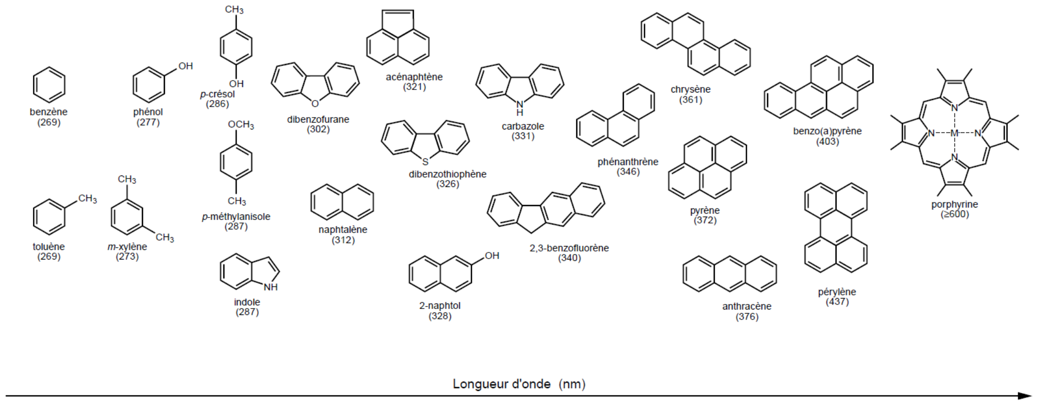




C-Dots – Challenges - Fluorescence

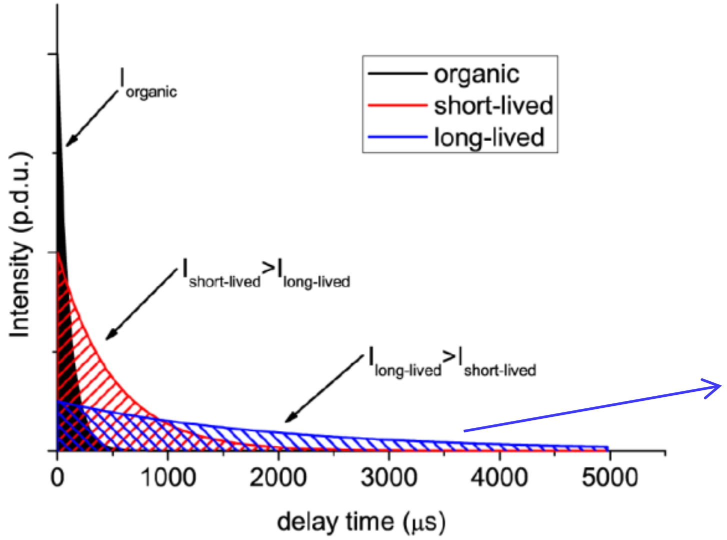
Emission/excitation interference from oil in production water



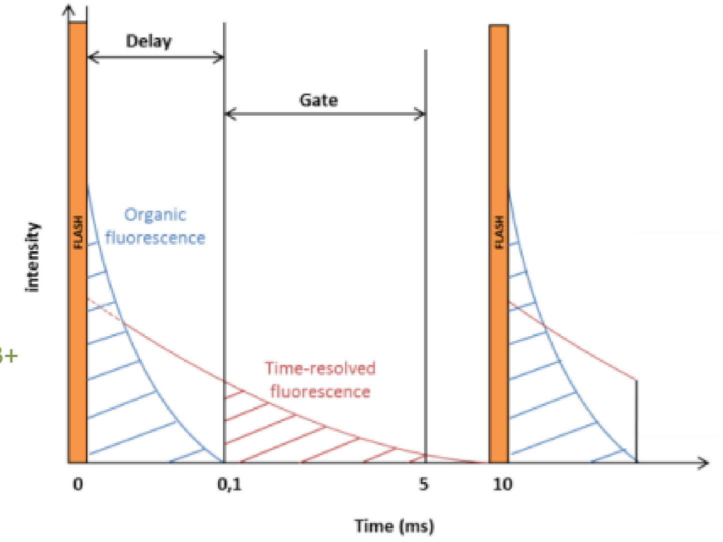


Brichart, T. (2014). **Traceurs fluorescents à base de lanthanides en milieu complexe.**, PhD thesis University Claude-Bernard Lyon1, defended on the 7 July 2014.

Emission lifetime of organic compounds and lanthanides: Time-resolved fluorescence

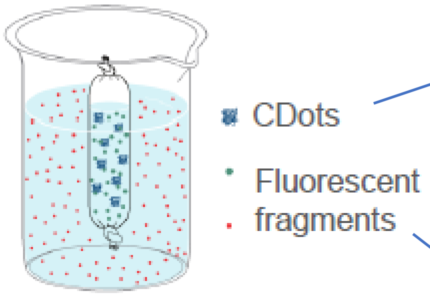


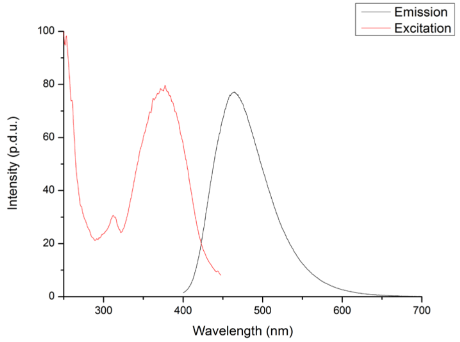
Eu^{3+} Gd^{3+} Tb^{3+} Dy^{3+}

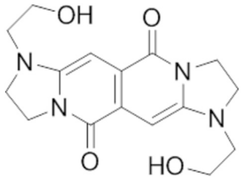


C-Dots – Challenges - Purification

Dialysis: 1 kDa (<0.1 nm)

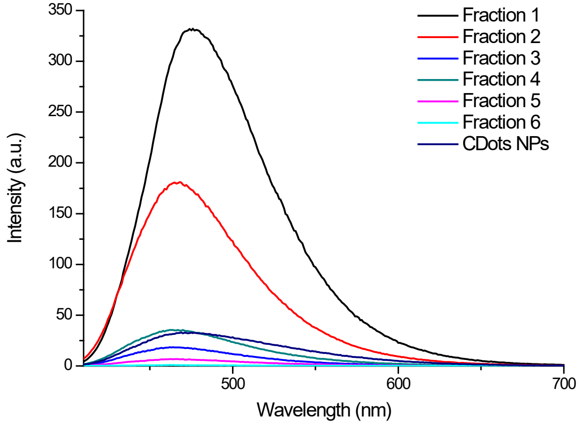






Alaskar, M. and D. Kosynkin (2017). "Successful crosswell field test of fluorescent carbogenic nanoparticles." Journal of Petroleum Science and Engineering 159: 443-450.

Fluorescent byproduct in the washing volumes



Future Works

- Core Flooding experiments on Nanogels

- Experimenting other potential nanocarriers
  - Polymeric Vesicles (PolyButaiene-co-PolyEthyleneGlycol)
  - Nanogels based on Poly(ethylene glycol)-b-poly(methacrylic acid)
  - Mesoporous Silica nanoparticles

- Summarizing our work on C-Dots

Acknowledgements

Research Partners

- With funding from The Research Council of Norway
- Norwegian Petroleum Directorate
- petoro
- Universitetet i Stavanger
- IFE
- The National IOR Centre of Norway

Industry Partners

- Neptune Energy
- Lundin Norway
- wintershall dea
- AkerBP
- ConocoPhillips
- Halliburton
- Schlumberger
- equinor
- eni
- vår energi

Inter-Well Partitioning Tracers for Water-Flooded Reservoirs: Development and Potential

Mário Silva

Research Scientist, Tracer Technology Department, IFE

(Summary of PhD Thesis UiS 578)

Brief History of the PITT

• Partitioning Inter-well Tracer Test (PITT) is introduced to the oil industry. (Cooke, C.E.J., US Patent 3,590,923)	**1971**
• Improvement of interpretation of production curves. (Deans, H, SPE-7076-MS)	**1978**
• Improvement of interpretation of production curves. (Tang, J. & Harker, B., PETSOC-91-04-01 30)	**1991**
• Improvement of interpretation of production curves. (Tang, J., PETSOC-91-04-01 31)	**1992**
• A new class of partitioning tracers is introduced. (Viig, S., et al., SPE-164059-MS)	**2013**

S_{OR} and IOR

The importance of S_{OR} for IOR:

- Identification of IOR targets
- Design of IOR projects
- Evaluation of IOR/EOR operations
- Optimisation of reservoir exploitation methodologies

Determination of S_{OR} on the field:

Single-well chemical tracer test (SWCTT)	Partitioning inter-well tracer test (PITT)
• Routinely used • Yields results in a few weeks • Samples a small volume (a few meters around the well)	• Few tests performed • Yields results after months • Samples the whole swept volume

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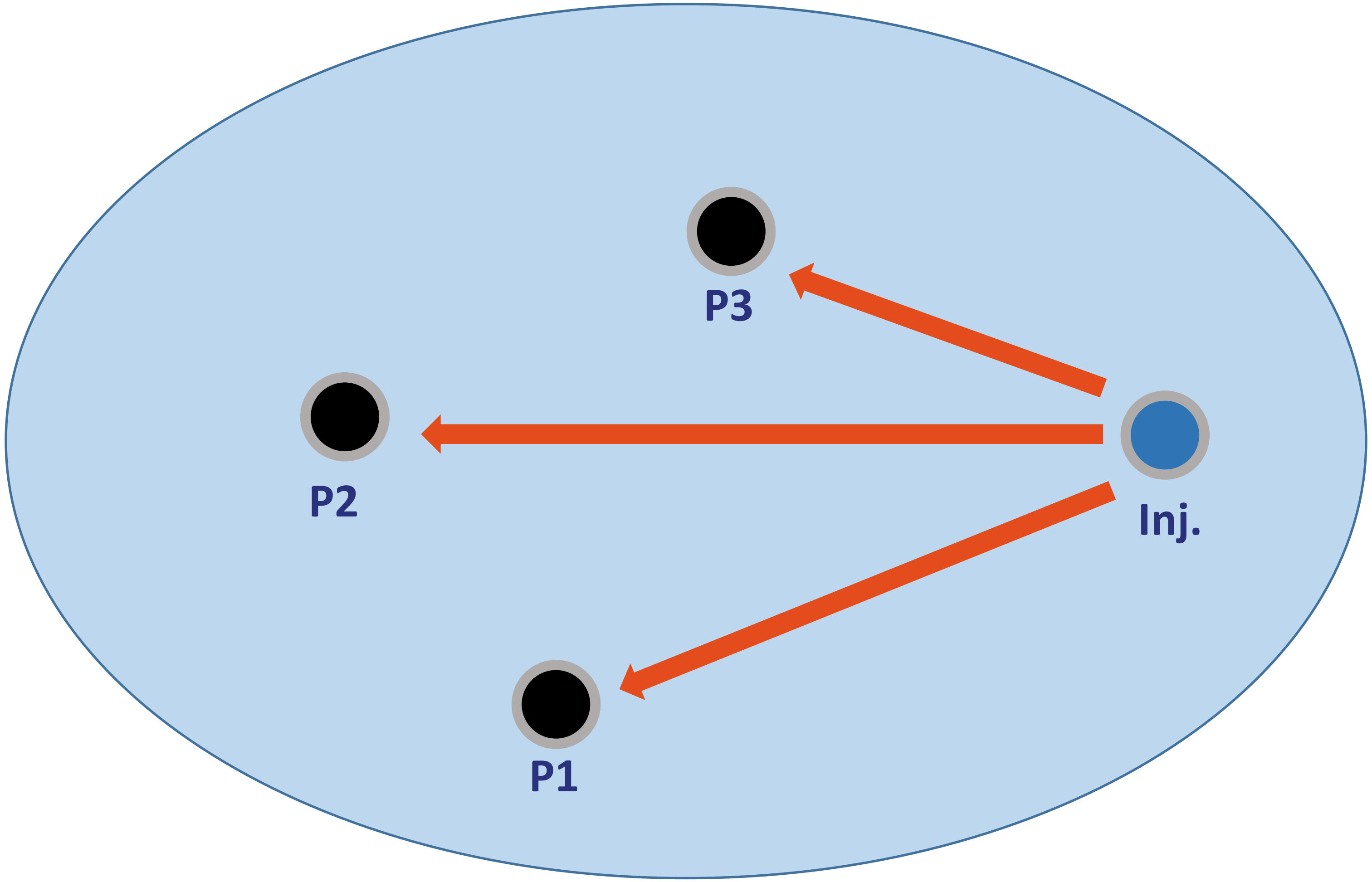
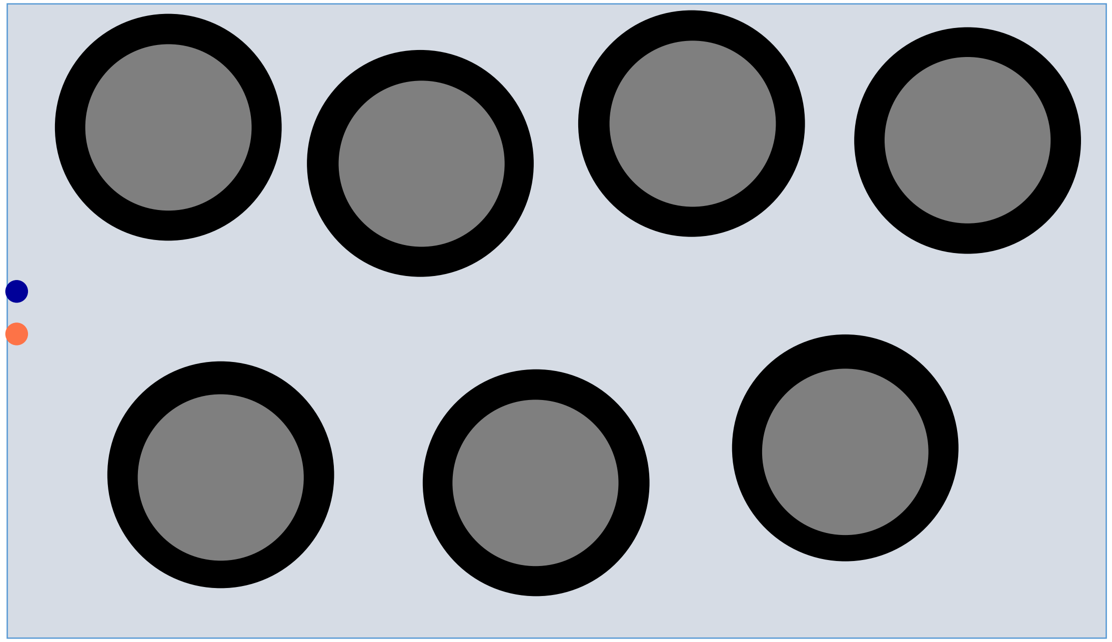
Visualising the PITT

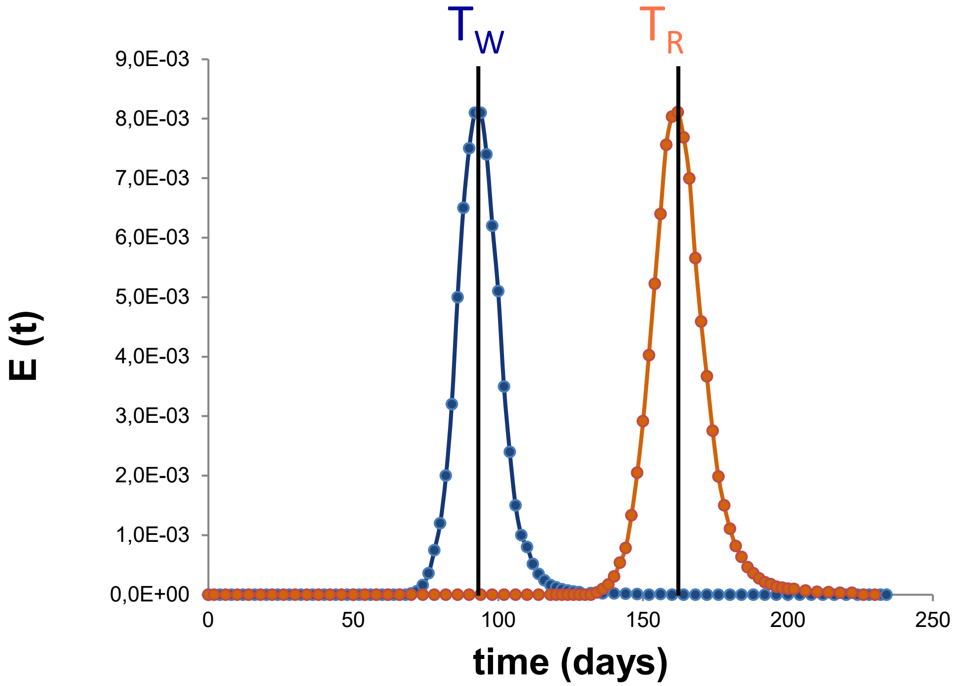


- The whole water-flooded volume is sampled.
- The different flow paths are characterised.
- Different IOR strategies can be tailored/evaluated.

Visualising the PITT

- Partitioning Tracer
- Passive Tracer





$$S_{OR} = \frac{T_R - T_W}{T_R + T_W(K-1)}$$

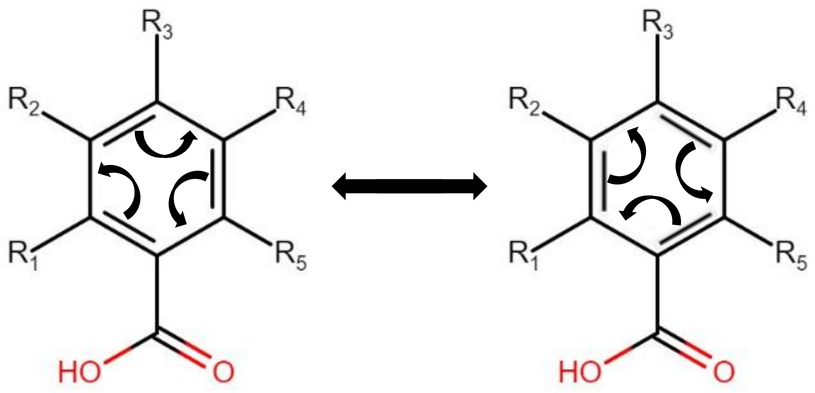
The National IOR Centre of Norway

Oil/water partitioning tracer candidates

- Main requirements for inter-well water-based tracers:
  - Long thermal, chemical and biological stability;
  - Absence of significant interactions with reservoir rocks;
  - A passive tracer must follow exclusively the aqueous phase in which it is injected
  - The tracer must not modify reservoir fluids;
  - Absence from any fluid present in the reservoir (eventually, presence in low and constant concentrations is acceptable);
  - Analysable in low concentrations (low µg/L – ng/L range)
  - Must be environmentally acceptable;
  - Must be commercially available at acceptable prices;

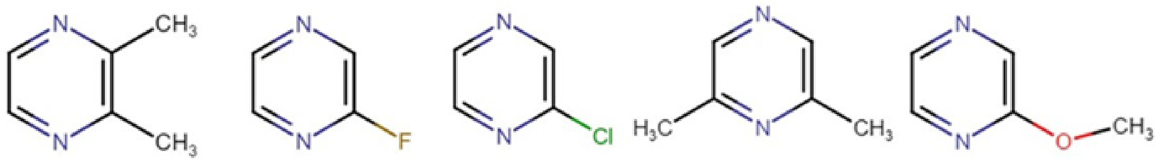
Oil/water partitioning tracer candidates

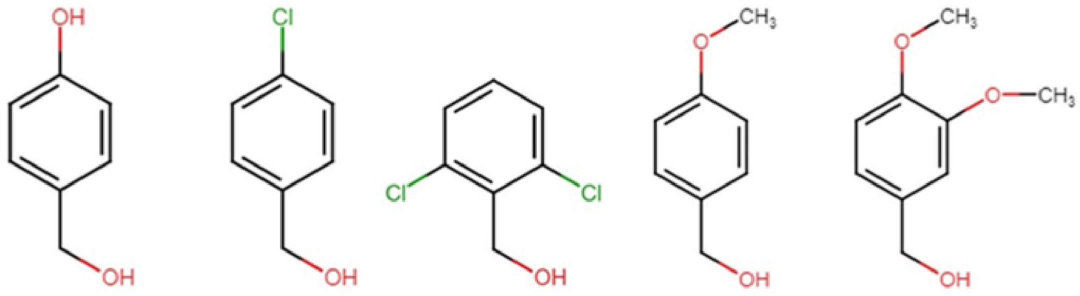
- Selection of oil/water partitioning tracer candidates:
  - The substances must be non-ionic under reservoir conditions (pH dependence is acceptable)
  - The estimated K-value should be within boundaries (2 – 5 is a good indication). K can be “tunned” with different substituents in a molecule.
  - Chemical structure of the molecules (i.e., chemical resonance is a good indicator of chemical stability)
  - Analysability of the candidates
  - Commercial availability



Oil/water partitioning tracer candidates

- 16 molecules from 4 chemical families were selected for testing:

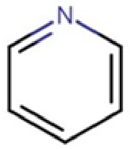
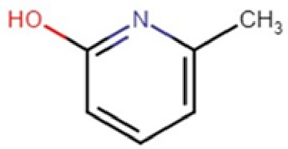
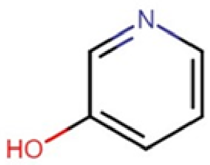
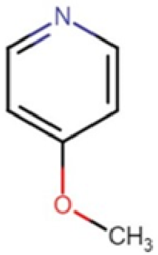


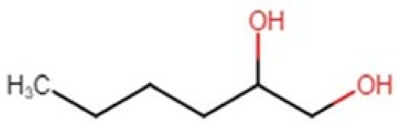


- Pyrazines are potent scent and flavour additives, never described in any reservoir fluid.
- The benzyl alcohols investigated have little industrial use and were never described in any reservoir fluid.

Oil/water partitioning tracer candidates

- 16 molecules from 4 chemical families were selected for testing:

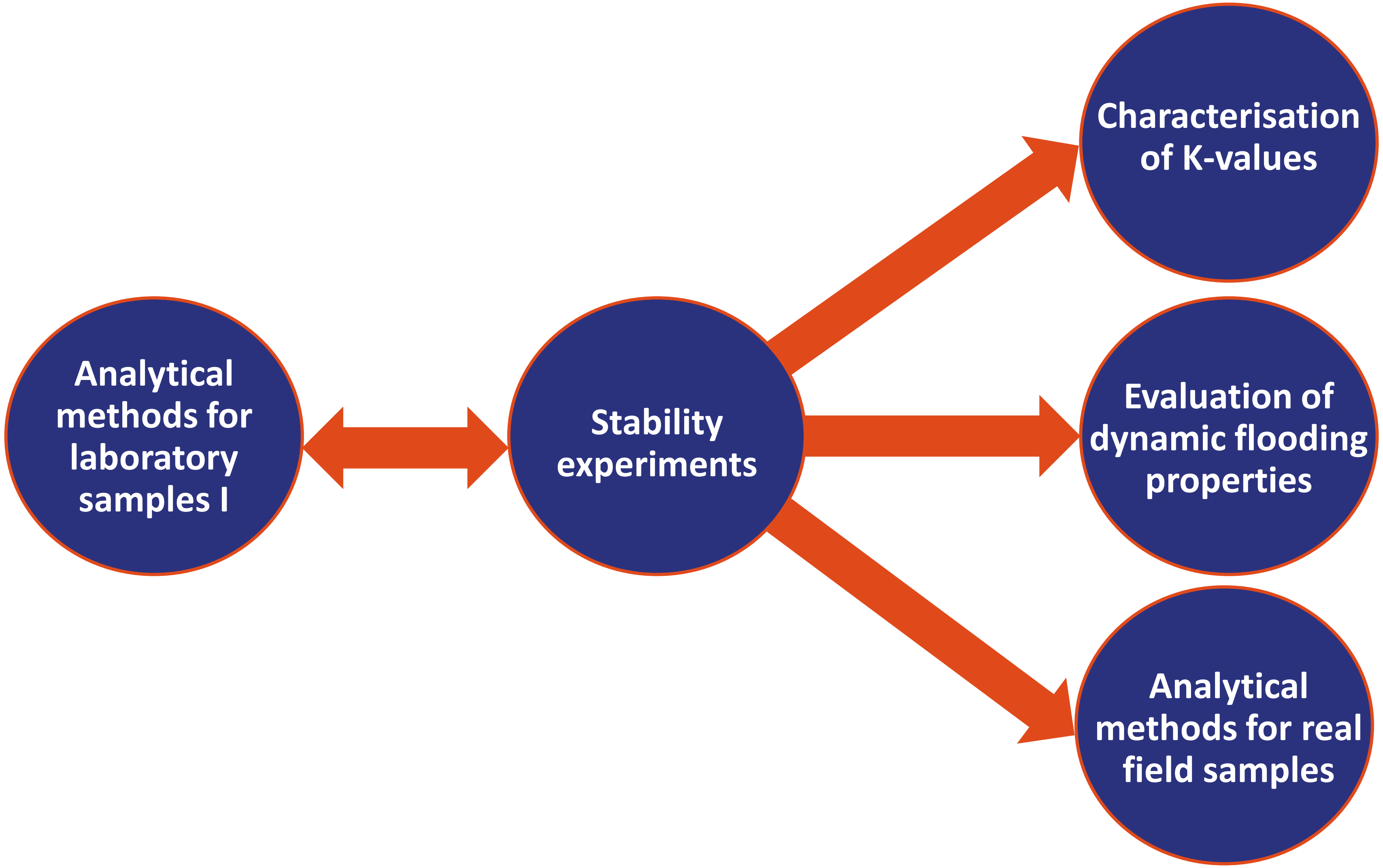








- Pyridine has been described as constituent of oils. These candidates exhibit pH dependant ionic forms.

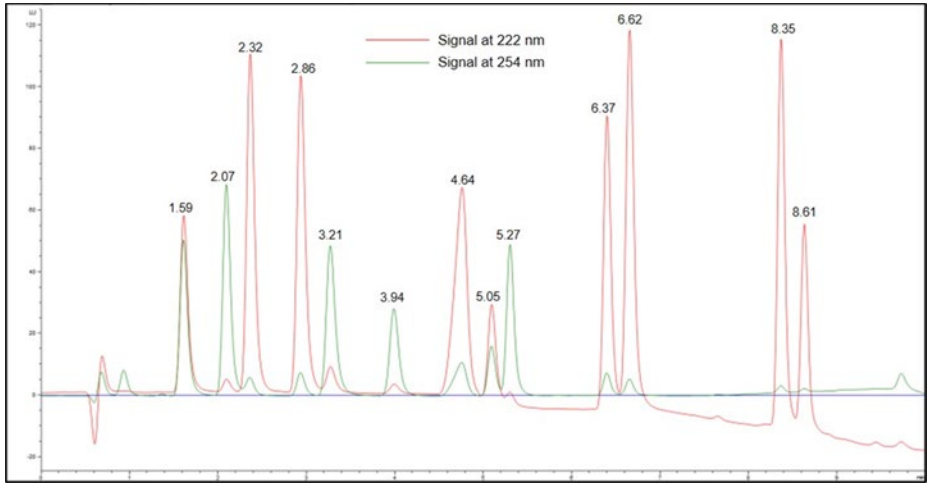
- These diols were selected from earlier reported PITTs and have not been described in any reservoir fluid.

Experimental Approach

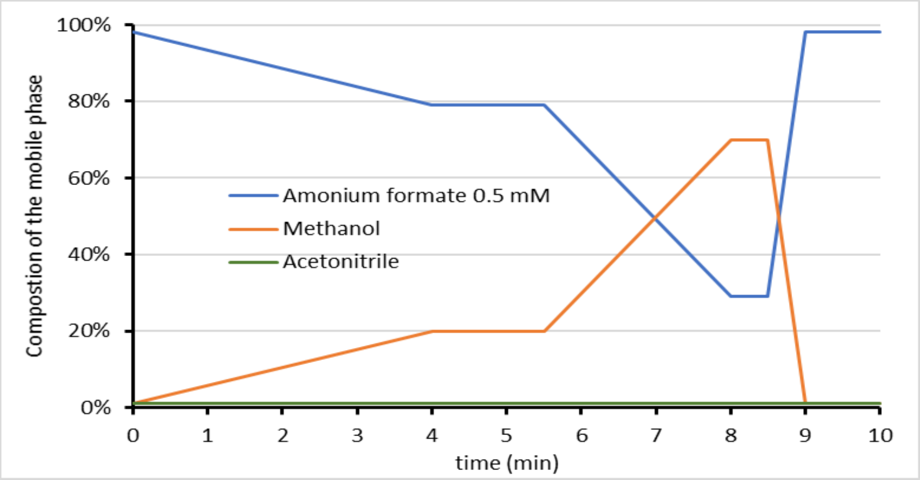


Experimental Approach

- Analytical methods for laboratory samples:



Example chromatogram

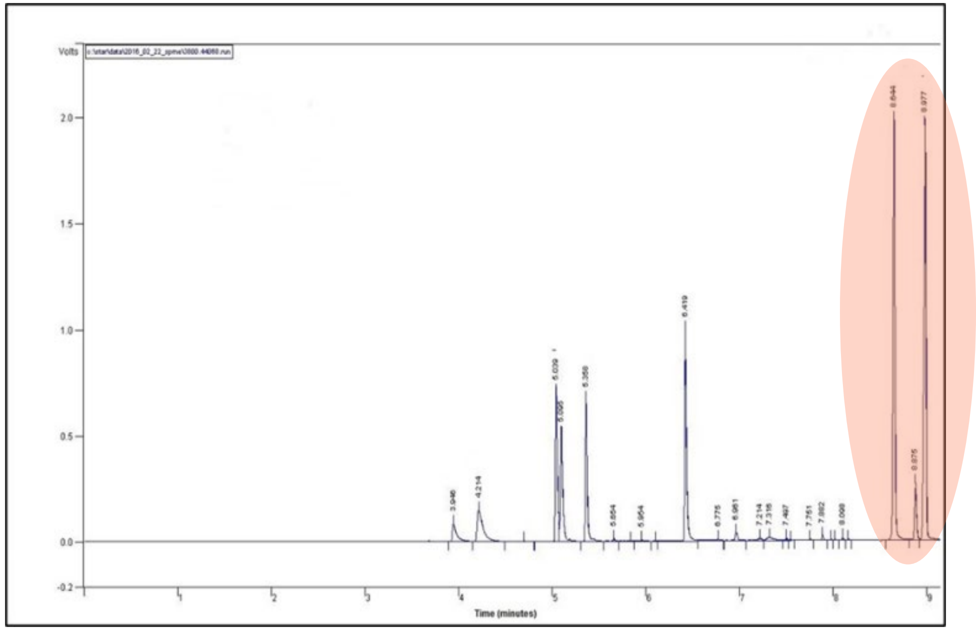


Gradient vs time

- Method developed: 10 µL of sample, mobile phase flow of 0.5 mL/min with gradient elution on a RP C18 1.7 µm packed column at 60 ℃.
- Effective for analysis of 14 of the 16 tracer candidates.

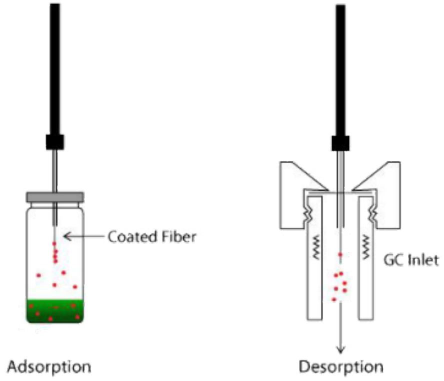
Experimental Approach

- Analytical methods for laboratory samples:



Example chromatogram

Extraction	GC-FID Analysis
• PDMS-DVB fibre • 80 ℃ • 10 min headspace • Sample saturated with NaCl	• HT5 capillary column • Oven with 30 ℃/min from 50 ℃ to 250 ℃ • Fibre desorption for 5 min at 250 ℃ • Helium as carrier at 1 mL/min • Detector at 300 ℃



Sample pre-treatment: solid phase microextraction (SPME)

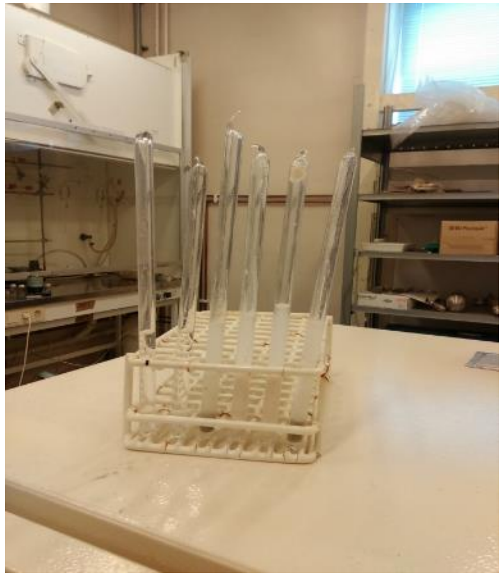
Stability Experiments

- Mixed tracer solutions with concentration of 10 mg/L were prepared in a synthetic brine;
- 3 different pHs were tested (5,5; 7,1; 8.0)
- The solutions were sonicated and sparged with argon
- 2 mL were sealed in tubes with no rock materials and with 600 mg of powdered kaolinite, Berea sandstone and limestone (125 µm – 250 µm)

Composition of the brine with the tracer candidates dissolved

Salt	Concentration (g/L)
NaCl	36,855
KCl	0,629
$CaCl_2.2H_2O$	3,814
$MgCl_2.6H_2O$	2,550
$BaCl_2.2H_2O$	0,088
$SrCl_2.6H_2O$	0,437
$NaHCO_3$	0,157
Na_2SO_4	0,046

Stability Experiments



- The samples were incubated for 12 weeks at temperatures 25 ℃ – 150 ℃.
- Samples were analysed after 1, 3, 6 and 12 weeks. All tests performed in triplicate.

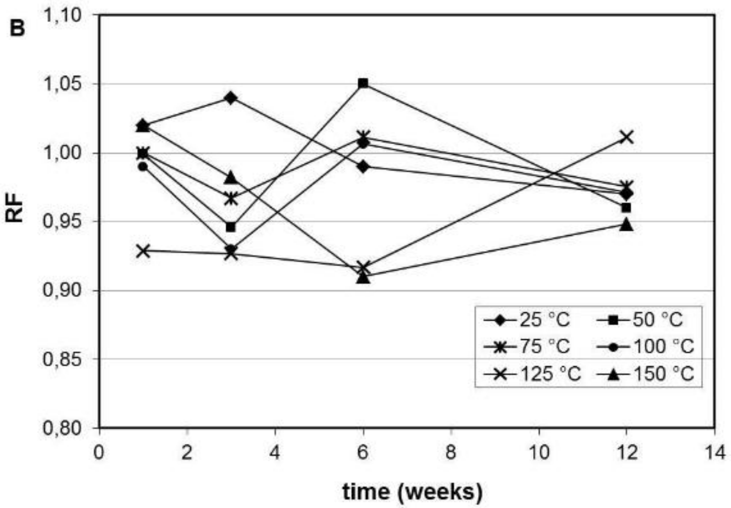
Stability Experiments

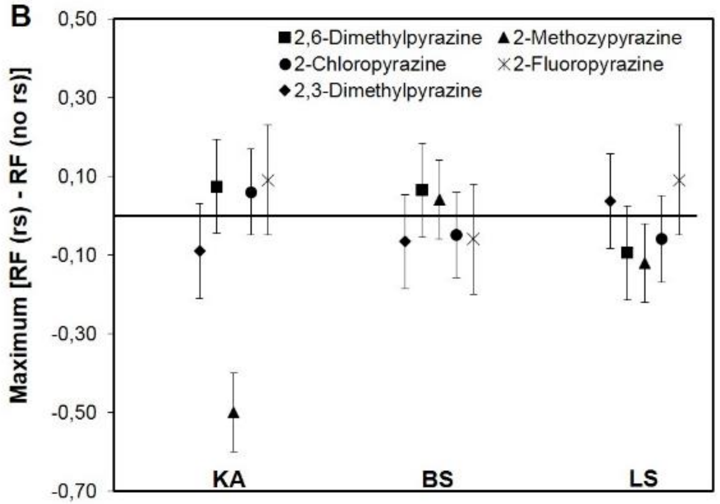
- Results can be divided into 4 groups:

Stability Experiments

- **Fully Stable compounds**

$$RF = \frac{[T_R]_{(t,T)}}{[T_R]_{(initial)}}$$





rs – rock substrate

KA – kaolinite

BS – Berea sandstone

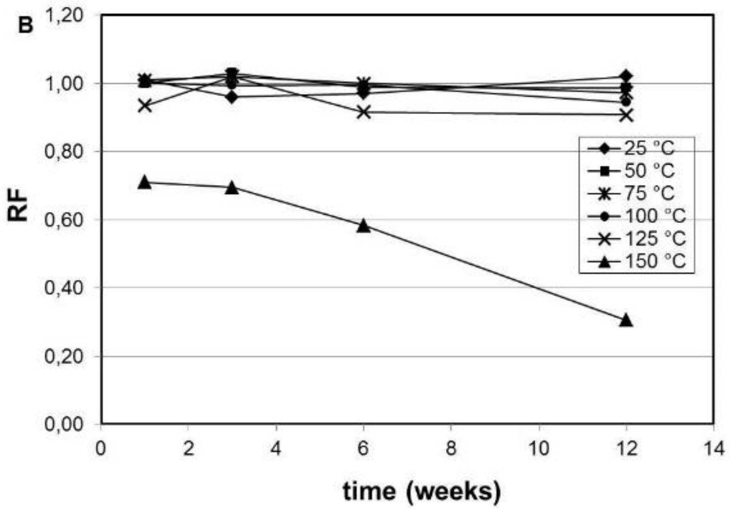
LS – limestone

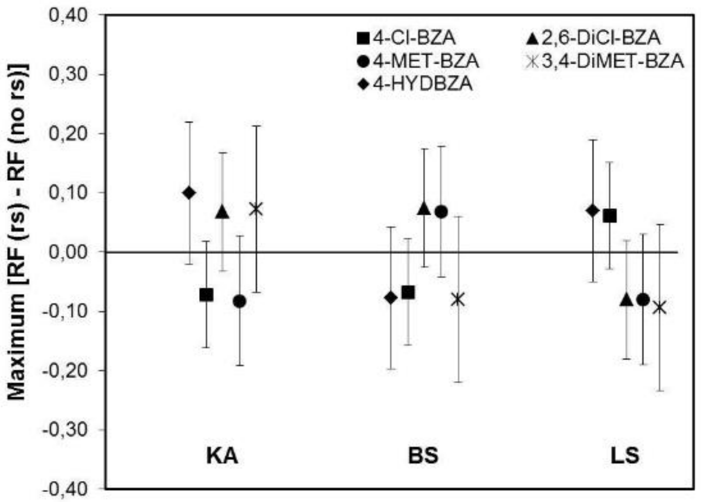
- Results at pH 7,1 for 2,3-dimethylpyrazine (from paper IV)

- No thermal degradation, no significant interaction with rock substrates

Stability Experiments

- **Compounds with limited stability**



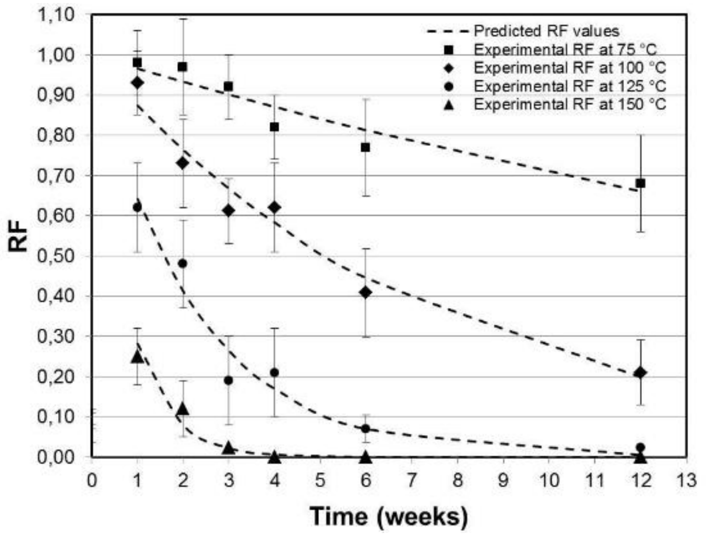


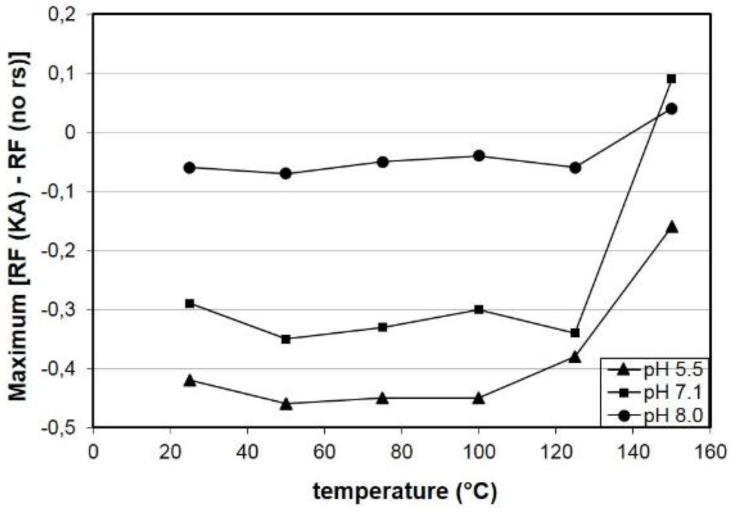
  - Results at pH 7,1 for 3,4-dimethoxybenzyl alcohol (from paper III)

- Sudden onset of thermally driven degradation
- No significant interaction with rock substrates

Stability Experiments

- **Compounds with other potential uses**



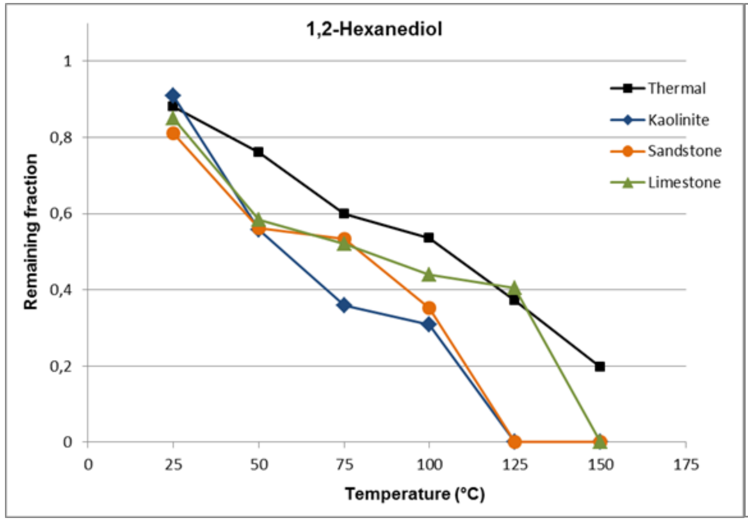


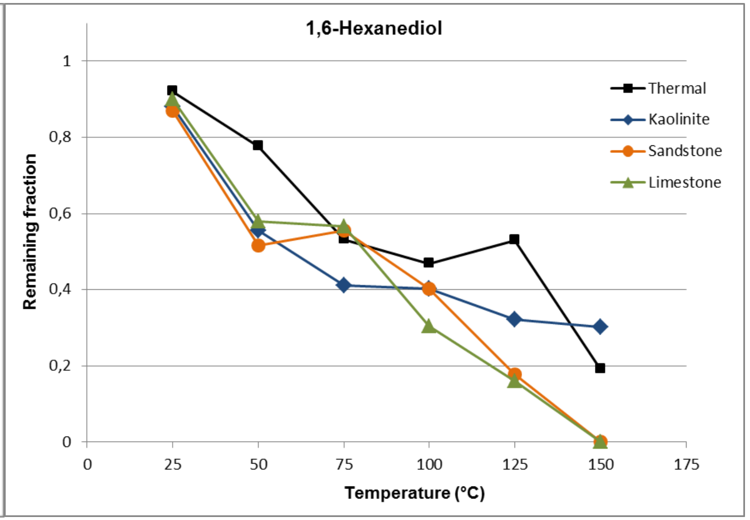
- Experimental vs predicted RF values of 4-hydroxybenzyl alcohol (paper III)
- Influence of kaolinite, pH and temperature on 3-Hydroxypyridine (paper V)

- 4-Hydroxybenzyl alcohol, a temperature probe (pseudo-first order degradation kinetics)?
- 3-Hydroxypyridine, a possible geochemical “probe”?

Stability Experiments

- **Compounds with no apparent interest as tracers**

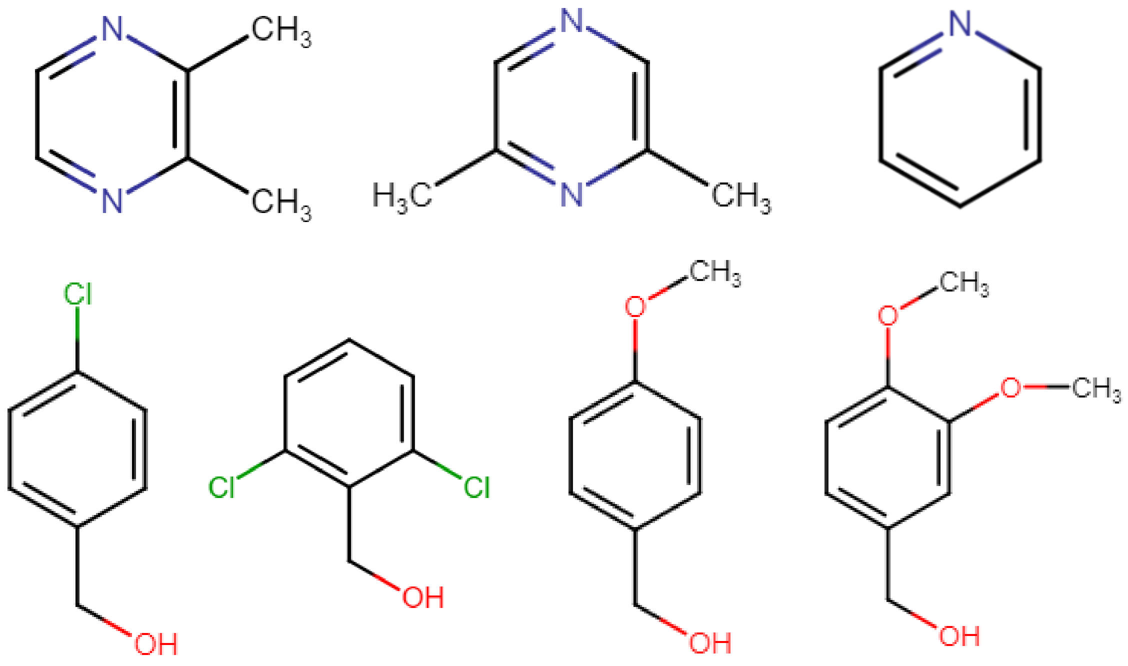




- RF of 1,2 and 1,6-hexanediol as a function of temperature after 12 weeks with and without rock substrates.

Stability Experiments

- **Summary:**
- 7 tested molecules were investigated further as potential oil/water partitioning tracers
- The other 9 compounds were excluded from the rest of the qualification process



- Sufficient thermal stability
- Absence of significant interactions with typical reservoir rock materials

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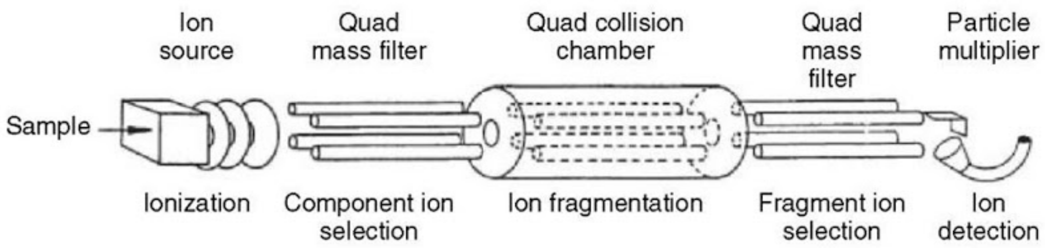
Analytical methods for field samples

- Focused on the candidates qualified by the stability experiments
- The previously developed methods were not sensitive enough: dilutions of 10^9 – 10^{12} are common in PITTs
- The previously developed methods were not selective enough (UV and FID detection provide little qualitative information, particularly in complex matrices such as produced waters)
- Several analytical and sample preparation techniques were considered
- A method consisting of SPME-GC-MS/MS was developed

Analytical methods for field samples

- Previous results suggested that SPME could be an effective pre-treatment technique
- SPME reduces significantly labour and use of solvents in the laboratory
- Optimisation of a selective reaction monitoring SRM quantification method was performed (EI mode)



Simplified triple quadrupole scheme

Compound	MS/MS transitions (identification)	MS/MS transitions (quantification)	CE (eV)
Pyridine	52 → 39 79 → 52	79 → 52	30
2,3-Dimethylpyrazine	40 → 39 67 → 52 108 → 93	108 → 93	25
4-Chlorobenzyl alcohol	77 → 75 107 → 90 142 → 125	142 → 125	20
2,6-Dichlorobenzyl alcohol	113 → 77 141 → 123 176 → 159	113 → 77	20
4-Methoxybenzyl alcohol	109 → 95 121 → 90 138 → 107	138 → 107	25
3,4-Dimethoxybenzyl alcohol	139 → 95 151 → 120 168 → 137	168 → 137	25

Analytical methods for field samples

- Validation of the developed method

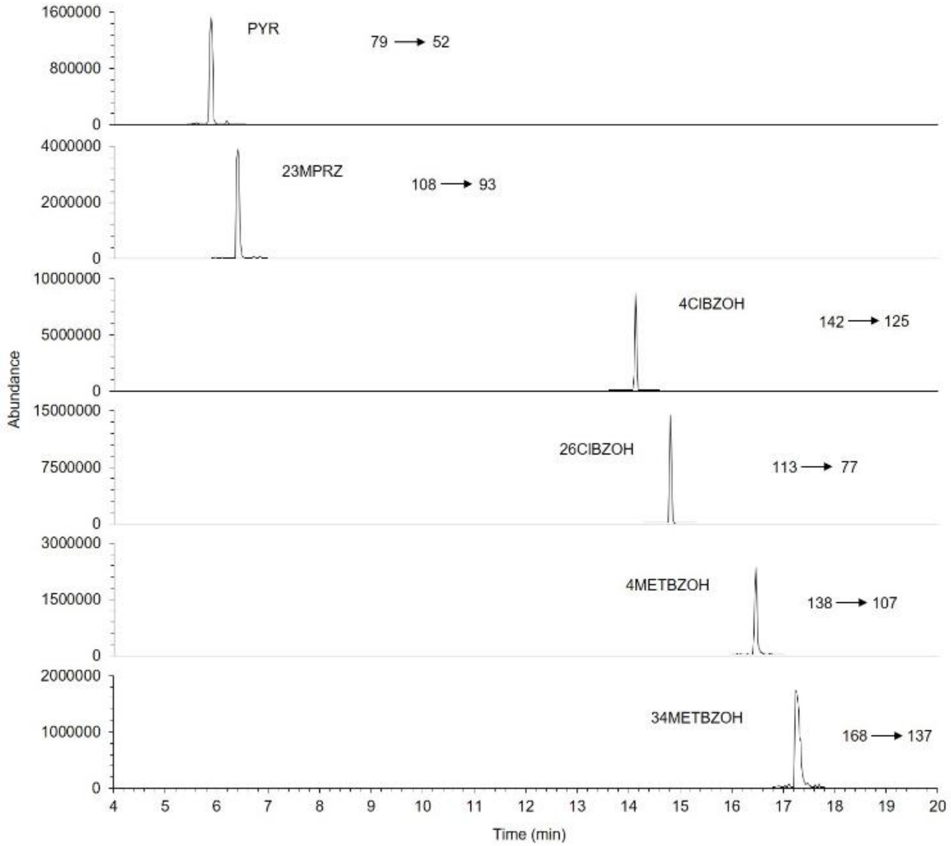
- LOQs, linear range, intra-day and inter-day precision, and recovery were evaluated
- Production waters from 8 different Norwegian continental shelf oilfields (Snorre A, Snorre B, Ekofisk M, Gullfaks C, Heidrun A, Eldfisk A, Eldfisk S and Vigdis B) were used.
- All parameters produced acceptable results

Tracers					Precision (% RSD)						
	Linearity				Intra-day precision				Inter-day precision		
	Range[a]	R^2	LoQ[a]		0.50[a]	2.5[a]	10[a]		0.50[a]	2.5[a]	10[a]
PYR	0.50 – 10	0.9995	0.35		7.9	6.6	6.2		9.4	7.4	7.3
23MPRZ	0.30 – 50	0.9970	0.20		4.7	5.3	4.2		7.1	7.2	5.3
4BZOH	0.10 – 20	0.9962	0.08		8.2	7.0	4.7		10	8,5	5.7
26BZOH	0.10 - 20	0.9971	0.10		6.2	7.1	4.2		8.1	7.8	5.6
4METBZOH	0.40 – 25	0.9974	0.25		9.4	7.4	5.9		12	9.1	5.4
26METBZOH	0.40 – 25	0.9984	0.25		10	6.4	3.9		12	11	7.5

[a] $\mu g\ L^{-1}$

Analytical methods for field samples

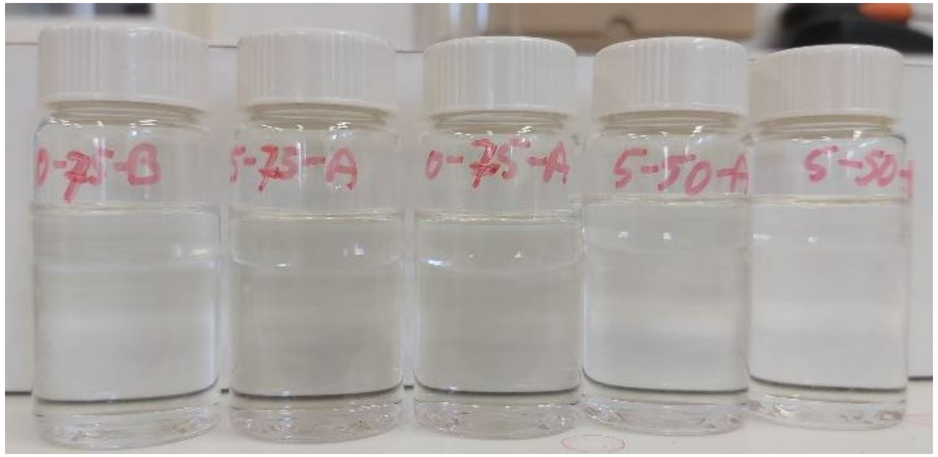
- Conditions of the DI-HS-SPME-GC-MS/MS method developed
- 5 mL of sample at pH 9.0 with 1.8 g of NaCl, constant stirring, 5 minutes of DI-SPME followed by 15 minutes of HS-SPME at 70 ℃ using a DVB/CAR/PDMS fibre.
- GC with a temperature ramp between 50 ℃ and 290 ℃ with a low polarity column “Restek Rtx®-5MS” column (30 m X 0.25 mm X 0.25 µm)
- MS operated in EI mode and SRM for quantification.



Reconstructed SRM chromatogram of produced water from Ekofisk M spiked at 1 µg L^{-1} with the tracer candidates.

Characterisation of the K-value

- The so called "shake flask method" was used

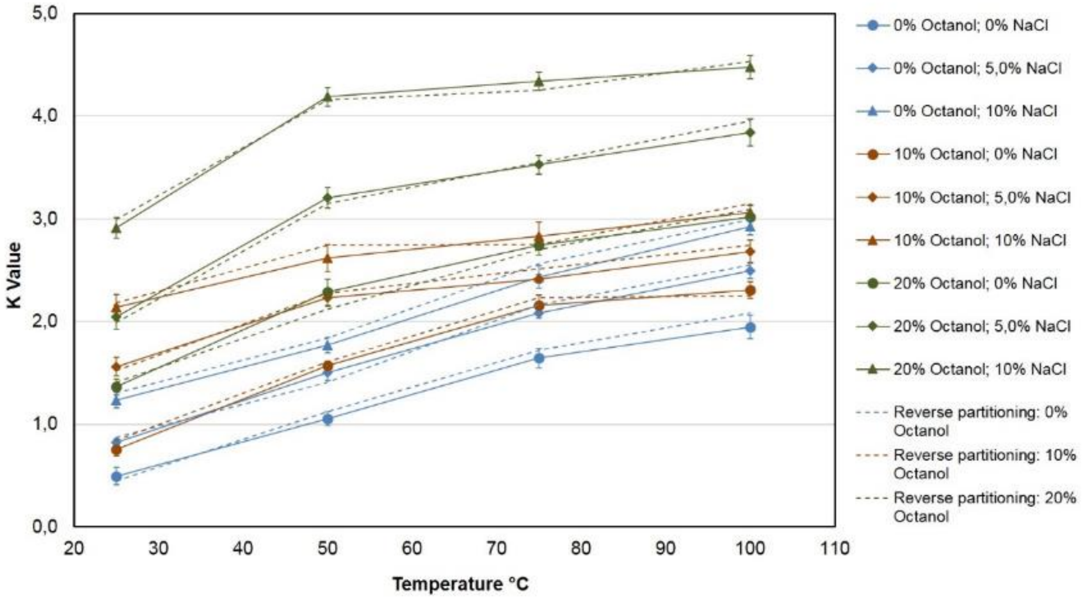


- Brines with ionic strength (I) between 0 – 1,71 M were used. Divalent vs monovalent ions were also evaluated.

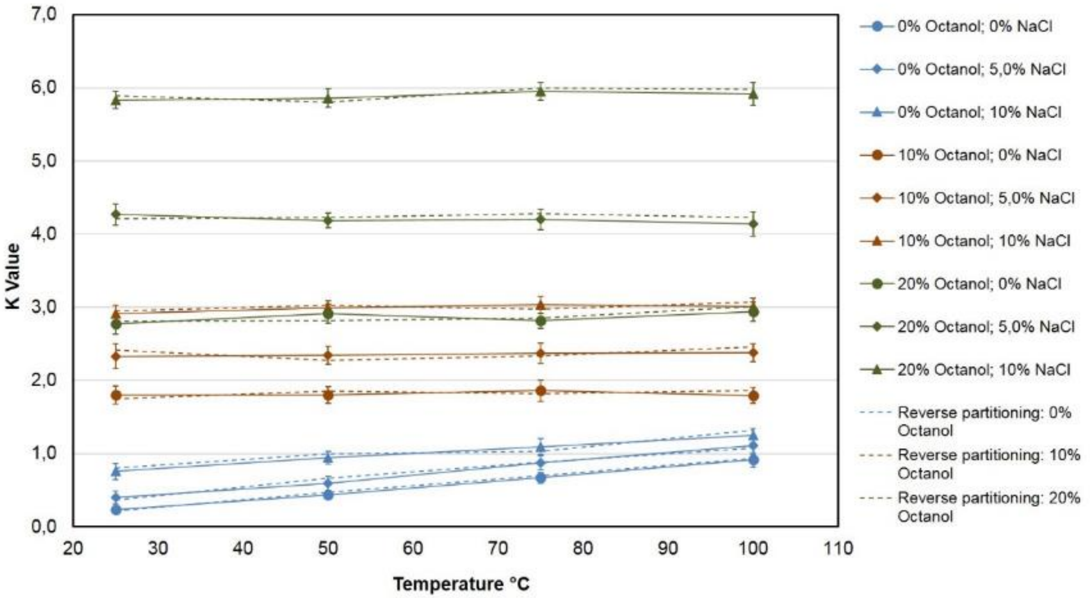
$$I = \frac{1}{2}\sum_{i=1}^{n} C_i Z_i^2$$

- Aliphatic, aromatic, polar and nonpolar characteristics of the hydrocarbons were evaluated by varying the composition of a mixture of hydrocarbons (isooctane, toluene and 1-octanol).
- Tests with real oils were performed.
- Temperature effects up to 100 °C were evaluated.

Characterisation of the K-value



K values of 2,3-dimethylpyrazine ± σ (N=3) as function of temperature, composition of the hydrocarbon phase and salinity



K values of 4-methoxybenzyl alcohol ± σ (N=3) as function of temperature, composition of the hydrocarbon phase and salinity

- Temperature, ionic strength and oil composition impact the partitioning
- Reversibility of partitioning is verified

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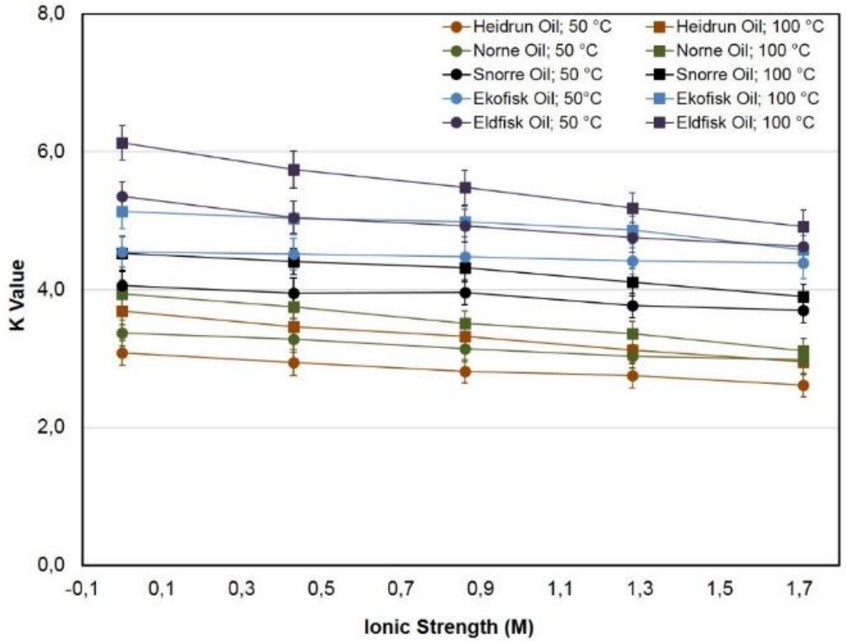
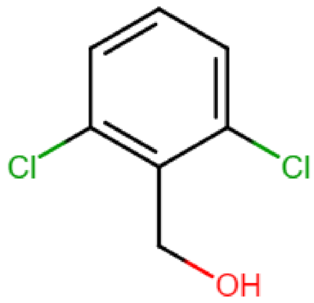
Characterisation of the K-value



K value of 2,6-dichlorobenzyl alcohol to 5 different crude oils as function of the ionic strength of the aqueous phase at 50 °C and 100 °C.

- The particular case of 2,6-dichlorobenzyl alcohol:
- Increasing salinity, reduces K
  - Possible dipole effect?
  - Density effect reducing interface transport?



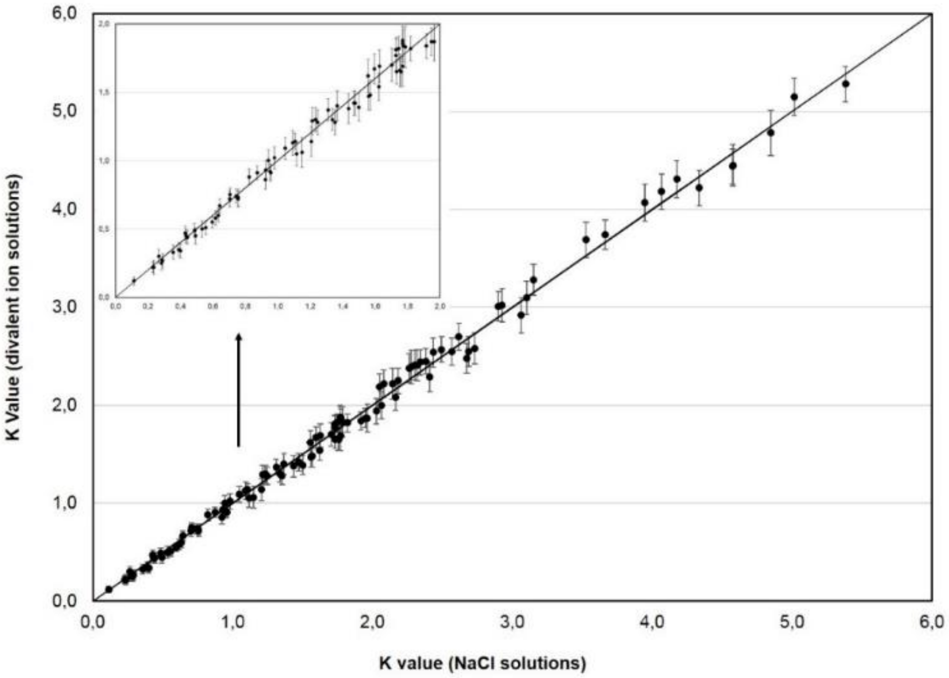
Characterisation of the K-value

- Effect of divalent ions

Salt, respective amount present, and ionic (I) strength of the aqueous solutions used to evaluate influence of divalent ions

C. NaCl (g/L)	C. NaCl (M)	C. Na_2SO_4 (g/L)	C. Na_2SO_4 (M)	C. $CaCl_2$ (g/L)	C. $CaCl_2$ (M)	C. $MgCl_2$ (g/L)	C. $MgCl_2$ (M)	I (M)
25 *	0,43	0	0	0	0	0	0	0,43
50 *	0,86	0	0	0	0	0	0	0,86
75 *	1,28	0	0	0	0	0	0	1,28
100 *	1,71	0	0	0	0	0	0	1,71
6,25	0,11	2,0	0,014	5,1	0,046	4,4	0,046	0,43
12,5	0,22	2,0	0,014	11,1	0,10	9,5	0,10	0,86
18,75	0,32	2,0	0,014	17,0	0,15	14,6	0,15	1,28
25	0,43	2,0	0,014	23,0	0,21	19,7	0,21	1,71

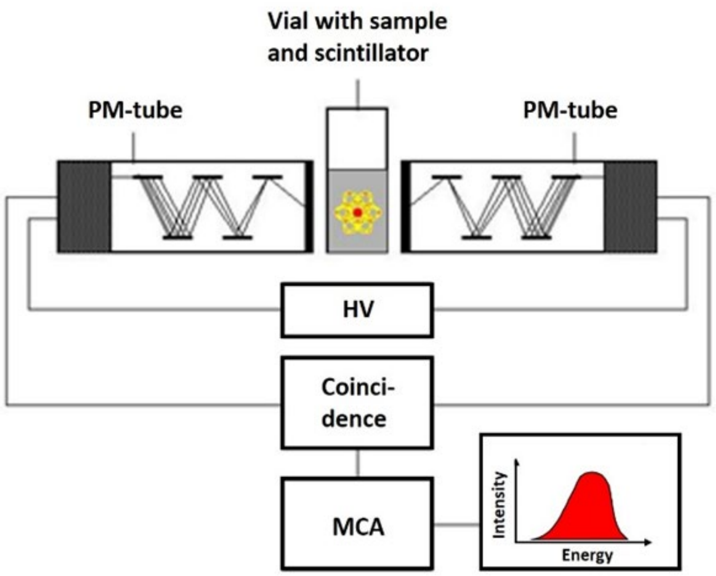
- The valency of ions does not seem to influence partitioning



K values ± σ (N=3) of the tested compounds in experiments with an aqueous phase containing divalent ions vs the K values in experiments with an aqueous phase containing only NaCl.

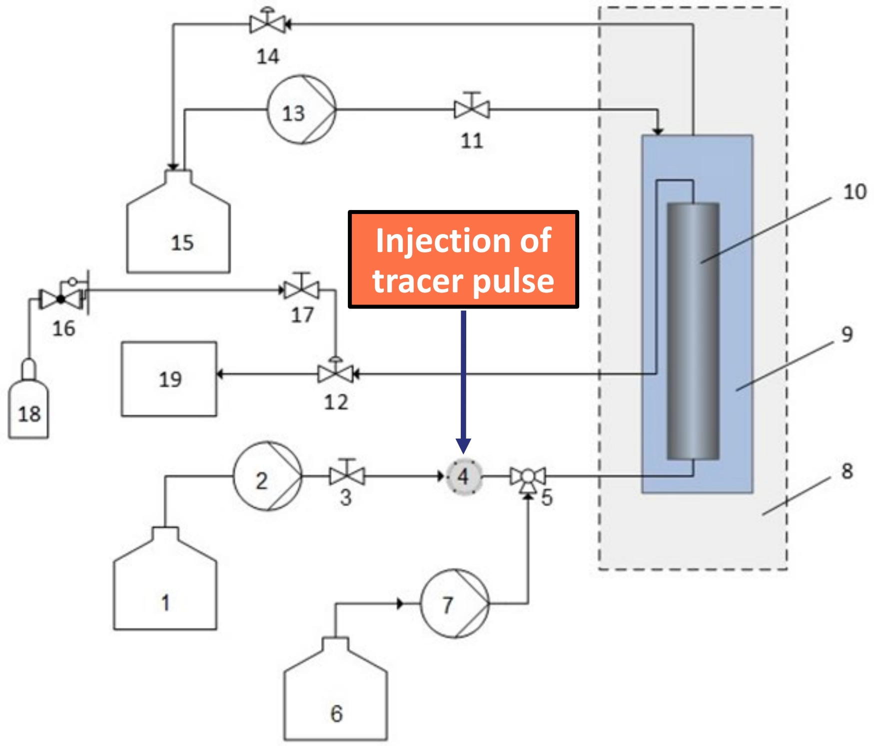
Core flooding experiments

- Performed in Berea sandstone and Stevns Klint chalk cores (38,1 mm x 70 mm)
- In water saturation and S_{OR} conditions
- Tritiated water (HTO) was used as reference water tracer



Simplified liquid scintillation counter scheme

Core flooding experiments



Scheme of the experimental setup used in the flooding experiments

1. Reservoir of injection brine
2. Brine Injection pump
3. Manual isolation valve
4. 6-way injection loop
5. 3 way plug valve
6. Reservoir of oil
7. Oil injection pump
8. Thermal cabinet
9. Hassler cell
10. Polyurethane core plug
11. Manual isolation valve
12. Back pressure regulator
13. Core plug pressure pump
14. Back pressure regulator
15. Reservoir of deionised water (for core plug pressuring)
16. Pressure regulator
17. Manual isolation valve
18. Pressurised nitrogen

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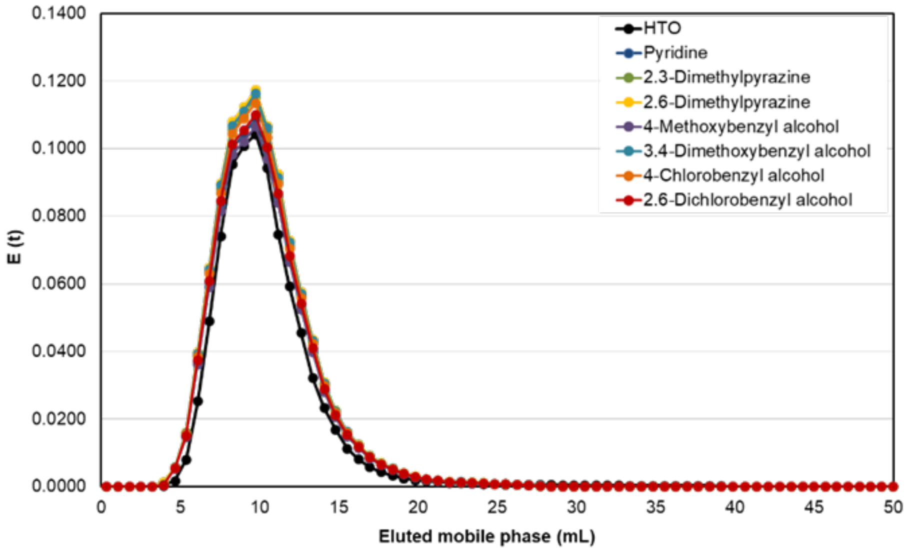
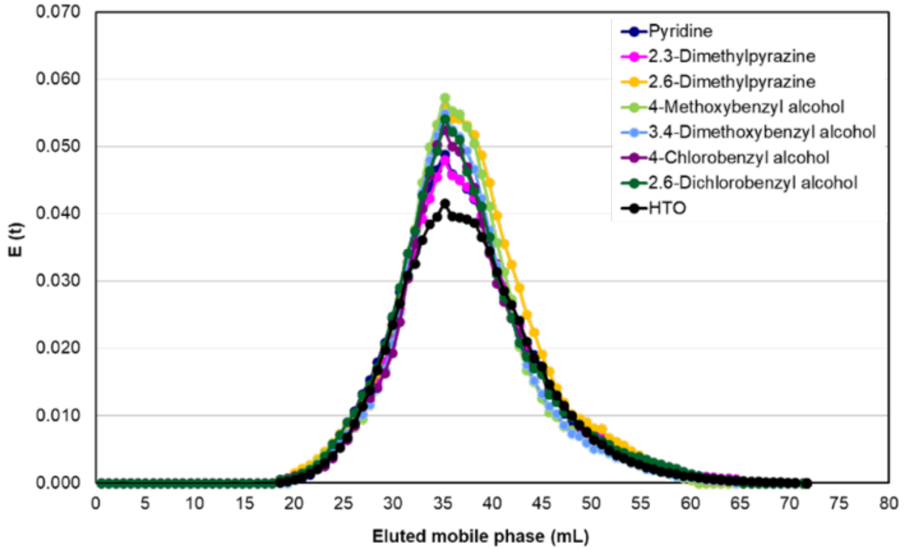
Core flooding experiments

- Water saturated cores

$$E(t) = \frac{C(t).Q(t)}{m}$$



E (t) vs eluted mobile phase in the flooding experiments with a water-saturated sandstone core.



E (t) vs eluted mobile phase in the flooding experiments with a water-saturated chalk core.

- Production curves show the same production profiles of the tracer candidates as HTO (reference tracer)

Core flooding experiments

- Water saturated cores

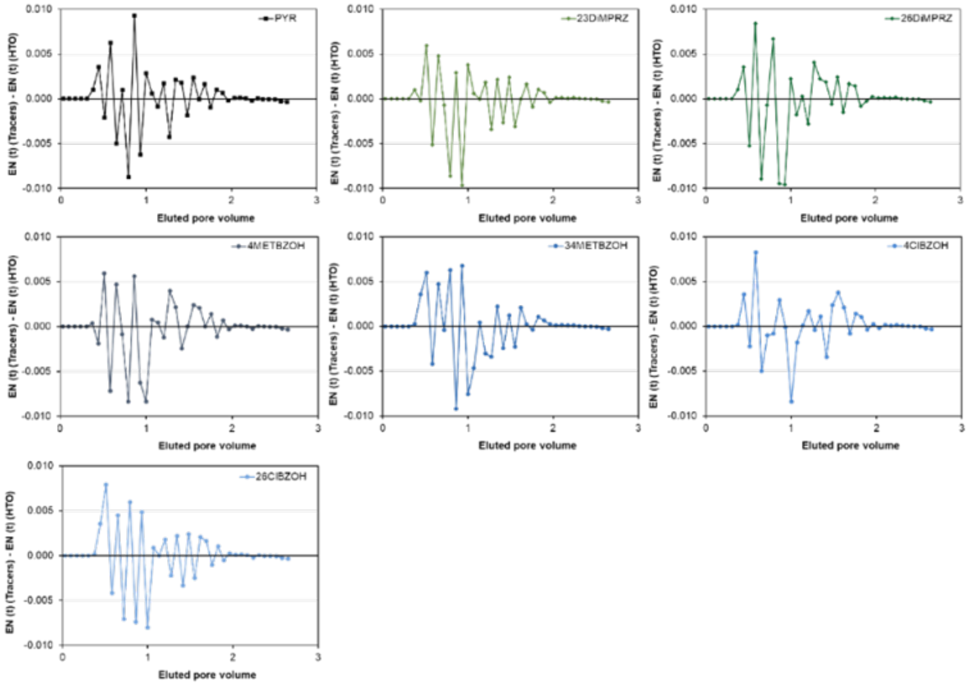
$$V_S = \frac{\sum_i^n C_i v_i Q_i}{\sum_i^n C_i Q_i}$$

	Berea sandstone core		Stevns Klint chalk core	
Tracer	Recovery (%)	Total core pore volume (mL)	Recovery (%)	Total core pore volume (mL)
HTO	86,1	10,4 ± 0,2	86,9	37,6 ± 0,4
PYR	97,8	10,3 ± 0,2	94,2	37,2 ± 0,4
23MPRZ	103	10,3 ± 0,2	91,8	37,2 ± 0,4
26MPRZ	105	10,5 ± 0,2	106	37,8 ± 0,4
4METBZOH	95,7	10,4 ± 0,2	97,7	37,4 ± 0,4
34METBZOH	104	10,5 ± 0,2	92,5	37,8 ± 0,4
4ClBZOH	102	10,3 ± 0,2	92,4	37,6 ± 0,4
26ClBZOH	98,8	10,4 ± 0,2	97,4	37,4 ± 0,4

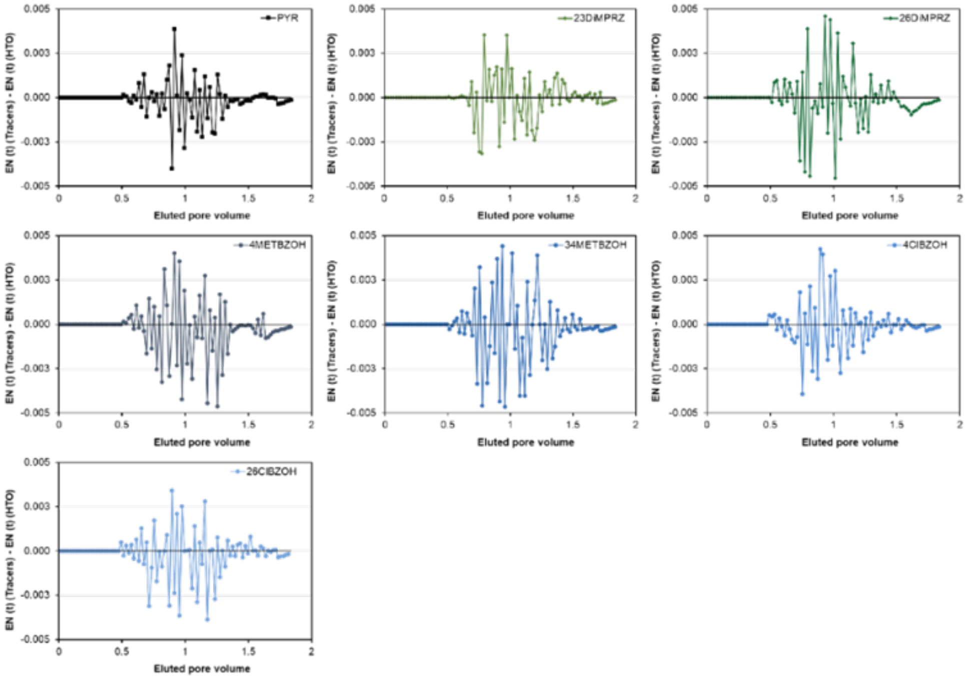
- Same pore volume measured by the tracer candidates and HTO
- The partitioning tracer candidates behave as water tracers in the absence of hydrocarbons.

Core flooding experiments

- Water saturated cores: residual analysis. EN (t) = E (t) normalised to 100% recovery



EN (t) of the PITT tracer candidates – EN (t) of HTO vs eluted pore volume in the water saturated sandstone core.



EN (t) of the PITT tracer candidates – EN (t) of HTO vs eluted pore volume in the water saturated chalk core.

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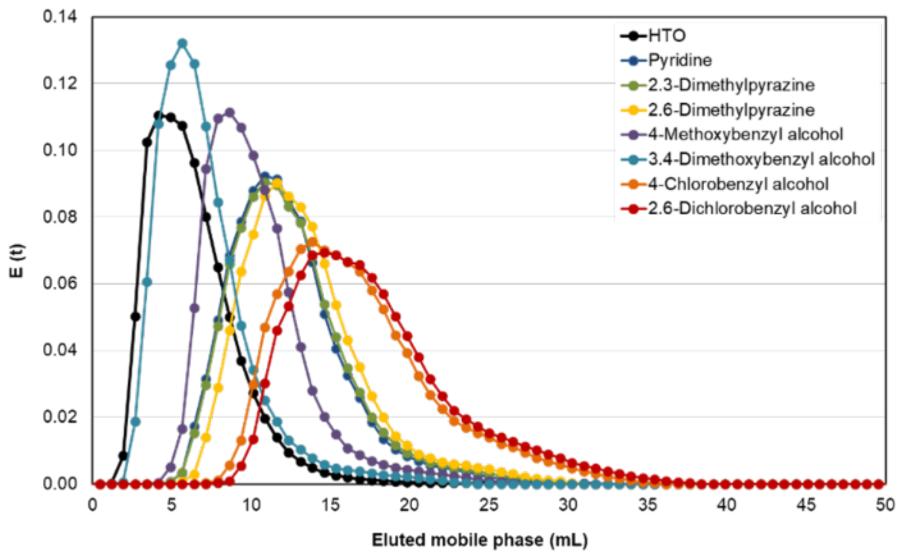
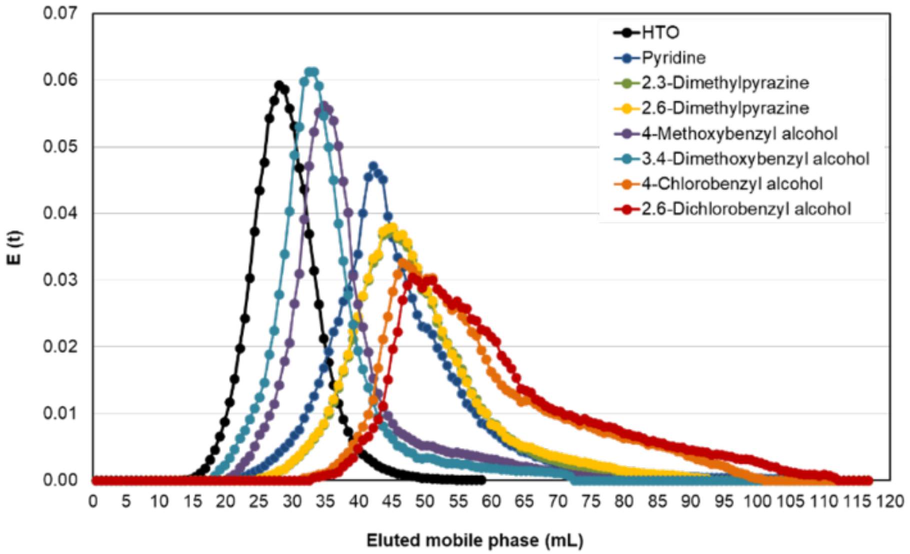
Core flooding experiments

- Cores in S_{OR} conditions



E (t) vs eluted mobile phase in the flooding experiments with the sandstone core in S_{OR} conditions



E (t) vs eluted mobile phase in the flooding experiments with the chalk core in S_{OR} conditions

- The partitioning tracer candidates are delayed relatively to HTO

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Core flooding experiments

- Cores in S_{OR} conditions

S_{OR} values determined by each of tracer candidates at 50% and 70% recovery in the sandstone core in S_{OR} conditions.

		T_W = 54,65 min.		T_W = 74,90 min.	
Tracer	K vs. (Heidrun oil)	T_R (50 % recovery) (min)	S_{OR} (%)	T_R (70 % recovery) (min)	S_{OR} (%)
PYR	2,01	127,1	39,7 ± 0,4	175,2	40,0 ± 0,4
23MPRZ	2,11	129,8	39,5 ± 0,4	179,6	39,8 ± 0,4
26MPRZ	2,39	141,3	39,9 ± 0,4	192,7	39,7 ± 0,4
4METBZOH	1,14	96,26	40,0 ± 0,4	132,1	40,1 ± 0,4
34METBZOH	0,92	88,50	40,2 ± 0,4	120,6	39,9 ± 0,4
4ClBZOH	3,12	167,1	39,7 ± 0,4	227,9	39,6 ± 0,4
26ClBZOH	3,31	171,7	39,3 ± 0,4	235,8	39,4 ± 0,4
S_{OR} from the balance to the injected oil = 39,5%					
S_{OR} from the reduction in water pore volume measured by HTO = 39,8%					

S_{OR} values determined by each of tracer candidates at 50% and 70% recovery in the chlak core in S_{OR} conditions.

		T_W = 290,2 min.		T_W = 321,1 min.	
Tracer	K vs. (Heidrun oil)	T_R (50 % recovery) (min)	S_{OR} (%)	T_R (70 % recovery) (min)	S_{OR} (%)
PYR	2,01	462,6	22,8 ± 0,3	510,6	22,7 ± 0,3
23MPRZ	2,11	470,5	22,7 ± 0,3	524,7	23,1 ± 0,3
26MPRZ	2,39	494,8	22,8 ± 0,3	540,2	22,2 ± 0,3
4METBZOH	1,14	389,6	23,1 ± 0,3	431,4	23,2 ± 0,3
34METBZOH	0,92	371,1	23,3 ± 0,3	411,7	23,5 ± 0,3
4ClBZOH	3,12	555,7	22,7 ± 0,3	613,8	22,6 ± 0,3
26ClBZOH	3,31	570,6	22,6 ± 0,3	629,5	22,5 ± 0,3
S_{OR} from the balance to the injected oil = 22,7%					
S_{OR} from the reduction in water pore volume measured by HTO = 22,9%					

- Good agreement is encountered between the S_{OR} values obtained by the tracer candidates at 50% and 70% recovery, and the mass balance to the injected oil and variation of water pore volume measured by HTO.

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Concluding Remarks About the Tracer Qualification Process

- The methodology presented allows the development of new oil/water partitioning tracers ready for field tests
  - The stability of the candidates is evaluated under simulated relevant reservoir conditions
  - Interactions with reservoir rock materials are identified/characterised
  - Variations of the partition coefficients are described
  - An analytical method to identify and quantify the tracers in real production waters in the ng/L concentration is developed
  - Flooding properties under water saturation and S_{OR} conditions are evaluated on consolidated sandstone and carbonate cores
- 2,3-Dimethylpyrazine; 2,6-dimethylpyrazine; pyridine; 4-chlorobenzyl alcohol; 2,6-dichlorobenzyl alcohol; 4-methoxybenzyl alcohol; and 3,4-dimethoxybenzyl alcohol are ready for field testing as PITT tracers and commercially available (15 – 140 USD/kg)

Intra-Centre Collaboration: Environmental risk Assessment (ERA) of the Partitioning Tracers

- There are legal requirements to evaluate the risk that the PITT tracers developed pose to the maritime ecosystems in case of accidental release on the sea.

John-Sigvard Gamlem Njau

Master's student with the NIORCN

Mehul Arun Vora

PhD candidate with the NIORCN

Steinar Sanni

Chief Scientist at NORCE and Associate Professor at UiS

Overview of the ERA of the Partitioning Tracers

Steps	Measured parameter	Standard followed / model used	Principle
1	Bio-degradability	OECD 306 - Closed bottle test	Biological oxygen demand of tracer measured over 28 days
2	Toxicity	ISO 10253 - Exposure to algae (Skeletonema costatum)	Growth inhibition of algae cells measured over 72 hours
		ISO 21115 - Exposure to fish gill cell line (Rainbow trout)	Mortality of fish gill cells measured over 48 hours
3	Environmental impact factor (EIF)	Dynamic risk and effects assessment model (DREAM)	Risk contribution from tracer calculated in terms of EIF

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Further Reading

- Mario Silva (2021). “Development of new oil/water partitioning tracers for the determination of residual oil saturation in the inter-well region of water-flooded reservoirs”.
    - ISBN: 978-82-7644-994-5
    - ISSN: 1890-1387
    - PhD thesis UiS No. 578

- URL: https://uis.brage.unit.no/uis-xmlui/handle/11250/2735350

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Notes from Tracer Webinar in the Field Application Delivery Forum 04.05.2021

Participants:

Andreas Grøteide Polden (Lundin)
Gael Chupin (Lundin)
Egil Boye Petersen (AkerBP)
Ricardo Andreas Burgos Spangen (AkerBP)
Thierry Laupretre (AkerBP)
Johanna Normann Ravnås (WintershallDEA)
Bjørn-Ove Heimsun (Vår Energi)
Kjetil Skrettingland (Equinor)
Robert Moe (ConocoPhillips)
Jone Urdal (ConocoPhillips)
Jarle Haukås (Schlumberger)
Steinar Sanni (UiS/NORCE)
Mehul Arun Vora (UiS)
Aksel Hiorth (UiS/NORCE)
Tina Puntervold (UiS)
William Chalub Cruz (NORCE)
Børre Antonsen (IFE)
Jan Nossen (IFE)
Tor Bjørnstad (IFE)
Sissel Viig (IFE)
Mario Silva (IFE)
Arun Kumar Paneer Selvam (IFE)
Mahmoud Ould Metidji (IFE/IOR Centre 2016-2020)

Comments (C), questions (Q) and answers (A) for "Chemical tracers for near-well SOR monitoring" presented by Mahmoud Ould Metidji:

Q1: What is the accuracy in results from a single-well chemical tracer test compared to results from other types of tests (e.g., core examinations)?

C1: One of the participants referred to a test performed at Snorre where results corresponded well with SCAL measurements.

Comments (C), questions (Q) and answers (A) for "Nanoparticle tracers for petroleum reservoir studies" presented by Arun Kumar Panneer Selvam:

C1: It will be important to estimate the number of particles needed for a field operation and also the amount of tracers needed for such an operation. Arun answered that such estimations will be included in a feasibility study.

C2: It was also commented that the particle size and stability at reservoir conditions is important parameters.

Q1: Will the passive and partitioning tracers be loaded into separate capsules and how will the two slugs (capsules and trigger) be injected?

A1: The two types of tracers will be loaded into a different set of equal capsules. The capsules and the trigger will be injected as two separate slugs, but the capsules will have a surface that makes the particles to be slowed down in the reservoir compared to the trigger slug which will move with the speed of injected water. Therefore, the trigger will catch up on the capsules in the reservoir at a depth that can be estimated based on a model of the near-well volume.

Q2: Will the pH effect observed for the capsules be the same as what is experienced for smart water?

A2: pH effect has a different nature in this case.

Q3: How important is it to know the exact concentration of the tracers loaded into the capsules to get accurate results from the tracer test?

A3: This should not be important as long as the concentration is high enough to detect the tracers when they are produced. It is the difference in production time that matters, i.e., the difference in the first moments of the tracer production profiles.

Q4: What is the advantage of using capsules compared to a traditional SWCTT?

A4: Use of capsules will shorten the SWCTT test time because no well shut-in time is required. In addition, since there are no chemical reactions involved on the capsules during injection and no reactions of the tracers during back-production, the uncertainty that prevails with the traditional SWCTT on hydrolysis (during injection and back-production) is non-existent with the capsule method. This will produce "cleaner" tracer dispersion profiles. Also, it can be designed how far into the reservoir the capsule should go based on knowledge of the chromatography of the particle in the specific formation rock and knowledge on the permeability (fissures, fractures, pore size distribution) in the near-well volume.

C3: An audience participant commented that it would be nice to see some interpretation of field test with C-dots.

<u>Comments (C), questions (Q) and answers (A) for "Interwell partitioning tracers for water flooded reservoirs: Development and potential" presented by Mário Silva:</u>

Q1: How will the K-value (partition coefficient) vary as a function of temperature?

A1: Results from lab experiments show that this variation is small. Water salinity will have a much higher influence on the K-value.