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Time-Resolved Fluorescence for Real-Time Monitoring of Both Scale and Corrosion Inhibitors: A Game-Changing Technique

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Abstract

Inhibitor products have been widely used to reduce both steel corrosion and scale deposition on pipelines, tubings and safety valves in critical offshore sites. Their simultaneous injection with brine has gradually increased the lifetime of production wells with incontestable benefits for the oil and gas industry. However, the large use of such additives has not been followed by the development of any efficient technique of dosage that considers the strict requirements of an offshore platform.

In this paper, we describe a novel method of chemical dosage that takes into account all the on-site technical and practical parameters, such as the quantification thresholds or molecule specificity, the real-time monitoring, the resistance and compactness of measurement device and a user-friendly interface of measurement. Current techniques of inhibitor monitoring that use elemental analysis (ICP, MS, . . .) for sub-ppm quantifications appear too complex for a successful long-term on-site industrial exploitation due to the bulky dimensions of apparatus and the delay and complexity of analysis.

A long-term collaboration between University of Lyon and Total EP has defined a simple and accurate method for inhibitor monitoring based on the use of lanthanide tracers and the Time-Resolved Fluorescence (TRF) technique. We exploited the fact that inhibitor chemicals (i) are good chelators for lanthanide ions and (ii) amplify the luminescence signal of rare-earths. In this way we are able to collect the photon emission signal by TRF apparatus and dosing the inhibitors. The portability of the TRF device as well as the sensitivity of detection have been engineered in order to obtain a sub-ppm quantification threshold via 1-click task. Several carboxylates, phosphonates and sulphonates additives diluted in brine solutions has been easily measured out even in presence of residual oil. More than 20 commercial inhibitor solution samples were quickly prepared and dosed with an impressive linearity of response (R^2 test > 0.996) in the 10 – 100 ppm range. Any further extension of range has been possible. In addition, we were able to quantify two inhibitors (scale – scale, scale – corrosion) in the same brine water. This technique certainly opens a new conception of smart chemicals management in oil and gas production for (i) a dramatic minimization of the injected compounds (ii) a reducing delay in pipelines monitoring, and (iii) a shut down of expenses of pipeline maintenance.

Introduction

The industrial innovations in oil and gas production has been the driving force behind (i) the minimization of ecological impacts of exploitation and (ii) the increase of profit margin. Even in a scenario where the stock market price of crude oil is low, the increasing of production efficiency will be crucial for the future of industry business. The quest for innovation becomes more than ever essential in remote offshore platforms where expenses are still dramatically higher than for on-shore extraction.

We would like to take up the challenge, focusing on the critical problem of scale and corrosion management. In this paper, we suggest a novel powerful tool for the smart monitoring of inhibitors additives injected during oilfield exploitation. Our method - based on Time-Resolved Fluorescence (TRF) technology - allows a simple, accurate and efficient quantification of chemicals below ppm ranges, even for double (scale – scale, scale – corrosion) quantification. A series of laboratory experiments proves the feasibility of TRF in further real-time offshore applications.

Why TRF should be considered as a game-changing technique?

The prevention of scale formation and corrosion damages in petroleum pipelines and facilities remains an important challenge (Figure 1). Several interconnected factors, such as (i) the seawater composition used for secondary and enhanced-recovery waterflooding operations; (ii) the formation water of oilfield; (iii) the gradients of temperature and pressure within pipelines are the origin of pipeline damages and the drop of extraction efficiency. For example, the incompatible mixing of injected brine water (high concentration of SO_4^{2-} anions) and formation water (containing Ca^{2+} , Sr^{2+} , and Ba^{2+} divalent ions) dramatically induces the precipitation of insoluble calcium, barium or strontium sulphates or even other incompatible fluids include sulphide scale (ZnS). The oil production may fall to zero in few hours due to the pores restriction, with further dangers for installations including blockage of pipeline and equipment, energy leak, accelerate corrosion, and severe accidents. Moreover, the presence of corrosive agents in produced water such as carbon dioxide, hydrogen sulphides, organic acids and salts such as chlorides and sulphates decrease the average-life of facilities.



Figure 1—Scale formation in pipeline. The combined effect of high pressure and temperature with the high concentration of scale precursors dramatically decrease the efficiency of oil and gas production.

A lot of strategies have been deployed by companies in order to reduce the scale and corrosion damages; the periodical addition (squeeze treatment) of inhibitor chemicals seems to be the best way to control scale and corrosion formations. Inorganic (such as sodium hexametaphosphate, sodium poly(phosphate), sodium pyrophosphate), organic (such as phosphate esters, phosphonates, poly(acrylic acid) and poly(maleic acid)) inhibitors are frequently injected within pipelines. The high amount of inhibitors daily used - not only in oil exploitation but also in many other industrial treatment processes - requires a continuous and efficient monitoring (Graham, G. et al. 1995, Boak, L.S. and Sorbie, K., 2010).

The current techniques used for chemicals management (colorimetric analysis, inductively coupled plasma (ICP) spectroscopy, UV spectrophotometry, hyamine method, ion chromatography HPLC) often display some limitations *e.g.* setup and running costs, size, real-time monitoring. Some of them also require laborious pre-treatment/purification stages for the separation of inhibitor chemicals from the interfering brine salts (Johnstone, et al., 2014). In the case of Hyamine 1622 assays, the quantification is time consuming, labour intensive and difficult to apply in real-time mode (Chilcott et al. 2000, Graham et al. 2010) HPLC and ICP detection techniques – that achieved accurate measurements within a 5-10% error bar – require P-tagged co-polymer type scale inhibitor (SI) that it is not classified as environmental-friendly additive.

Here, the TRF method – as detailed in the following paragraphs – may represent a breakdown from the past technologies not only because it display sub-ppb accuracy as well multi-detection (scale or corrosion chemicals) but also because it can be set up in offshore sites (Agenet et al. 2012, Brichart et al. 2014, Martini et al., 2015).

Principles and engineering

With TRF technique, we would like to shift from the hard-working management procedures (preparation and shipment of samples, laboratory facilities, exploitation of results) to a user-friendly portable monitoring device. The principle of TRF is simple: the production sample is (i) firstly mixed with a revealing solution and (ii) then analyzed with a portable apparatus for direct result. The product engineering focused on 1-click interface where all scientific aspects have been *a priori* optimized.

TRF apparatus With the abbreviation of Time-Resolved Fluorescence we indicate an evolution of classic luminescence techniques, widely applied to cells and biomolecules detection. Instead of illuminating the sample with a continuous beam of light and recording the intensity simultaneously, in time-resolved measurements the marked sample is exposed to a series of microsecond light pulses (typically shorter than the decay lifetime of the marker) and the emission signal of marker is then collected after a fixed delay, *e.g.* in the millisecond range (Figure 2). This cycle is repeated until the signal to noise (S/N) ratio is adequate for the application.

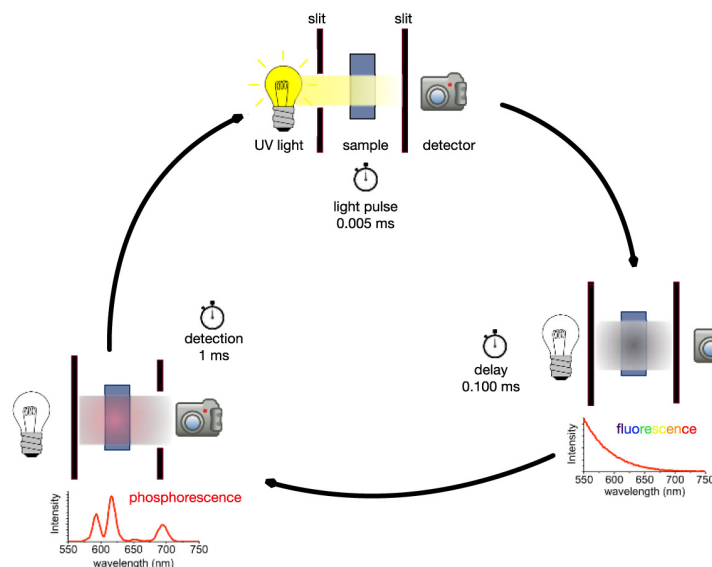


Figure 2—TRF principle: (i) an UV flash lamp irradiates the cell containing the sample, (ii) a triggered device delays the acquisition of signal (fluorescence signal not recorded), (iii) acquisition of emission light during a gate time. The cycle is repeated for increasing S/N ratio.

As shown in Figure 3, a typical TRF device is composed of:

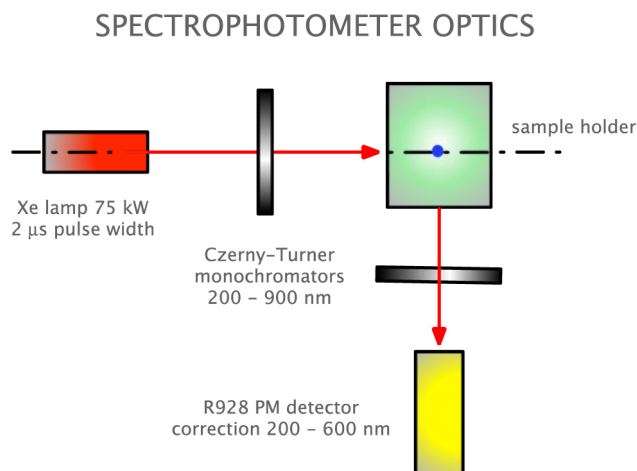


Figure 3—TRF optical configuration. The principle of measurement for both commercial Cary Eclipse (Agilent Technologies) and INIBOX is similar but the electronics and interface is dedicated.

- an UV flash lamp (5 s light pulse) that irradiates the cell containing the sample;
- a triggered electronics that delays the acquisition of signal;
- a Czerny-Turner monochromator that selects the excitation and emission wavelengths
- a 90° PM detector that collects the specific emission light of marker during a gate time.

Revealing solution The test sample must be tagged by specific luminescent marker before the TRF measurement; the revealing solution contains lanthanide ions such as europium (Eu^{3+}) and/or terbium (Tb^{3+}). Some of Rare-Earth (RE) elements display interesting luminescence properties due to their unique electronic configuration; in the trivalent state (Ln III), the shielding of the 4f orbitals by the filled $5p^6 6s^2$ sub-shells induces parity-forbidden 4f–4f electronic transitions. Each transition – that involved characteristic long-live narrow-line emission bands in the visible region – is then quantified by TRF apparatus (Figure 4). Characteristic optical signatures (emission and excitation spectra, lifetime values) are obtained as a function of (i) the nature of marker ions and (ii) the interaction between the marker and chemicals.

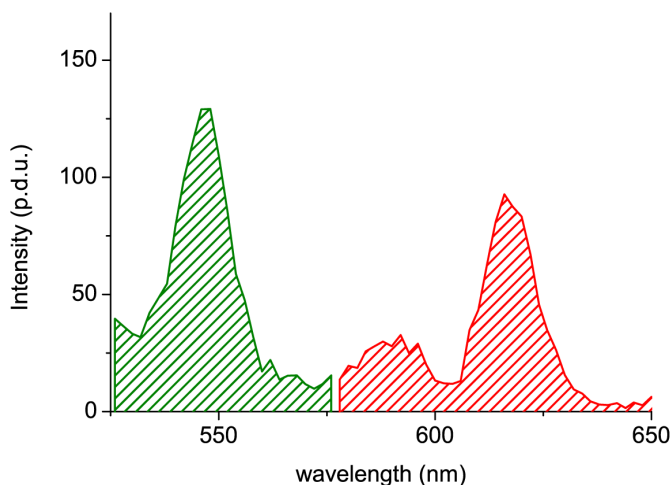


Figure 4— Tb^{3+} (green) and Eu^{3+} (red) ions emission spectra under UV excitation detected by TRF apparatus. Characteristic emission energy gap peaks of lanthanides are not influenced by brine composition nor temperature/pressure gradients. Vice versa, the intensity of peak is correlated to the surrounding conditions of RE ions.

Lanthanide – inhibitors interaction The TRF signal of revealing solution is very sensitive to the surroundings of lanthanide ions (Liu, 2005, Lakowicz, 2008). Pure aqueous solutions of rare-earths display poor TRF luminescence signal due to the non-radiative desexcitation pathways of 4f-electrons via O-H phonons. Elegant theories established that the quenching of Ln^{III} luminescence is proportional to the number of O-H oscillators in the first coordination sphere (Brichart et al., 2014).

The addition of inhibitors chemicals (anti-scale, anti-corrosion, polymers) to the revealing solution forms chelating species that wraps the Ln^{III} ions (Figure 5) and sweeps away the water molecules. Depending on chemical used, the number of coordinated water molecules decreases from nine (100% water – high luminescence quenching) to almost zero (best TRF signal), e.g. in dipicolinic acid complexes. Commercial inhibitors include a wide variety of ligand moieties - such as simple chloride and sulfate anions, carboxylate groups and nitrogen donor atoms, phosphonates or the new carboxymethyl inulin biopolymers – that interact specifically with lanthanide ions once mixing with the revealing solution (Collins, I.R. et al. 2001).

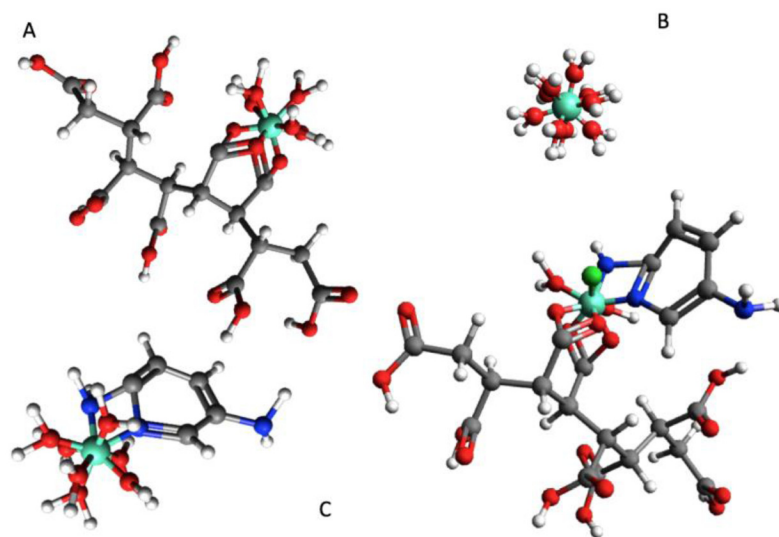


Figure 5—3D representation of chemical structure of Terbium complexes (Tb: green, O: red, N: blue). A: Terbium ions chelated by maleic acid derived macromolecules; B: Terbium ions in aqueous solution coordinate 9 H_2O molecules; C: Terbium ions chelated by anti-corrosion chemicals

Materials and Methods

Chemicals

Synthetic Perpetua (Angola), and Elgin (Scotland) seawater were prepared following the composition described in Table 1. Angola brine composition remains below 50 000 mg/L whereas in Elgin site the concentration of salts is higher than 300 000 mg/L. Commercial scale inhibitors SC1 and SC2 (based on phosphonate) and corrosion inhibitor CR1 (based on imidazole) were chosen because of their co-exploitation in both sites. The real concentration of commercial solutions in terms of active chemical compound is unknown (probably between 10 % and 50 %) and then not considered in our dosage. A flask containing europium salt diluted in 20 g/L NaCl 6.5 pH buffered aqueous solution (NanoH S.A.S., Lyon, France) was used as revealing solution RS1. A second flask containing terbium in 20 g/L NaCl 6.5 pH buffered aqueous solution (NanoH S.A.S., Lyon, France) was prepared as revealing solution RS2.

Table 1—Brine water composition (Perpetua and Elgin).

Ion	Concentration (mg/L) Perpetua	Concentration (mg/L) Elgin
Na^+	15190	90480
Ca^{2+}	417	18597
Mg^{2+}	184	1088
K^+	161	10594
Ba^{2+}	14,7	2266
Str^{2+}	14,5	614
Cl^-	23648	186863
HCO_3^-	737	0

Samples preparation

A calibration curve is performed for each production site and chemical used. A set of samples containing an increasing amount of inhibitors (*e.g.* 0 - 100 ppm) were (i) prepared in reconstituted brine water, (ii) diluted (*e.g.* 10%) in the revealing solution, and (iii) - after 30 min of mixing at room temperature - measured by TRF.

The same protocol and parameters were then applied to the unknown sample and the TRF signal compared with above-mentioned calibration curve. LabView interface managed the whole technical and analytical procedure and gave the concentration of unknown sample by mathematical extrapolation of linear fit.

TRF instrument setup

Standard samples used for calibration have been prepared in PMMA cells 10 x 10 mm optical path. No heating steps were required and cells have been stocked at room temperature. The TRF luminescence signal was collected by using Cary Eclipse spectrophotometer (Agilent Technologies). In time-resolved configuration (75 kW Xenon flash lamp, Czerny-Turner monochromators, $\Delta pulse = 2 \mu s$, 800 V PM detector), the excitation wavelength was adjusted to 350 nm for RS1 revealing solution and 310 nm for RS2. The gate window of detector as well as the slits depend on the experiment and they will be detailed later. All data have been treated by OriginLab software.

A portable field TRF apparatus (INIBOX) developed by Axint SAS (Lyon, France) is tested. It includes a tuned pulsed Xenon lamp, an optical fiber that pipes the source light till the sample holder (10 x 10 mm PMMA cell), dycroic filters for the light selection, and a hi-sensitivity PM counting detector. All the electronic and triggering between light source and detection are customized for oil field applications. A simple interface was compiled by LabView software and is compatible for Windows 10 operating systems.

Data analysis

Single-inhibitor brine samples dosage is achieved by extrapolation from linear calibration curve:

$$I_{TRF} = I_0 + K[inhibitor]$$

I_0 represents the signal of revealing solution in absence of chemicals, K is the slope obtained from calibration, and $[inhibitor]$ is the unknown chemical concentration.

Double-inhibitors mixture (scale-scale, scale - corrosion) quantification requires two calibration curves under conditions *a* or *b*, chosen from the following list: revealing solution composition, delay and/or gate time, excitation and/or emission wavelength. The resolution of equation system gives directly the concentration of both species:

$$\begin{cases} I_a = A_a + K_{1a} [1] + K_{2a} [2] \\ I_b = A_b + K_{1b} [1] + K_{2b} [2] \end{cases}$$

I represents the TRF intensity of unknown sample, K and A values are the slope and the intercept obtained from calibration curves respectively. Then:

$$\begin{aligned} [1] &= \frac{(K_{2b}I_a - K_{2a}I_b) - (A_aK_{2b} - A_bK_{2a})}{(K_{1a}K_{2b} - K_{2a}K_{1b})} \\ [2] &= \frac{(K_{1b}I_a - K_{1a}I_b) - (A_aK_{1b} - A_bK_{1a})}{(K_{2a}K_{1b} - K_{2b}K_{1a})} \end{aligned}$$

Discussion of results

Advantages of using lanthanide revealing solutions

The use of Europium or Terbium ions as revealing solution for anti-scale and/or anti-corrosion chemicals gives undoubt advantages, firstly in term of signal-to-noise (S/N) ratio, and second in term of specificity. The mixture of revealing solution and inhibitors samples forms stable chelating species at room temperature, easy to detect by TRF apparatus.

Under UV light irradiation, lanthanide ions display monochromatic phosphorescence in the visible range, green colour for Terbium and red colour for Europium (Figure 4). The emission bands are narrow and remarkably insensitive to environmental changes (temperature, solvent); the wavelengths barely shift more than 5 nm. Moreover, the presence of large Stokes shift (the energy gap between absorption and emission transitions band) - often higher than 200 nm - prevents any problem of self-absorption characteristic in organic dyes. As consequence, (i) the two emission colours open perspectives for any further chemicals coding, and (ii) the excitation wavelength within 300 – 400 nm range allows the use of low-cost disposable plastic cells.

Another interesting peculiarity of lanthanides is related to their long-lived millisecond range luminescence. Such timescale emission is excellent for the monitoring of complex phenomena *e.g.* the presence of oil waste, contaminated production waters. The collection of TRF long-lived luminescence signal (*e.g.* 0.7 ms delay time) allows the separation of the marker signal (long-life emission) from the crude oil background noise (nanoseconds or microseconds range due to organic compounds). In general, the S/N ratio as well as the limit of quantification (LOQ value) increase with the delay time. For example, with TRF we are able to separate luminescence signal by portions of gate time: *e.g.* (0 ms delay, 0.2 ms gate), (0.2 ms delay, 0.2 ms gate), (2 ms delay, 0.2 ms gate) values give information about the organic phase (the shortest timebase), about an intermediate inhibitor chemical (0.2 ms delay, 0.2 ms gate), and about another chemical (2 ms delay, 0.2 ms gate) (Figure 6). We would like to highline that the revealing solution is sensitive to the affinity between lanthanide ions and inhibitors, *i.e.* an inhibitor with excellent chelating properties increases both TRF signal and lifetime of marker.

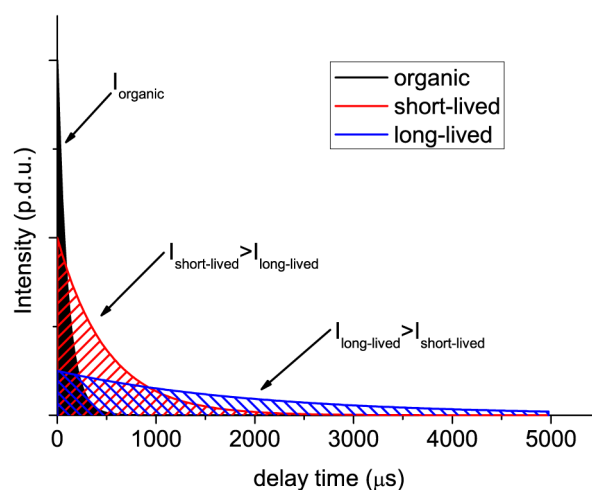


Figure 6—Temporal selection of TRF signal. The selection of delay time allows (i) the quantification of different inhibitors signal (e.g. short lived inhibitor at 0.2 ms, long-lived chemicals at 2 ms) and (ii) the organic phase removing.

Single inhibitor quantification

We investigated the TRF quantification of two separate inhibitors (scale SC1 and corrosion CR1) diluted in production brines (Perpetua and Elgin waters). Following the protocol described above, a set of brine samples ranging from 0 to 100 ppm of the specific inhibitor (a typical range of concentrations used in exploitation) were mixed with revealing solution RS1 (10% dilution) and analyzed by laboratory TRF apparatus and the portable field one. The detection parameters such as PM voltage, slits and integration time were fixed for all measurements. The signal has been collected in a 10 nm - window centered at 615 nm, under 350 nm monochromatic irradiation. A dozens of independent repeats have been performed during a 7-day time period (Figure 7).

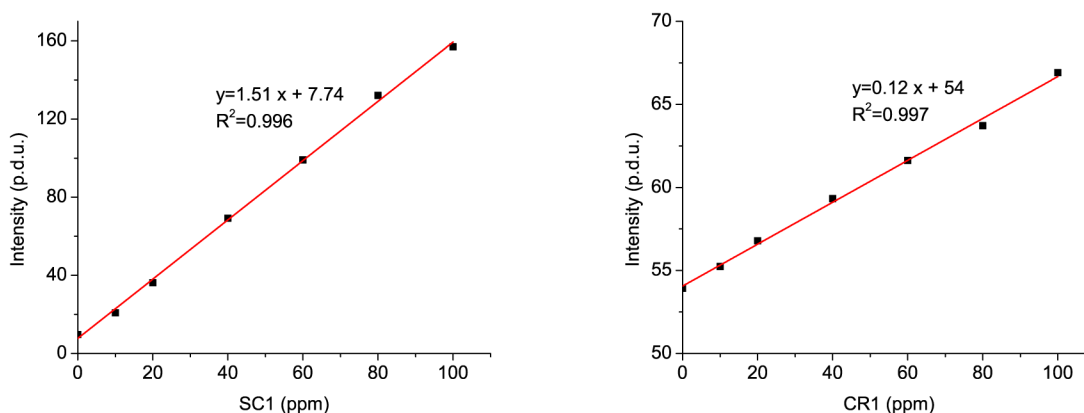


Figure 7—Calibration curve of scale inhibitor in Elgin brine (left), corrosion inhibitor in Perpetua brine (right). Revealing solution based on europium ions, 350 nm excitation, 617 nm emission, delay time 0.6, gate time 1 ms. The fitting of experimental points in the 0 ppm – 100 ppm range prove the linearity of TRF signal with the inhibitor concentration.

The slope of linear fitting found in SC1 chemical is equal to 1.51 ppm^{-1} whereas in CR1 samples is around 0.12 ppm^{-1} . As expected, anti-corrosion chemicals display lower affinity with the revealing solution than the anti-scale chemicals. Additional samples measured in Elgin reconstituted water with -20% salts content displayed superimposes calibration curves, *i.e.* the influence of brine composition is almost negligible (Figure 8). Indeed, the simple 10% dilution of samples in revealing solution reduces all interferences due to the brine composition.

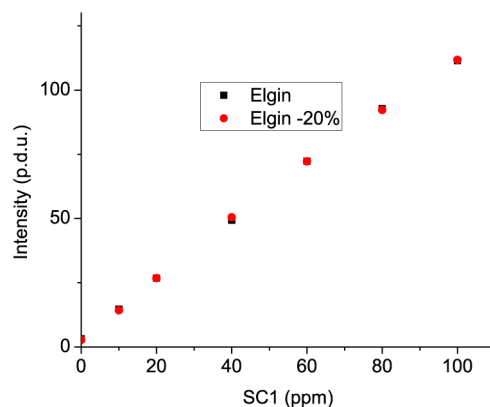


Figure 8—Influence of salt on TRF measurements. The scale inhibitor SC1 displays the same linear TRF trend in both brines (20% composition difference)

Double scale – scale inhibitors quantification

The simultaneous quantification of two scale inhibitors (SC1, SC2) in brine waters (Elgin) needs two calibration curves under conditions *a* and *b*, as described previously. Two revealing solutions based on europium ions were prepared as condition *a* and condition *b*; the first one buffered at pH 4.5 (condition *a*), the other one buffered at pH 6.5 (condition *b*). For the measurements, we fixed the excitation wavelength (350 nm) and the emission wavelength (615 nm), but the delay time depends on the conditions: delay 0.1 ms – gate time 1 ms for the condition *a*, 0.6 ms – 1 ms for condition *b*.

Under condition *a*, the scale-inhibitor SC1 displays low TRF signal due to pH effect; even at high concentrations (closed to 250 ppm), the signal is not far from the LOQ and the slope is equal to 0.0014 ppm⁻¹ (Figure 9). The second inhibitor SC2 has good linear correlation with a slope of 0.22 ppm⁻¹. Under condition *b*, both series display good linearity with the inhibitor concentration; as observed for condition *a*, the SC2 slope value is higher than the one obtained in SC1 series.

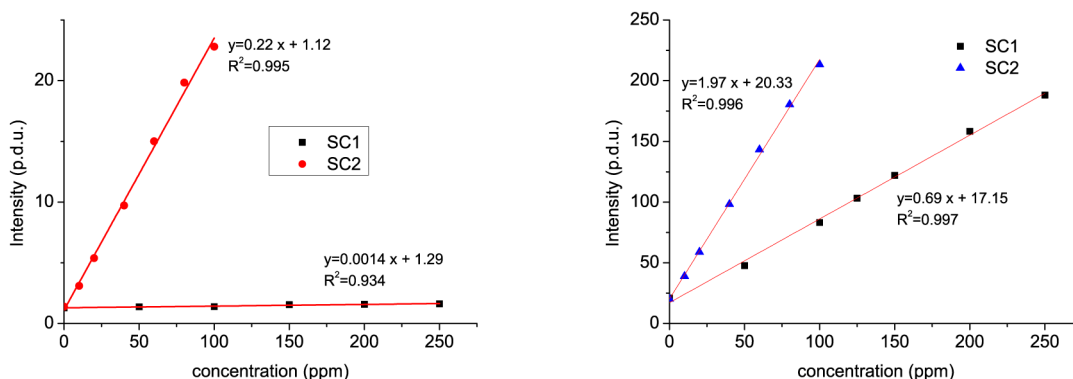


Figure 9—Double quantification of scale inhibitors SC1 and SC2 in Elgin brine under condition *a* (left) and condition *b* (right). The condition *a* (revealing solution at pH = 4.5) is sufficient to distinguish the nature of anti-scale chemicals (SC1 slope = 0.0014 ppm⁻¹, SC2 slope = 0.22 ppm⁻¹).

We then prepared a test mixture composed of 150 ppm of SC1 and 40 ppm of SC2 in Elgin water. Using the equation system described in data analysis paragraph, we obtained 140 ppm of SC1 and 42 ppm of SC2, values in accordance with the theoretical concentrations.

Double scale – corrosion quantification

For production waters containing both scale (SC1) and corrosion (CR1) inhibitors, we prepared two separate revealing solutions as condition *a* (RS1) and *b* (RS2). The first one based on europium ions was

buffered at pH 6.5 whereas the second one was based on terbium ions at same pH. Parameters of analysis were setup as a function of revealing solution: 350 – 617 nm, delay 0.2 ms for RS1 and 310 – 545 nm, 0.6 ms for RS2.

The plot of data reveals some interesting details (Figure 10): under condition *a*, the increments of CR1 chemicals does not affected the TRF intensity (constant and equal to 11.6 p.d.u.). Vice versa, the luminescence signal is proportional to the concentration of scale inhibitors (SC1) and the slope is equal to 0.22 ppm⁻¹. The other condition (*b*) is selective only for the anti-corrosion chemicals and not for the anti-scale additives. Since each condition was selective for only one inhibitor species, we were able to eliminate two parameters ($K_{2a} = 0$, $K_{1b} = 0$) from the equation system. A test sample (60 ppm SC1, 40 ppm CR1) was analyzed under condition *a* and *b*. Using the simplified equation system, we obtained 57.3 ppm for SC1 and 39.4 ppm for CR1, in accordance with the theoretical concentrations.

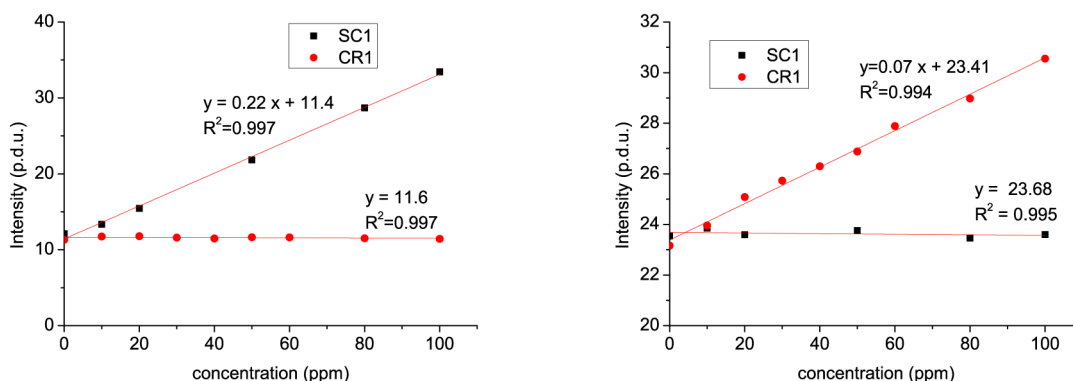


Figure 10—Double quantification scale – corrosion inhibitors. The condition *a* (left) influenced the TRF signal of SC1, and keeps constant the luminescence of revealing solution for CR1 increments; the condition *b* (right) unchanged the signal of CR1.

Conclusions

Time-resolved fluorescence detection has been applied for the quantification of scale and corrosion inhibitors in brine production waters. The procedure does not require any time-consuming pre-treatments, and the test sample – collected from site or directly on-line – is simple mixed with a revealing solution and analyzed by TRF apparatus. Lanthanide ions as Eu^{3+} and Tb^{3+} (the constituents of revealing solution) interact specifically with inhibitors chemicals and give TRF signal proportional to the concentration of additives. The quantification of single corrosion or scale inhibitors in brines needs one set of samples for the calibration whereas the double quantification requires two calibration curves under different conditions (*e.g.* revealing solutions, analysis parameters). The detection is done in less than 2 minutes, and in almost the totality of cases studied we obtained a linear proportion with the concentration in the range 0 - 100 ppm. The use of low-price disposable cells for measurements as well the availability of portable field apparatus (INIBOX) enhances the potentials of technique for any further offshore quantification. Excellent results were obtained with more than 20 commercial scale and corrosion inhibitors in both single and double quantification. A first industrial *on-site* test will be planned (March 2016) in Elgin field (North Sea) and some other collaborations will be launched soon.

Some features of technique are listed below:

- timesaving: 60 minutes for calibration curve preparation and extrapolation, 1 minute for quantification;
- compact: 1 m² for portable field apparatus and disposable materials;
- simple: sample preparation (test brine + revealing solution) and analysis (1-click device);
- resolution: sub-1 ppm limit of quantification (LOQ);

- concentration range: linearity in range 0 - 500 ppm;
- robustness: no interferences with oil, sand or salt contents;
- versatility: tested in a wide range of scale and corrosion inhibitors and ready for hydrate inhibitors and polyacrylamide polymer (Enhanced Oil Recovery).

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