EUROPEAN FEDERATION OF CORROSION
Working Party 13 - Corrosion In Oil & Gas

MOM of the Moscow Business Meeting
Monday 13th September 2010
14:00 – 18:00, Gubkin University

T. Chevrot, Chairman of WP13 opened the meeting at 14:00

1. General Information

T. Chevrot welcomed the 50 members of WP13 attending the meeting and thanked warmly Gubkin University and its students for helping in the organization and the logistics of the meeting.

EFC WP13 Chairmanship

Thierry Chevrot, current Chairman of EFC WP13 will move within Total in the coming year and proposes that Michel Bonis (TOTAL) replaces him when this move will take place. Jean Kittel (IFP Energies Nouvelles) will remain as vice-chairman. The Chairman asked the participants whether anybody had any objection about such changes. The proposal was approved unanimously.

Post meeting note: The handover between T. Chevrot and M. Bonis will take place during Eurocorr 2011 in Stockholm.

It was also proposed that as done in the past, TOTAL continues to chair WP13 for the full 5 year term, before another country takes the lead. It was suggested that Shell could take over the chairmanship of WP13 in 2014, but no candidate for chairmanship has been identified so far.

Eurocorr 2010

Eurocorr 2010 conference programme was quickly presented. The programme includes two joint sessions with NACE:

- Sour service: Organized jointly with NACE TEG 374X “Materials for Oil & Gas” co-chaired by Thierry Chevrot (EFCl) and Bob Badrak (NACE).
- Corrosion inhibition and monitoring: Organized jointly with NACE STG 61 “Corrosion and scale inhibition” co-chaired by T. Chevrot (EFC) and Moshen Achour (NACE).

The traditional prize offered by, Antikor for the best paper by a presenter less than 35 years old will also be awarded during the conference.

NACE 2011 – Houston

Two joint sessions between NACE and EFC will be organized during NACE 2011:

- TEG 374x / WP13 on Materials in Oil & Gas: subjects for informal discussions should be sent to Richard Thompson RMTH@chevron.com or to Thierry Chevrot (Thierry.chevrot@total.com).
- STG61 WP13 on corrosion and scale inhibition: subjects for informal discussions should be sent to Mohsen Achour (Mohsen.H.Achour@conocophillips) or to Thierry Chevrot (Thierry.chevrot@total.com).

Eurocorr 2011 – Stockholm

Eurocorr 2011 will take place in Stockholm 5th to 8th September, 2011.
WP13 plans a joint session with EFC WP9 'Marine corrosion".

2. Progress of active Working Groups

"Recommended Practice for Pipeline Corrosion Management in Oil and Gas production and Transportation", T. Chevrot, TOTAL

Thierry Chevrot presented the progress of the working group, and the presentation can be found in Appendix 1.

The work has progressed very well, thanks to the involvement of several members of WP13 and the support of their respective companies (BG, BP, ConocoPhillips, Statoil, Technip, Shell, Total, Wellstream, WGIM) and the work of Bijan Kermani (KeyTech) to chair the working group.

The group aims to publishing the document towards the end of 2011 (50% of the document completed as of today). The final draft should be ready in June 2011 for peer review and T. Chevrot asked for volunteers for reviewing & commenting the document.

Volunteers who proposed to review the document: Ulf Kivisäkk (Sandvik), Tom Mcwalter (ConocoPhillips), Patrizia Fassina (ENI), Marc Wilms (Shell), Ilson Palmieri (Petrobras), Mehdi Askari (Pars O&G Co.)

T. Chevrot also mentioned that NACE is writing a similar document, but that both organizations decided to prepare two separate documents with the possibility to merge them in the future, may be as an ISO standard.

Progress on the work on 4-Point Bend method for cracking resistance determination in sour service, Chris Fowler, Exova

Round robin testing and discussions on four point bend test procedures (ASTM, NACE, EFC) have been taking place between Exova, National Physical Laboratory, and The Welding Institute). C. Fowler emphasized the problems associated with the current test protocols, in particular with regards to specimen preparation and thickness, and which lead to tremendous scattering in the results. Clad specimen are part of the investigation, but all specimen (including welds) are machined in order to privilege reproducibility (i.e. no as-machined specimen are included in the test programme). Further data will be available for NACE 2011.

The presentation is included in Appendix 2.

Laboratory testing of materials to qualify clear brine fluids for use in wells, J. Martin, BP

John presented on behalf of S. Nodland (Statoil) the status of the working group dedicated to the preparation of an EFC document entitled “Laboratory testing of materials to qualify clear brine fluids for use in wells”.

The purpose of the document is to give guidelines for clear brines (i.e. completion, packer, workover and well suspension fluids) selection, including testing requirements, in order to avoid corrosion risks. Clear brine drilling fluids are not included in this work.

Both prequalification testing and qualification testing are covered. The document gives recommendations with regards to materials type, test environment, corrosion mechanisms, test methods, evaluation of result and reporting.

The draft document which is currently under final review is included in Appendix 3. Once the final draft is available, the document will need to be prepared for the editor’s review for publication. However, it has to be noted that EFC will not publish documents which are less than 20 pages.

Post meeting note: *S. Nodland has now changed position and somebody else from the working group needs to take the lead to “shape” the document in an editable format.*

3. Corrosion inhibition – Chaired by G. Dicken, Intertek

“New Technologies for Corrosion Protection in Oil & Gas Industry”, Pr. E. Lyublinski, NTCI

Pr. Lyublinski presented some applications for vapour phase corrosion inhibitors (VCIs) including:

- Corrosion protection of roofs inside storage tanks, including installation of a dispensing system on some storage tanks in a Petrobras refinery. Subsequent inspections showed a decrease i the corrosion rate by a factor 5 to 10. VCIs formulations may have to be adjusted depending on the gas cap composition in the tank.
- Corrosion protection of mothballed equipment and enclosures (including double bottoms).
- Corrosion protection of well annuli.

Unfortunately, the presentation is too long to be included in these MOMs, but further information can be sought from Pr. Lyublinski.

Corrosion Inhibition Science not Snake Oil, G. Winning, WGIM

George Winning made a review of some of the challenges associated with the formulation and use of corrosion inhibitors:

- Effects of thiosulfates,
- Role of fatty acid and amines (e.g. MEA-DETA-TEPA),
- Filming mechanisms and interactions of corrosion inhibitors with oil,
- They are surfactant inhibitors (adsorption of inhibitors onto metal surface),
- Cathodic polarization under deposits,
- Inhibition of preferential weld corrosion,
- High temperature and high pressure testing,
- Top of the line corrosion (effect of volatile fatty acids),
- Environmental challenges, cost effectiveness, HPHT, presence of oxygen.

The presentation is given in appendix 4.

4. Presentation of the Working Group of the French Anticorrosion Centre (CEFRACOR) “Corrosion in Oil & Gas”, H. Marchebois, Vallourec

Hervé Marchebois gave an overview of the “Corrosion in Oil & Gas” Committee of CEFRACOR and its activities. This committee can be considered as a “mirror group” of EFC WP 13 in France, and is structured in 9 Woking Groups:

- WG1: List of Companies active in O&G products or services
- WG2: List of academic or professional training centres in corrosion
- WG3: Internal corrosion
- WG4: Alternative materials
- WG5: Environmental cracking
- WG6: Corrosion under insulation
- WG7: External corrosion
- WG8: Internal inspection
- WG9: High temperature

The presentation can be found in Appendix 5.

5. AOB & Close of Meeting

No other business was brought by the attendees. Before closing the meeting, Thierry Chevrot thanked again all the students of Gubkin University who helped in running the meeting and the meeting was closed.

Appendix 1

EFC WP 13; Corrosion in Oil & Gas

WG: Pipeline Corrosion Management (PCM)

An Update

Bijan Kermani

TOTAL

Type of document

- Guidelines ?
- **Recommended practice**
- Minimum requirements ?
- Best practice ?
- State of the art ?

**It was agreed to produce a “Recommended Practice” document,
which will include some minimum requirements
to ensure the fundamental basics are followed
when managing corrosion in pipelines.
Wording of specific paragraphs will therefore
have to be carefully chosen.**

Objectives

- To produce a reference document
- To capture and compile industry-wide information in relation to CMP
- To define and outline common practices in CMP available amongst oil and gas operators and engineering design houses to:
  - Ease discussions with projects' partners worldwide
  - Deal with third parties tie-ins
- To agree with engineering design houses on processes and approaches necessary to define and characterise corrosion management requirements at the design stage
- The RP should be treated as an integral part of a pipeline management system (PMS)

Means & organisation of the work

- Working Group within WP13: BG, BP, ConocoPhillips, Statoil, Technip, Shell, TOTAL, Wellstream, WGIM, Managed by KeyTech (Bijan Kermani)

- NACE informed on progress as it is trying to produce a similar document (but tightly related to local legislation),

- Each member of the working group is be responsible for drafting assigned parts of the document,

- Draft review by all working group members,

- Draft will be submitted to EFC WP 13 volunteers for review & comments

Aim for publication: end 2011

TOTAL

To maximise the use of existing standards

- **To take advantage of work already carried out by EFC, ISO, NACE etc. organisations when possible:**
  - Simplification of the work to be achieved,
  - Avoids contradicting existing standards,
  - Will ease the publication of a future ISO standard (merging with NACE document?)
  - But need to avoid using standards tightly linked to local legislation (UK, US,…?)

- **To include the work of experts in specialised technical areas**

- **To provide a bridging document:**
  - One document referring to (some) available standards in the industry,
  - To provide a logical link between these standards,
  - To fill in the gaps for a fully integrated corrosion management of pipelines.

Scope

- **Rigid & flexible pipelines, including CRA clad and lined**
- **E&P effluents only including export lines (dry gas & oil)**
  - Including injection and/or produced water
  - Including CO_2 and steam injection
  - Not service lines
  - Not hydrocarbon refined products
- **Onshore & offshore, including pig launchers and manifolds**
- **Corrosion management from design to decommissioning**
- **Title:**

Recommended Practice for Corrosion Management of Pipelines in Oil & Gas Production and Transportation.

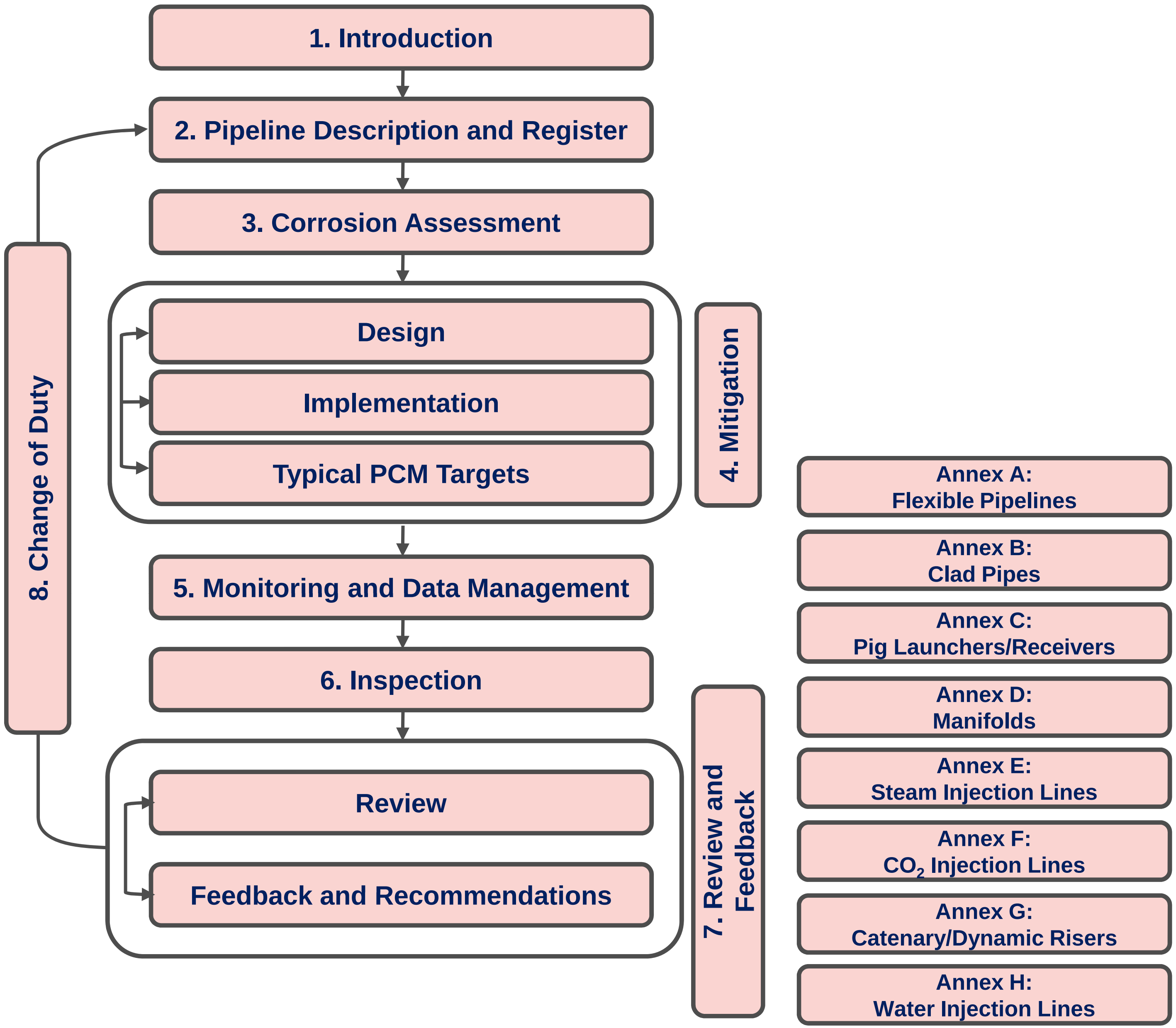
TOTAL

Coverage

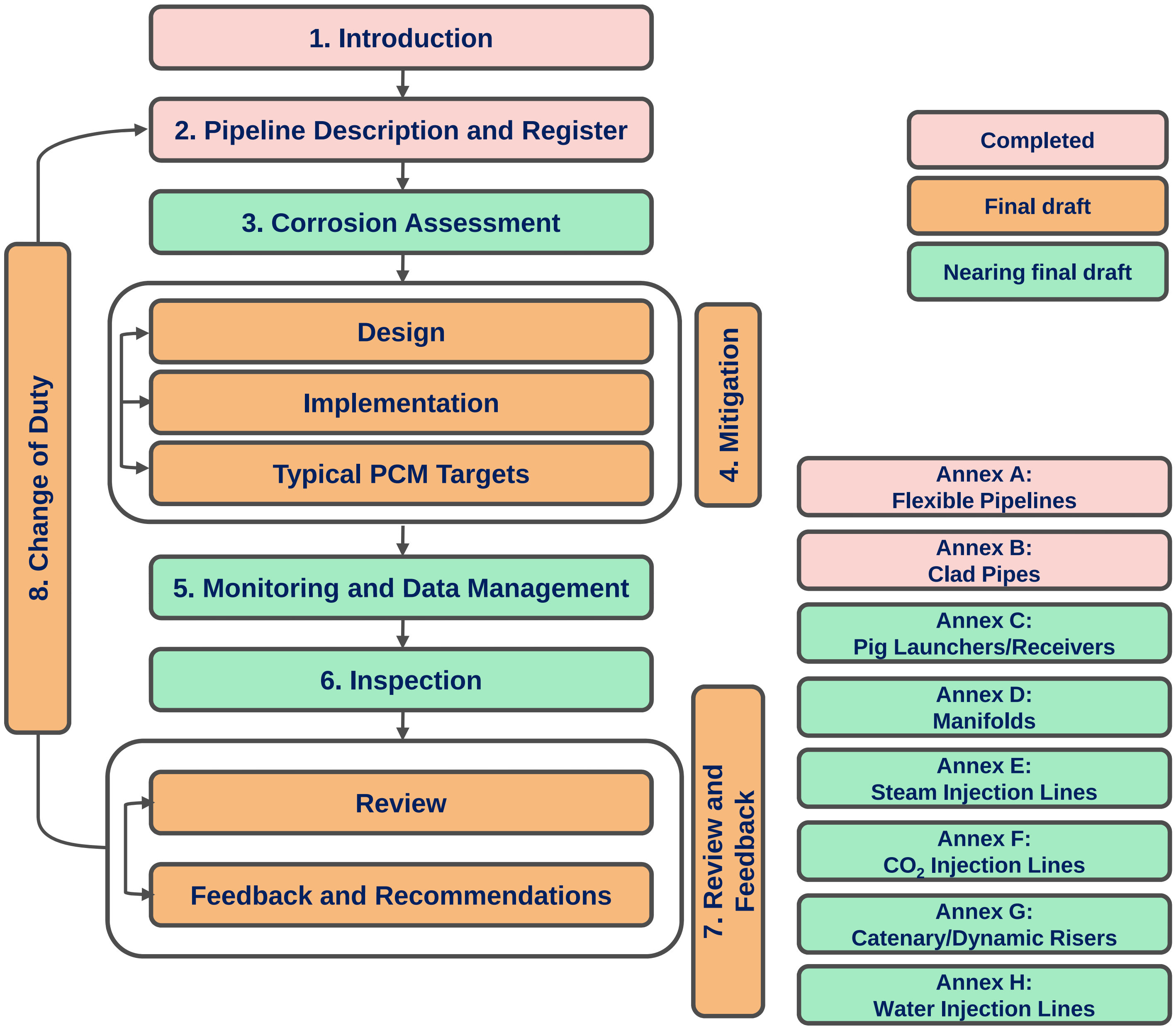
Items Covered by This RP	Excluded Items
Risers, transportation pipelines and trunklines for liquids, gases and multiphase fluids	• Lines handling gas prepared for domestic use (gas distribution systems) • Lines handling processed products • Valves and rotating equipment • Subsea templates • Pressure vessels • Topside/surface facilities • Compression stations • Utility lines/umbilicals/chemical injection lines • Drain lines • Non metallic materials
Flowlines and gathering lines	Crude oil storage, handling facilities, vessels and rotating equipment, heat exchangers, field facilities and processing plants
Manifolds	
Catenary/dynamic risers	J tube, Caissons
Pig launchers and receivers	Valves
Flexible pipelines	Associated pipe-line end manifold (PLEM) and termination manifolds (covered in Manifold Section)
CRA and CRA clad/lined and non metallic lined pipelines	
Injection lines (CO_2, water, steam)	Pumps, chemical injection lines, steam generation, CO_2 removal plant

TOTAL

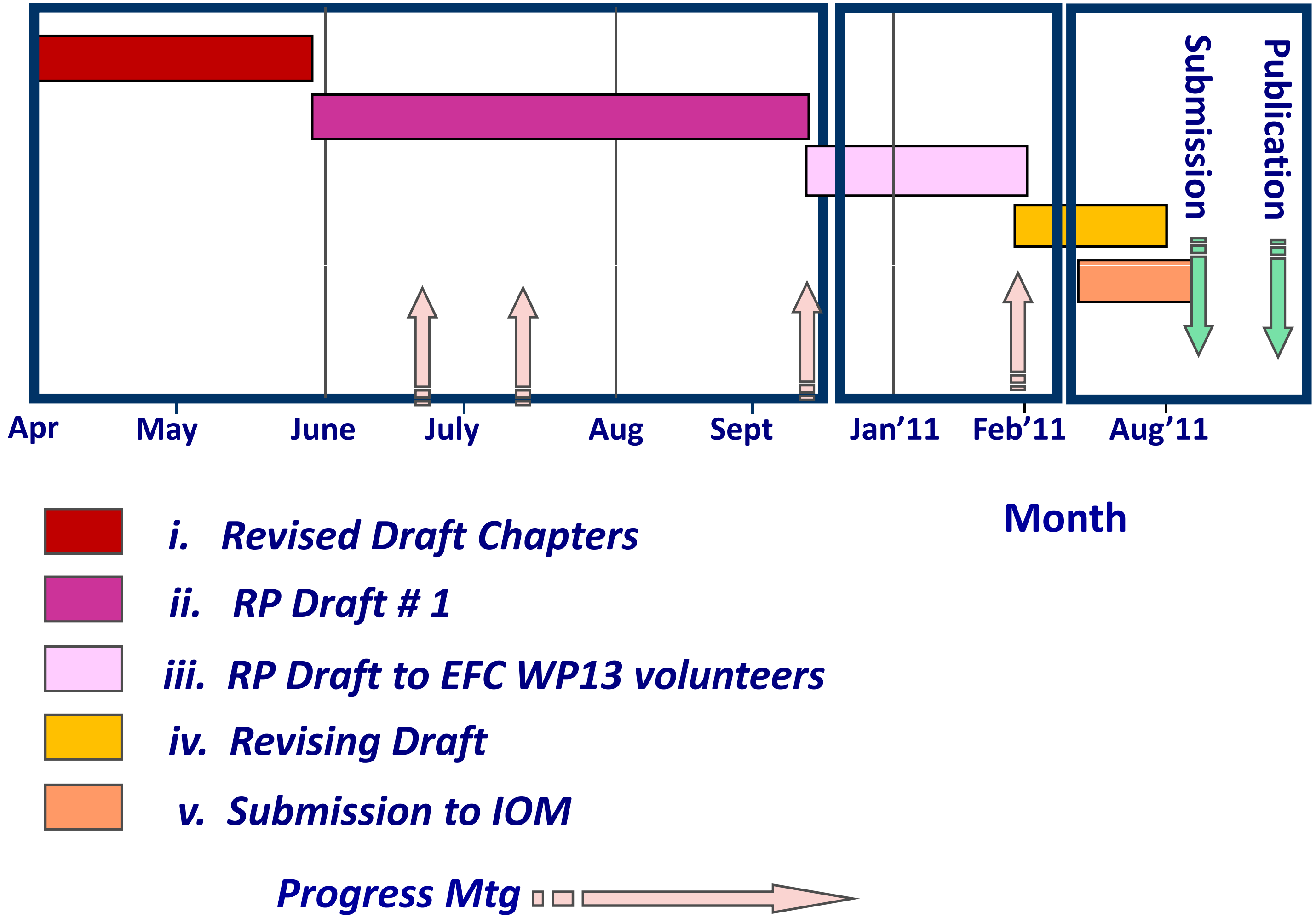
The structure



The status


1. Introduction
2. Pipeline Description and Register
3. Corrosion Assessment
Design
Implementation
Typical PCM Targets
4. Mitigation
5. Monitoring and Data Management
6. Inspection
Review
Feedback and Recommendations
7. Review and Feedback
8. Change of Duty
Completed
Final draft
Nearing final draft
Annex A: Flexible Pipelines
Annex B: Clad Pipes
Annex C: Pig Launchers/Receivers
Annex D: Manifolds
Annex E: Steam Injection Lines
Annex F: CO_2 Injection Lines
Annex G: Catenary/Dynamic Risers
Annex H: Water Injection Lines

Timeline



Next Meeting

- 5-6 October in London area

Appendix 2

EFC WP13 Working Group on the 4-Point Bend method for cracking resistance determination in sour service lab testing. Stuart Bond stuart.bond@twi.co.uk T: +44 1223 899 000 Document: March 2010

Introduction

The 4-point bend (4pb) method has been cited in EFC16 and EFC17 as a test technique suitable for determining the cracking resistance of metallic materials in sour service laboratory conditions. Thus it is also cited in ISO15156/MR0175 as a preferred method. However, although EFC16 and EFC17 provide guidance, when considered in combination with industrial practice it is clear that there are variations in the application of this test method and indeed it is not presently included in NACE TM0177.

Therefore EFC WP13 set up a working group to collate information and experiences of use of the method with the objective of improving guidance in future editions of EFC 16 & 17, and to provide initial input to NACE TM0177 via NACE work group WG085f, in order that standardisation of the method can be achieved for sour service cracking resistance testing.

Presently the following have kindly volunteered to provide input to this Working Group: Stuart Bond, Christoph Bosch, Thierry Cassagne, Brian Chambers, Chris Fowler, Shuji Hashizumi, John Martin, Stein Olsen, Adam Rubin, Alan Turnbull, Masakatsu Ueda and Marc Wilms.

Further contributions and input are actively encouraged.

This topic has been discussed during 2009 at NACE Corrosion2009 (Joint NACE/EFC session) Atlanta, USA and at EuroCorr2009 (WP13) Nice, France plus awareness provided at the corresponding ISO15156 meetings which were both held in conjunction with these conferences. It is intended to discuss this further in March 2010, at NACE Corrosion2010 San Antonio, USA.

Objectives

Through consensus, derive clearer guidance on the 4pb test method for parent materials but most especially weldments in carbon & low alloy steels and corrosion-resistant alloys. This will provide for more uniform application of the method and thus eliminate/mitigate inconsistencies arising when testing weldments.

Overview

Specific items raised by contributions from the working group are tabulated below to allow input, but in summary, the information provided to date can be divided into the following primary subjects:

- Derivation of the mechanical properties of the material to be tested so that it is appropriate for the 4pb method i.e. bend specimens vs. uniaxial tensile specimens which are traditionally used for stress-strain data generation
- Specimen geometry for parent materials and special products (e.g. wires for flexible risers, pipelines etc)
- Use of strain gauging; only for weldments?
- Issues associated with the testing of weldments in carbon and low alloy steels
- Issues associated with the testing of weldments in corrosion-resistant alloys (CRA)
- Issues associated with the testing of weldments in clad materials

The primary users of the method are:

- Materials suppliers or equipment suppliers with items which are not welded, and,
- Test houses/R&D organisations who undertake qualification work for new materials and welded components, and/or derivation of new application limits for materials to support guidance in ISO15156/MR0175 or confidential domain data for operators.

Additional Information Anticipated

BP/NPL - may be able to release internal standard on 4pb testing for EFC and NACE
Exova - undertaking work with another company on issues related to 4pb testing, anticipate being able to release to EFC and NACE.
TWI - internal report on modelling 4pb specimens, may be made available for non-Members.
EFC16 references 4pb in Appendix 2
EFC17 references 4pb as “bent beams” in Appendix 7.4 Loading: Bent Beams

Table Revision 2 - 13 March 2010

Item N°	**Subject / Issue**	**Applicability (C-steel /CRA /All)**	**Summary**	**Supporting comments, data requirements and recommendations**
1	Derivation of mechanical properties of materials	All	Alan Turnbull - we should be loading to 0.2% plastic strain as determined under bend, and using that and the force-strain curve under bend, to determine the corresponding total strain and deflection. It seems to me fundamental since after all we are testing under bend and not under uniaxial strain. In our experience the difference between our approach and the approach of using the total strain from a uniaxial stress-strain test and deflecting to that value can be significant (up to 20% or so) or very small depending on the alloy and the thickness of the specimen. Intuitively, the thicker the specimen the smaller the stress gradient in the bend specimen and the more likely that the two approaches will give closer agreement Other comments: Note retaining UT derived data is helpful when comparing NACE TM0177 Method A results for threshold determination. Uniaxial stressing (NACE TM 0177) vs. 4-point bend stressing; Stress pattern in a specimen exposed to uniaxial loading is different from the stress pattern in a 4-point bend specimen. Can't expect 100% convergence in results between the two loading methods.	Supporting FEA modelling and test data required to address this specific issue. Technical issue is that material mechanical properties are usually derived from uniaxial tensile (UT) specimens. Such testing is governed by numerous international and national standards and as such it should be "repeatable". Experience reported (C-steel) that materials which consistently developed HIC type damage (not SSC failure) in four point bend testing were absolutely free of internal cracks in similar tests where the only difference was a change of loading to uniaxial stressing. **Recommend** placing in EFC16 only as separate item on HIC rather than under 4pb SSC heading. Paper from Richard Pargeter NACE2009 to be discussed JWM - the approach proposed by NPL is the one we have standardised on in BP
2	Parent material testing - routine QA/QC requirements not to be compromised for other test needs.	All	Desire to ensure limited range of sizes are covered by document e.g. 115-140x15x5 mm (L x W x T) to ISO 3183 Products such as wires in flexibles don't conform to dimensions. Need to ensure these can be tested accounting for their geometry. Non-welded samples: calibration curves from typically 5 specimens with strain gauges, deflected in 4pb in 10 steps to yield point monitoring at each step and correlating to ASTM G39. Excellent correlation	ASTM G39 provides good guidance for testing uniform specimens and may not need strain gauging in these cases? Supplier/user needs flexibility in the document to accommodate such issues whilst still complying with the guidance to assure clients. JWM - not sure I see the advantage of limiting sizes in

			For non-welded samples do not mount strain gauge on test specimens, but load based on the formula and the validation load curve. Can also be used for wires with a non-rectangular cross-section (for instance C-shaped profiles).	accordance with ISO 3183. - the equation does not work well with CRAs as indicated in EFC 17 (see comments already in Point 7) **Potential recommendations:** Limit variation so test labs have consistent specimens to test? Avoid specification of standardised specimen size. Rather, advise on ratio between jig size and specimen size. In addition specification for some widely used specimens can be given, for instance pipe wall sections? Use full wall thickness where feasible, or, minimise machining to retain "thick" specimens? JWM - see BP recommendation on **minimum** thickness (if not using full thickness) in Point 6
3	Rounded edges and similar preparations	All	Guidance is needed on the quality of finish for specimens including rounded edges, loading pin location preparation and tolerance on alignment.	Should this be specific or generalised statement? Note TWI recommend for weldments rounding at root toe is essential.
4	Orientation of weldment for testing	All	It seems that all testing on girth weldments uses the specimen extracted with the weld transverse to the specimen long axis. Whilst EFC16 has the diagram indicating extraction along the weldment, this is not generally practicable for pipe girth welds (due to curvature) but guidance could be given. Longitudinal seam welds can be tested as "all-weld" but rarely is this carried out?	**Recommendation:** Document to just note the usual orientation is to extract transverse to the weld but alternative arrangements are acceptable if client or product so dictate? Note: for pipe girth welds axial loading does not represent the hoop stress etc in service.
5	Retaining weldment root intact	All	Provides the geometric condition of the weld root, features such as root intrusions may be present i.e. the most representative specimen. Retains the surface condition of the material as it will be on installation.	Confirm that this is the current industry practice, especially if there are differences in any QA/QC testing vs WPS qualification or cracking domain data generation. This is likely to be an area of contention. See 260x25xwall thickness specimens in

			Retains the chemical composition of diluted root. Note root and hot pass may be different consumable to the fill passes in some joints also. Also, heavy wall pipe needs the root sampled separately from cap if not using full thickness, and of course the cap is not exposed to sour service. Leads to a non-uniform specimen, plus additional costs for jigs, exposure tanks etc. Does require strain gauging for loading. Root profile and degree of excess penetration can be important in performance. Suggestion has been made to photograph prior to testing. Possible preferential weldment corrosion of the HAZ may be observed if the material is susceptible (low alloy steels) BP require that for welds the weld reinforcement on the test-side (tensile surface) shall be left intact with the exception of weld in clad pipe where it shall be removed unless specified otherwise.	CORROSION 2004, paper 04130 C Bosch TWI can accommodate 19x25 with 200 and 40mm spans in constant load testing. Others? Cladding will require masking of the substrate or for metallurgically bonded removal of substrate; is it agreed removal of substrate and testing weld overlay is not practical? JWM - do not agree that substrate removal is impractical for weld overlay, we have done it on a number of occasions. **Potential recommendations:** Test specimens with degreased as-received finish for the given product, including welds for qualification in a simulated operation environment. For ranking of materials and for comparison purposes, standardised surface finish can be applied Characterise degree of misalignment either side of weld and also root protrusion profile (former can be measured but latter will require photo in section)
6	Dimensions for testing weldments - ideally "full thickness"	All	Misalignment which naturally occurs in real joints can have significant impact if the specimen is machined down in thickness. NPL recommended testing full thickness see paper. Carbon steels - large jigs but in essence achievable with sufficient solution reservoirs. Some concerns that there is need to limit specimen size range CRA - require autoclave testing usually and this will constrain maximum jig size which can readily be accommodated.	See Corrosion Engineering, Science and Technology Vol40, 2,pp103-109 A Turnball **Potential recommendation?** Recommend that weldments be tested at full thickness or at least 8mm thick (whichever is smaller) with root intact. Does it matter is specimen is thinned outside the loaded region?

			From BP, minimum sample thicknesses shall be:- o For parent material 4mm, or full thickness if less o For welds 8mm, or full thickness if less o For clad layers 2mm	
7	Strain gauging of all specimens	All	Certainly this is required for weldments, need guidance on location and for CRA the use of a calibration specimen to derive the compression face response for the actual test specimens so that they retain their original surface condition (heat tint etc) Curvature from pipe can give 10% variation in stress across specimen for axial specimens. Misalignment of 1.5mm or greater, can lead to increased stress at weld root. Welded samples of high strength wires for flexibles have flash butt welds. Prefer to test welds in uniaxial tension, since we then load the entire cross section of the weld. If welds are tested in 4pb, found deviation between stress and deflection larger than for non-welded samples. In consequence for welded samples we always mount strain gauges on each specimen	Note EFC17 Appendix 7, in 7.3 Loading: General, already requires that CRA loading requirements be derived by use of strain gauges Two partial exceptions are given; ferritic and martensitic stainless steels can rely upon use of the equations for deflection for homogeneous specimens free of cold work. Similarly the NACE TM0177 C-ring equation can also be used. It is recommended that strain gauges be used to determine accuracy. Is it necessary for C-steel parent materials which are homogenous and simply a beam? Experience from QA/QC tests seem to indicate these are acceptable. **Potential resolution?** CRA - retain the need to strain gauge as per EFC17 C-steels - strain gauge weldments and inhomogeneous/non-uniform specimens, and for parent materials at elastic limit?
8	Evaluation of specimens	All	Visual examination is insufficient - especially for weldments. Need agreement on magnification for examination and then sectioning (and appropriate magnification for examination of the metallographic section). **Note: ISO15156-2 requirements in B.4.3.2** **B.4.2.3 Evaluation and acceptance criteria for FPB test specimens** A wet-magnetic-particle examination shall be carried out on the side of the sample that was under tensile stress during H_2S exposure. Below any magnetic particle indications running perpendicular to the stress axis, metallographic sectioning	Should specimen be etched or unetched for examination? (See NACE TM0284) Maginfication <100X and take metallographic section? Acceptance criteria required. **Potential resolution?** Derive consensus that sectioning is required especially where no visible cracking can be seen but, note avoid too high a magnification for inspection (say, X200 max?) BP examination requirements after exposure

			shall be made perpendicular to the indications or, in the absence of magnetic particle indications, at least two metallographic sections shall be made parallel to the stress axis of the specimen. Sections produced in these ways shall be examined for possible ladder-like HIC features and other cracks related to SOHIC or to the soft zones of a weld (SZC). No ladder-like HIC features nor cracks exceeding a length of 0,5 mm in the through thickness direction are allowed. To assist the detection of damage, specimens may be plastically deformed by 5 % in the previous bending direction prior to metallographic sectioning. Prior to deformation, the specimens shall be heated to 150 °C and maintained at that temperature for 2 h to remove absorbed hydrogen. Damage developed on the tensile side of a specimen in the form of blisters less than 1 mm below the surface, or on the compression side regardless of the depth of the blister, may be disregarded for the assessment of SOHIC/SZC but shall be reported.	require:- Examine specimens using low powered microscopy at ×40 magnification. Photographic evidence of cracking shall be recorded. Where no surface cracks are visible, undertake longitudinal sectioning at two locations (typically at 1/3 and 2/3 through thickness) and conduct metallographic examination at up to ×100 magnification. If cracking is observed, the maximum crack depth and path shall be recorded.
10	Evaluation of specimens - pitting	Steel	Distinction between pits and cracks when pits are sharp features	**Potential resolution?** Agree flowchart approach for this to provide consistent interpretation
11	Evaluation of specimens - pitting	CRA	Need to reach consensus on assessment of pits on CRA specimens - define dimensions, agree on sectioning and polishing back through pit?	**Potential recommendation** Per TWI JIP on 316 sour service limits, pitting was discussed with industry and agreement reached on sizing per BP approach. BP approach:- "If pitting is observed, the maximum pit depth shall be recorded (ISO 11463:1995). Pit depths of less than 25 µm may be considered shallow and after an exposure of 30 days are likely to be non-propagating. The observation of deeper pits is of more concern as it may lead to transformation to a crack." Need agreement upon the acceptability critieria

12	Stress distribution on weldments	All	FEA models show the non-uniform nature of the stress distribution hence thicker specimens preferred	**Potential recommendation** Full thickness or 8mm which ever is greater, as above
13	Constant deflection - creep under load at room temperature	CRA	Procedure to apply additional load during period prior to placing specimen in autoclave. Monitor with strain gauges but only prior to testing.	Constant deflection is most experimentally convenient in autoclaves, anyone using constant load? **Potential recommendation** Creep curves in EFC17 and hence overload to achieve 100% proof stress load during test?
14	Loading to match the properties of the material at test temperature	CRA	ISO15156-3 actually requires loading to the test temperature value, which of course has to be done at room temperature outside of the autoclave for 4pb. Using a calibration specimen (parent plate or weld specimen as appropriate) load the specimen at the test temperature. Note the displacement/deflection required. Then, at ambient temperature, load all subsequent specimens to this deflection. Assumes that we will achieve the same strain level despite possible variations in weld characteristics from specimen to specimen; actual strains will be unknown and may be greater or less than 0.2% value; load will initially be high before relaxing to final value. (Meets ISO15156 guidance) NPL recommend load at ambient temperature to achieve the ambient temperature 0.2% proof stress under bend as monitored by strain gauge and then fix the deflection. On raising the temperature to test value the deflection is fixed and so the load will relax. The specimen is now overstrained - conservative. BP requirements are that the Method 1 (specimen loaded at ambient temperature to achieve the ambient temperature 0.2% proof stress under bending) is applied.	Consider this as warm pre-stressing? Loading jig could possibly be designed with higher CTE material thus raise load during test? Need to agree on the basis of loading and modify ISO15156 if industry so determines if BP method is preferable in terms of loading at ambient temperature.
15	Clad materials	CRA & Steel	How to adequately mask substrate especially around loading rollers?	Could consider modelling for the removal of substrate or is this a similar issue to consideration of residual stress in small-scale vs. full-scale tests?
16			Test without substrate removes masking issue	Not clear what load would be applied
17		ALL	Acceptance criteria	

Appendix 3

Laboratory testing of materials to qualify clear brine fluids for use in wells

1 Introduction

To avoid corrosion related problems when using clear brine fluids, the use of materials compatibility and corrosion prequalification testing is important. These guidelines give information on the types and extent of expected corrosion mechanisms and advice on the requirements for materials compatibility and corrosion testing needed before considering a brine with no or limited field experience.
In this document, clear brines include completion, packer, workover and well suspension fluids. Clear drilling fluids are not included in the scope of this document.

2 Guidelines

All brines without previous adequate relevant testing or adequate relevant field experience should be tested. Any change in salt content, additives and/or substitution of chemicals may require additional testing. If chemical concentrations are modified then the need and requirement for any additional testing is subject to end user evaluation/approval. The brine make-up shall be defined in a written specification that identifies all components, compositional limits and controlled properties.

The testing may be divided into two stages, with different responsible parties for each stage;

1. Pre-qualification tests; to be undertaken by the Brine Vendor
2. Qualification Tests; to be undertaken by the Brine Vendor together with end user collaboration.

Both test stages will, in most cases, require a test pressure above atmospheric pressure. Autoclave testing should therefore be expected.

The pre-qualification tests will use a simple test set-up, aimed to identify chemicals which either;

- decompose under the simulated operating conditions, or
- cause corrosion and/or environment stress cracking (ESC) of materials commonly used downhole, ref Table 1.

Important parameters in the pre-qualification test will be temperature, pressure and brine chemicals/constituents, including pH.

The qualification testing should normally be undertaken after successful pre-qualification tests. The qualification testing will more accurately reproduce the expected operating conditions for specific applications and will assess the intended downhole materials. More detailed test methods will be used to more accurately evaluate both general and localised (pitting/crevice) corrosion, and ESC (including hydrogen embrittlement) of metallic materials.

All test specimen preparation and loading shall be in accordance with the following Standards (Ref Section 2.7~~3.3~~):

- Weight loss specimens: ASTM G1
- U-bend specimens: ASTM G30
- Tensile test specimens: ISO 8692-1
- SCC testing in accordance with ISO 15156 part 1-3.

2.1 Pre-qualification tests

This testing should be completed by the vendor prior to the brine being offered to the end user. The testing can be used to establish a recommended maximum temperature for the brine to avoid decomposition into products which may crack materials or accelerate corrosion. The testing should also ensure compatibility with commonly used metallic oilfield materials at this maximum temperature.

The testing should comprise thermal stability testing and materials testing, separately or together. Test requirements are shown in Table 1.

Common Test Parameters	Requirement, All Tests
Temperature	Test temperature should be higher than, or equal, to the intend~~t~~ed application temperature (to be stated by end user) See also Section 2.3
Brine and chemical additives	Industrial grades (i.e. similar to those to be used in the final product) shall be used. The full chemical package in accordance with the written specification shall be used in the test[1]
Test environments	Environment 1: No acid gas contamination and no forcible oxygen removal condition Environment 2: Gas contamination condition, the actual partial pressures of CO_2 and other gases anticipated in service should be used[2]
Exposure time (min)	90 days (minimum)
Material types[3]	• Low alloy steel (LAS) tubing/casing grade (110 ksi strength grade preferred) • 13Cr steel (e.g. L80) • 13Cr/5Ni/2Mo Super martensitic stainless steel (SMSS) (110ksi strength grade preferred) • 25%Cr Super duplex stainless steel (SDSS steel) (125ksi strength grade preferred) • Super-austenitic stainless steel (e.g. 28%Cr 125ksi strength grade preferred), • Ni alloy (125 ksi strength grade 718 preferred)
Material Sample Types	• Weight-loss/pitting corrosion coupons • Tensile Tests before and after exposure[4] • U-bend SCC for CRAs [5]
Reporting	• General corrosion rate • Localised corrosion (rate and type) • Environment sensitive cracking (ESC)[5] • Change in ductility (elongation and/or area reduction). • Chemical degradation, change in autoclave pressure and pH should be identified [6]

Table 1: Test Parameters and Requirements, pre-qualification test

Notes:

1. Corrosion inhibitors, oxygen scavengers and all other chemicals that are likely to be present in an actual field application should be included in the tests. The brine make-up should be defined in a written specification identifying all components. Note that tap water chemistry may vary significantly.
2. H_2S may influence test result and should be considered when expected in operation. The solution should be purged for at least 1 hour or until equilibrium is reached using the test gas mixtures and prior to pressurising the autoclave/starting the test.
3. Decomposition may be catalysed by certain materials, especially Ni-alloys. This should be considered when selecting autoclave and other test equipment materials. All types to be included for generic testing. For specific

applications materials not to be used may be omitted or additional alloys substituted by agreement with the end user. Care should be taken when mixing different materials in the same autoclave. Galvanic corrosion and other interactive effects should be considered,.

4. Tensile Test (at 10^{-5} s^{-1} strain rate) to be undertaken on tested and untested samples to evaluate ductility loss. Minimum 2 repeat tests recommended.
5. U-bend selected for consistency and simplicity. Other tests may be used if agreed by the end user, but these are not encouraged. Evaluation of test results should include specimen sectioning once at centre line for round-bar specimens; minimum t~~T~~wice ~~(3 times?)~~ (1/3 and 2/3 through width) for strip specimens AND/OR through any 'suspect' areas identified from the previous visual examination.
6. This would typically include chemical analysis before and after test and, if necessary, identification of possible reaction mechanisms and end products. It is recommended to continuously monitor and record temperature and pressure for the duration of the test. pH measurements should be made in a standardised atmosphere.

2.2 Qualification Test

This test should be undertaken by the vendor only after agreeing the testing conditions/programme with the end user. The purpose of the test is to qualify the brine for specific applications, as defined by the environment/materials combination (or a range of applications, environments and materials).

Typical test requirements are shown in Table 2.

Possible galvanic effects should be considered. ISO 15156-3 may be used for guidance on Galvanic Hydrogen Stress Cracking (GHSC) testing.

Common Test Parameters	Requirement, All Tests
Temperature	Design/Maximum Temperature (see also Section 2.3)
Brine and chemical additives	Industrial grades (i.e. similar to those to be used in the final product) shall be used The full chemical package in accordance with the written specification shall be used in the test[1]
Test environments	Environment 1: No acid gas contamination and no forcible oxygen removal condition Environment 2: Gas contamination condition; the actual partial pressures of corrosive gases anticipated in service (typically CO_2 and H_2S) should be used[2]
Exposure time	90 days[3]
Material types	Application dependent[4]
Material Sample Types	Weight-loss/pitting corrosion coupons Tensile Tests after exposure[5] SCC Tests[6] Crevice corrosion coupons (metal-to metal) Hydrogen samples[7]
Reporting	• General corrosion. • Localised corrosion (rate and type) • Environment sensitive cracking (ESC)[8] • Change in ductility (elongation and/or area reduction) • Hydrogen content before and after testing[7] • Chemical degradation, change in autoclave pressure and pH should be identified [6]

Table 2: Typical test Parameters Requirements, Qualification Test

Notes:

1. Corrosion inhibitors, oxygen scavengers and all other chemicals that are likely to be present in an actual field application should be included in the tests. The brine make-up should be defined in a written specification identifying all components. Note that tap water chemistry may vary significantly.
2. CO_2 and H_2S mixture shall be bubbled through the solution for at least 1 hour or until equilibrium is reached prior to pressurising the autoclave/starting the test.
3. Shorter exposure times can be justified based on pre-qualification test data or shorter field exposure periods.
4. All materials in contact with brine in service should be tested.
5. Tensile Test (at 10^{-5} s^{-1} strain rate to be undertaken on tested and untested samples to evaluate ductility loss. Minimum of 2 repeats recommended.
6. One or more test method of either 4PB (100% AYS), C-ring (100% AYS) or Constant Load (90% AYS). DCB testing may be used for low alloy steels if agreed with end user. Only the test methods suitable for the candidate materials being evaluated should be selected, ref ISO 15156 part 2 and 3.
7. Optional test; to be decided with end user. Hydrogen to be measured after cleaning by combustion method. Samples to be stored cryogenically immediately after removal of autoclave until combustion. The following should be approved by the end user before testing:
    - Specimen size and cleaning procedure
    - Technical and calibration procedures

   Hydrogen measurements should be evaluated together with tensile samples (ref note 4). Acceptable hydrogen levels will vary with alloy and acceptable levels should be established together with the end user.
8. Test specimen to be sectioned once at centre line for round-bar specimens; minimum Twice (1/3 and 2/3 through width) for strip specimens AND/OR through any ‘suspect’ areas identified from the previous visual examination.
9. This would typically include chemical analysis before and after test and, if necessary, identification of possible reaction mechanisms and end products. It is recommended to continuously monitor and record temperature and pressure for the duration of the test. pH measurements should be made in a standardised atmosphere.

2.3 Other considerations

In some cases the maximum temperature may not represent the worst case for some cracking mechanisms. Table 3 gives guidance to other temperature ranges which may be considered when testing brines for specific field applications.

Material type	Failure mode	Worst case temperature
Low Alloy Steels, 13Cr/S13Cr	SSC, SCC	Ambient for SSC Maximum design for SCC [1]
Duplex	SSC/SCC (mixed mode) SSC	80-100 ^oC Max. design
Alloy 718	HSC SCC	Ambient for HSC[1] Maximum design for SCC [1]

1. May become enriched by H at high temperature and brittle when temperature reduces.

Table 3: Worst case test temperatures

Additional information

2.4 Definitions and abbreviations

Abbreviations

4PB	Four point bent beam
DSS	Duplex Stainless Steel
ESC	Environmental Stress Cracking
GHSC	Galvanic Hydrogen Stress Cracking
GL	Guideline
HE	Hydrogen Embrittlement
HSC	Hydrogen Stress Cracking
LAS	Low Alloy Steel
N/A	Not Applicable
P	Pressure
SCC	Stress Corrosion Cracking (occurs at high T)
SDSS	Super Duplex Stainless Steel
SMSS	Super Martensitic Stainless Steel
SSC	Sulphide Stress Cracking (occurs at low/ambient T)
T	Temperature

2.5 Definitions

<u>Brine (or Clear brine fluid)</u>
- covers a range of solids free solutions that are used for the following applications:

- *Completion and Workover Fluids* - can be defined as any fluid that is pumped downhole to conduct operations after the initial drilling of the well. These fluids are employed to balance formation pressures, circulate / transport solids and protect the formation during well killing, perforation, fishing and gravel packing operations.
- *Packer Fluids* – are the fluids present in the annular volume between the tubing and the casing above the production packer. The completion fluid may remain in the annular space following the packer setting operation thus becoming the packer fluid. Alternatively, the completion fluid may be circulated out of the annular space to be replaced by dedicated fluid. Packer fluids typically remain in the annulus for extended periods during which time they may become contaminated with produced fluids or lift gas.
- *Well Suspension Fluids* - A fluid that is left in a completed or uncompleted well for a longer period of time, to maintain well control and integrity prior to bringing the well onto production.
- *Drilling Fluids* – are fluids used during the drilling of a well. These fluids are typically circulated down the drill string and up the annulus to cool the bit, transport cuttings and stabilise the well bore. Formate brines may be employed as drilling fluids for specialised applications. The selection of clear brine drilling fluids is considered out with the scope of this document.

2.6 Changes from previous version

N/A

2.7 References

ASTM G1	Standard Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens
ASTM G 30	Standard Practice for Making and Using U-Bend Stress-Corrosion Test Specimens
ISO 6892-1	Metallic materials - Tensile testing at ambient temperature
NACE MR0175/ ISO 15156	Petroleum, petrochemical and natural gas industries — Materials for use in H_2S-containing environments in oil and gas production
NACE TM0177	Standard Test Method. Laboratory testing of metals for resistance to specific forms of environmental cracking in H2S environments.

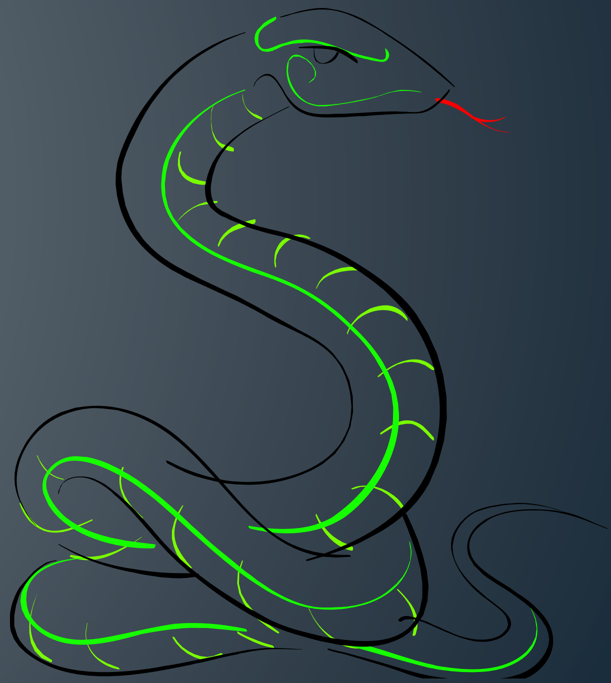
Appendix 4

Corrosion Inhibitor Science Not Snake Oil

Presentation

Focus from the oil and gas industry viewpoint

- Snake oil?
- Mechanisms
- Structure development
- Formulation
- Partitioning
- Solids
- Preferential weld corrosion
- Top of line corrosion
- Environmental considerations
- Compatibility
- Future challenges
- Summary



Snake Oil

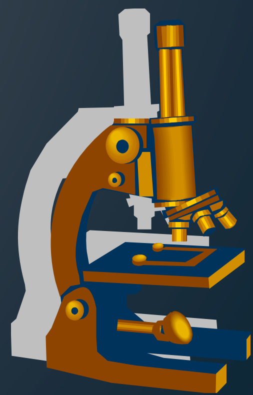
- Corrosion inhibitors have been known to be in use for at least 70 years
- Early days no one understood how they worked
- Earliest mention I have found 1940's when it was found long chain organic compounds with polar functional groups had corrosion inhibition properties [1]

- The mechanism of action was unknown but the study of how they worked began

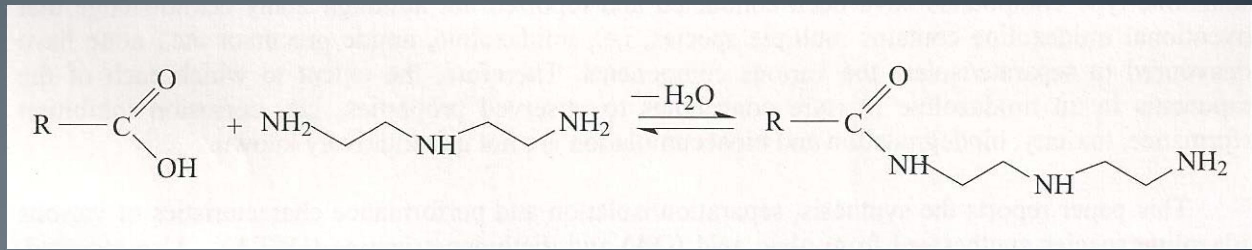
1. Gough, Durnie, Auty; Characterisation , isolation and performance characteristics of Imidazolines; Paper no 02301 NACE 2002.

Snake Oil

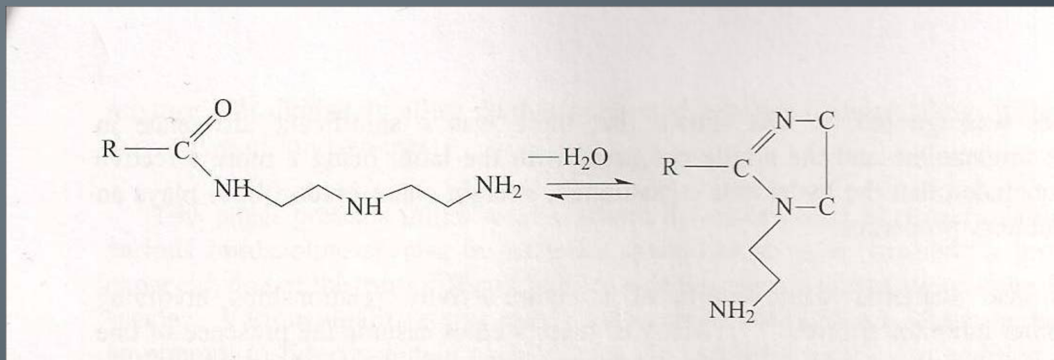
- First work studied the actual chemical composition
- Example Fatty acids and amines were know to give inhibition at low concentrations
  - Reacting these together at high temperatures it produced the first amides and imidazolines
- Tailor made corrosion inhibitor molecules of known structure
- Start of move from snake oil to designed chemicals
- Other chemicals studied
  - Nitrogen based active groups



Imidazolines



Fatty acid + Amine   (Condensation reaction)   Amide



Amide   (Condensation reaction)   Imidazoline

Acknowledgment. Gough, Durnie, Auty; Characterisation , isolation and performance characteristics of Imidazolines; Paper no 02301 NACE 2002.

Scientist (Chemical Structures)

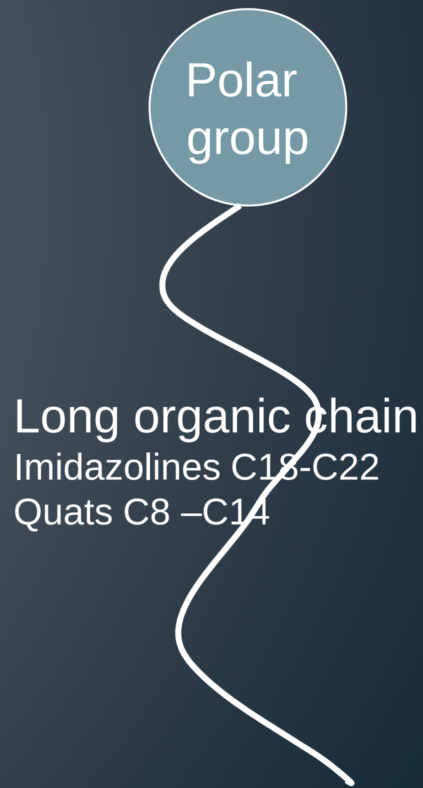
- Once structures were identified improvement could be made
- In the case of imidazolines
  - Chain length on fatty acids
  - Amine types (MEA, DETA, TEPA)
  - Reaction conditions
- Need to find improvements while keeping costs low
- Cost efficient products
- Similar developments for other types
  - Quaternary amines
  - Amines
- Although structures important formulation was also

Scientist (Formulation)

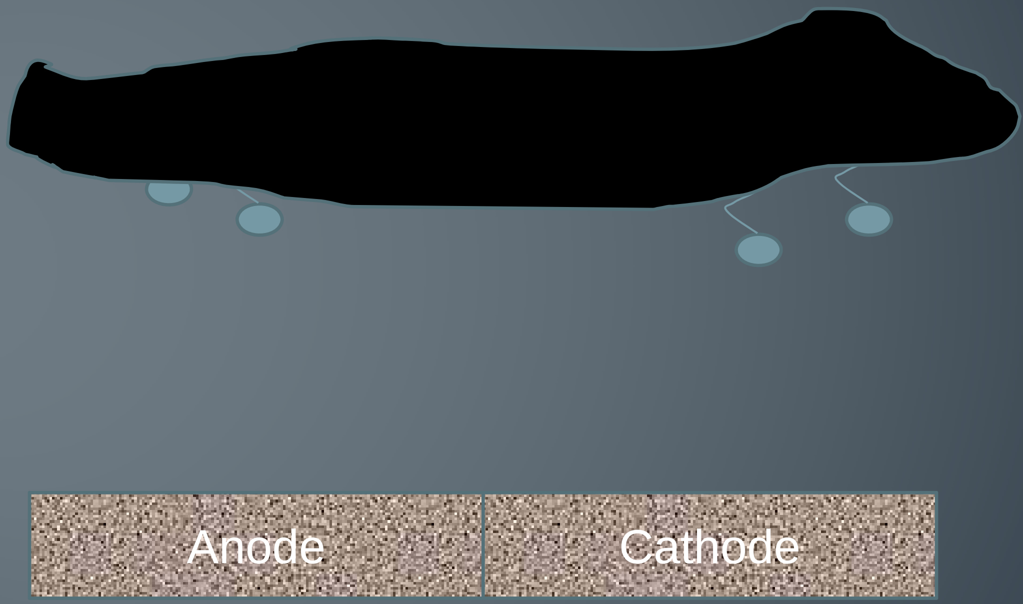
- Most commercial inhibitors combination of bases
- Including synergists
- Understanding of formulation effects
- Thiosulphate effects studied 90's
  - Formation of thin sulphide film
  - Other components in product then form more efficient film
  - Requirement of thiosulphate confirmed not just a way of passing lab test
- Partitioning another area affected by formulation
  - Neutralisation of components to make them water soluble
  - Level of neutralisation for water solubility without hydrolysing products
  - Imidazolines in acid conditions can be hydrolysed back to amide, forming less efficient product.

Mechanisms

- Once structures known, how do they work?
- They are surfactants (work at surfaces)
- Adsorption of inhibitors onto metal surface
  - Anodic
  - Cathodic
  - Mixed inhibitors
- Filming inhibitors attract oil film
  - Simple Mechanism
- Passivation other mechanism
- Combined effects of complex formulations
- More complex mechanisms
  - Colloidal interactions



Simple Mechanism



Scientific Approach to Mechanisms

- Adsorption isotherms
  - Langmuir
- Adsorbtion on scales
- Partitioning behaviour
- Synergists
- Effects of oil condensate properties
- Flow
- Structural activity relationships
  - Relationship of various parameters of chemicals including charge density and molecular surface area, volume, density
- Scientific studies have been carried out in all these areas

Advances

- Chemicals were blamed for alot of failures
- Not necessarily the chemistry the cause
- Application
- Understanding system requirements
- Inappropriate testing regimes
- All factors that can affect inhibitor performance
- Testing regimes extensive now
- Recent field developments require inhibition concept to be proven during design
- Key in design decisions

Partitioning

- Partitioning became high profile 1990's and 2000's
- Failures were attributed to partitioning behaviour of chemicals
- Water soluble product became favourable in high and low water cut systems
- Export pipeline partitioning into stagnant water areas important
- Can cause operational issues
  - Separation
  - Separated water quality
  - Formulation solutions for these

Solids / Under deposit corrosion

- Adsorption of inhibitors onto solids
  - Suspended and deposited
  - Reduced ‘availability’ to the metal surface
- Deposits in unpiggable system seen as causing problems
- Test methods needed development
  - CAPCIS IFE SGS methods available
  - Different ideas on procedure to use
- Adsorption on solids and need to penetrate the solids required
  - Studies found that under deposit polarisation could occur
  - It is thought that cathodic inhibitors are more effective under these circumstances
  - The search for efficient cathodic inhibitors?

Preferential weld corrosion

- Corrosion failures seen in weld or Heat Affected Zones (HAZ) in weldments
- Inhibitors injection can make the problem worse
- 1% nickel welds worse than matched consumable CMn steel
- Under dosing seen as big issue
- Partitioning and dose important for mitigation
- Testing procedures developed
- Testing for PWC routine for some operators inhibitor selection programs
- Vendors have adopted this approach

High temperature testing

- As system get hotter inhibitor selection harder
- Chemical can break down at high temperatures
- Need to test under high temperature conditions
- Use of autoclaves or high pressure flow loops required
- Particular requirement of synergy with surface films at high temperature
- High temperature testing routine now
- Used in conjunction with other testing methods
  - Under deposit
  - Preferential weld tests

High Pressure Testing

- High pressure fields being developed
- High concentration CO_2 and H_2S
- Testing under field conditions
- Realistic recommendations can then be made
- Autoclaves or high pressure flow loops

Top of line corrosion

- Inhibition techniques not easily applied
- Effect of Volatile Fatty Acids
- Volatile inhibitors
  - Similar chemistry to conventional products
  - Seem to need very high doses
    - Neutralisation verses inhibition
- Related to application
  - Spray pig
  - Flow regimes
  - Pigging

Environmental restrictions

- North Sea and other areas have environmental restriction on use of chemicals
- This has ruled out commonly used products
  - Imidazolines
  - Quaternary amines
- Development of new molecules or
  - Variations on standard chemicals
  - New molecules
- All this increase cost of chemical approvals
  - Chemical registration (EINECS, CHARM) expensive
  - Need market to justify development
- More stringent legislation in future?
  - Fate of chemical?

Compatibility Issues

- Was not normally considered
- Compatibility with other production chemicals
  - Hydrate inhibitors
    - KHI direct compatibility
    - Methanol (oxygen issues)
  - Glycol regeneration
- Demuslifiers dehydration systems
- Separated water cleanliness
- Materials Compatibility

Future Challenges

- Higher temperatures and pressures
- More stringent environmental restrictions
  - Fate
    - Breakdown product analysis
- Elemental Sulphur Corrosion Inhibition
  - Currently proving difficult
  - Testing procedures
- High levels of carbon dioxide inhibition
  - Carbon Capture and sequestration projects
- Oxygen inhibition
  - Still not easy
  - Methanol
- Reliability of Injection systems
- Others?

Summary

- Inhibition has developed over the years
- Much more known about mechanisms
- Recently developments have accelerated
  - New consideration every few years
- Suppliers are addressing the issues
- A lot of misconception on inhibitors not working
  - Application failures
- Challenges are still there
  - Environmental
  - Cost
  - High temperature
  - High pressure (CO_2 / H_2S partial pressures)
- Oxygen

Summary

- The chemical vendor (snake oil salesman) has moved to being a key scientist in oil and gas industry
- Many names known
- Not just organic chemists
  - Electrochemist
  - Chemical engineers
  - Metallurgists
  - Materials engineers
  - Corrosion engineers
  - Integrity engineers
  - Mechanic?
- All functions required
- A team of scientists required

Appendix 5

Corrosion in O&G industries Committee (CIPG)

French Federation of Corrosion (CEFRACOR)

http://www.cefracor.org

CEFRACOR

CENTRE FRANÇAIS DE L'ANTICORROSION

Brief history

- Septembre 2006: Creation of the committee, impulsed by Marcel ROCHE (Total)

- Definition of 9 working groups

- More than 150 people interested in the activities of the committee

- More than 50 active participants

WG1**: List of companies active in O&G products or services**

WG2**: List of academic or professional training centres in corrosion**

Goals of these 2 WG:

To provide useful informations to members of Cefracor (Suppliers, training, educated people…)

WG3**: Internal corrosion**

WG leader: Delphine ZUILI (Technip)

Working group focused on corrosion of carbon steel in oil and gas process stream and including utility units usually present in oil and gas plant

Main objectives**: to edit guidelines on:**

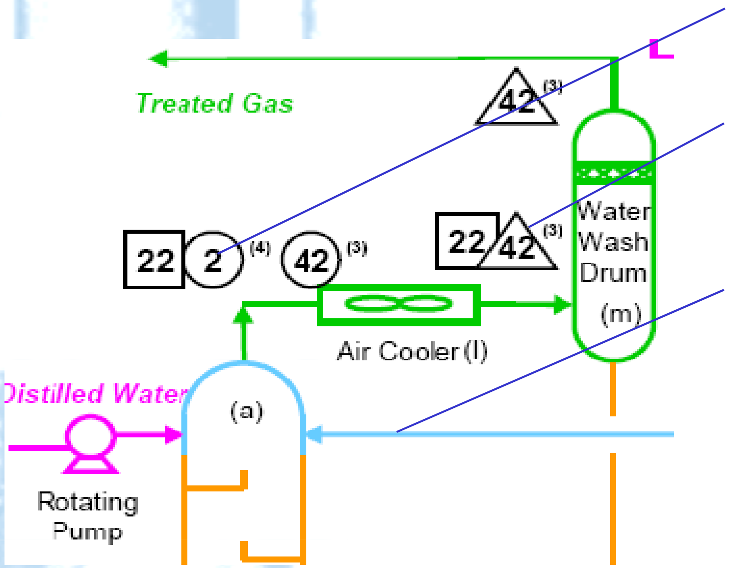
- Prediction of corrosion risks

- Corrosion monitoring

WG 3: Internal Corrosion

Prediction of corrosion

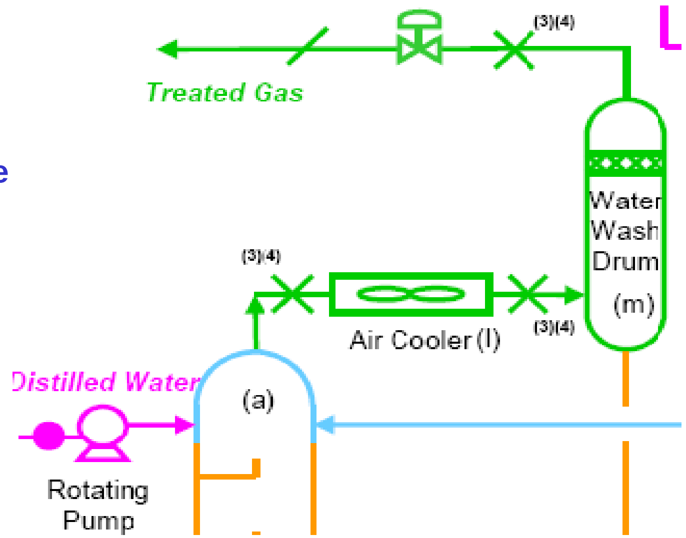
- Assessing corrosivity of process fluid is the first step to prepare corrosion control management plan, including **corrosion monitoring program**
- Taking into account analogue job done for refinery (ref to API RP 571), the working group develop schematic process flow diagrams per unit or per system:



Number refer to corrosion mechanism

Triangle refer to low severe
Square refer to medium severe
Round refer to severe

Colour refer to corrosion loop



1st scheme => corrosivity of the process fluid

2nd scheme => information about material selection

Corrosion monitoring

Corrosion monitoring consists of evaluating corrosivity of process fluid during operation by controlling the process fluid conditions and on-line corrosion rate of the equipment

It must be distinguished from inspection activities which give information of the equipment conditions.

Both are part of the global corrosion control management of the installation

The objective of the working group is to prepare a guideline to support corrosion engineer for designing or updating corrosion monitoring during design phase or during operation.

The **Prediction of corrosion** theme help for preparing the corrosion monitoring program during the design phase.

WG4**: Alternative materials (other than CS)**

WG leader: (Laurent MACQUET, Saipem)

Main activities**: Case studies:**

- to identify situations where CS is not fully satisfactory

- to propose other material solutions

WG5: **Environmental cracking**

WG leader: (H. MARCHEBOIS, Vallourec)

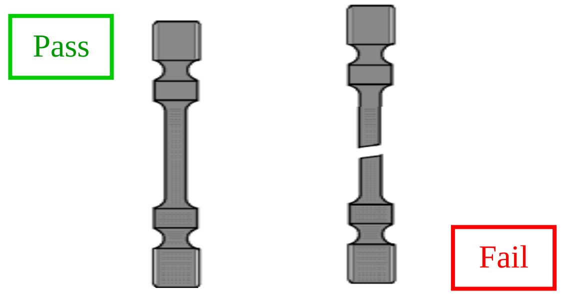
Main activities:

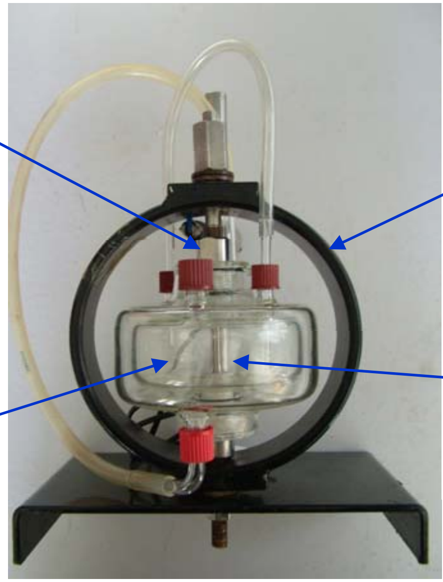
- To share information on the topic (both upstream and downstream)
- To discuss on test procedures (Unaxial Tensile, HIC, DCB, FPB, Corr-Fatigue)
- To visit corrosion laboratories
- To dicuss technical papers, standards relevance
- To present scientific works
- To organize one-day seminars
- ...

WP 5: NACE TM0177 Method A: Basics

Uniaxial Tensile test

- *NACE TM0177-05 Method A*
- *Standardized test in the OCTG industry*
- *Critical test seen as a reference*





Test is stopped after 720 hours (1 month) if no failure has occurred… otherwise, TTF is checked

WP 5: NACE TM0177 Method A: Back to the history

Method A = Unaxial Tensile test

Results of Interlaboratory Sulfide Stress Cracking Using the NACE T-1F-9 Proposed Test Method*

(Materials Performance Sept. 1977)

J. B. GREER

Exxon Production Research Company, Houston, Texas

Eighteen companies participated in interlaboratory sulfide stress cracking tests using the NACE T-1F-9 Proposed Test Method as a core program. The results of testing are presented along with suggested modifications for T-1F-9 consideration.

2. The sulfide stress corrosion cracking data are logarithmically related to time, *i.e.*, $\sigma = \sigma_0 - \beta_{log}t$, where σ is stress, σ_0 and β are material constants, and t is time.

3. Variations in NaCl content are not an important parameter for low alloy steels, however, it is necessary to exclude oxygen by continuous H_2S bubbling during the experiment.

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WP 5: Is Uniaxial Tensile the right methodology?

2.2.7 EC test results can show statistical variability. Replicate testing may be needed to obtain a representative value characterizing resistance to EC.

from NACE TM0177-2005

Variability induced: by the <u>SSC mechanism</u> vs. the <u>test procedure</u>?

WP 5: Influent parameters that may affect the SSC test result

During samples' preparation

Before sampling
- Inclusions
- Cracks

Sampling
- Cutting: tool
- Site: around circumference, thickness
- Storage, handling

Machining
- Tool, parameters
- Residual stresses
- Polishing
- Design
- Surface roughness

SSC Results scattering

Handling
- Storage
- Gloves
- Cleanliness

Equipment
- Proof rings
  - Calibrated
  - Uniform/ reproducible loading
- Threads
- Ball thrust bearing
- Airtightness
- Corrosive solution

Test implementation
- Gas composition
- Quality of water, NaCl, *etc.*
- Temperature maintained

Post test
- Stripping down
- Storage
- Chemical post treatment

During samples' tests

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WP 5: Important parameters to be checked

CIPG WG 5 "Environmentally Assisted Cracking" - Important parameters to be checked with regard to NACE TM0177 Uniaxial Tensile test (Method A)

Sampling/Machining of the specimen

Sampling/Machining of the specimen	Action	Check list	Recommandations/examens	Commentaires
	Condition of receipt	Data on material to be tested (origin, manufacturing process, mechanical properties, etc.)		
	Sampling	Sampling orientation, hardness, homogeneity/heterogeneity, sampling within the wallthickness (OD, middle, ID)	Conformity to Standard, Guidelines, Specification	DNV OSF 701: Location of the sample - Suggestion to use FPB test with full thickness + Test on as welded material if possible -> How to correctly apply the load? Sampling at mid-wallthickness on thicker products. Measurement of the Yield Strength - Reproducibility and Reliability in order to calculate the stress value (Rp0.2, Rt 0.5, Rt0.6, Rt0.65, Rt 0.7 - cf. ISO 11960/API 5CT) Remark: Flexible risers - the wire is tested without any machining nor surface premaration
	Cutting/Machining	**Machining tools ("rapid steel", carbide/ceramic tools)**	Adéquation tour/CNC avec vitesses de coupe, profondeur de passes	Adéquation NACE TM0177 avec nouveaux outils d'usinage CNC+plaquettes carbures/céramique ??? Non, selon les spécialistes d'usinage ! Effet de l'usinage / polissage : L'objectif étant de modifier le moins possible la nature de la couche de surface. **Quid des 0.05 mm pour les 2 passes finales (cf. NACE TM0177 Méthode A) ? Paramètres utilisable dans le cas d'aciers de coupe dits « rapides », et non dans le cas de méthodes plus récentes (plaquettes carbure et/ou céramique).**
	Design of the specimen	Type de design (conformité avec NACE TM0177)	Localisation de concentration de contraintes	1 rayon 15 mm mini pour le congé de raccordement, ou deux rayons, ou rétrécissement central afin de répartir les concentrations de contraintes Impact Standard size vs. Subsize size (difficulté d'usinage par CNC de faibles diamètres, 10% de sous-contrainte pour subsize - pertinence ?) REX : éprouvettes "subsize" (+ coeff. réduction de contrainte de 10%) plus critique que éprouvettes "standard"
	Surface preparation/Polishing	**Polissage mécanique sens travers**	homogénéité du polissage / présence de rayures (partiellement rebouchées ou non) ?	Différences d'état de corrosion lors de certains usinage - dissolution inégale entre le fût de l'éprouvette et les congés de raccordement Utilisation de meules de forme
		Polissage mécanique sens long	homogénéité du polissage / présence de rayures (partiellement rebouchées ou non) ?	
		Polissage mécanique automatisé/manuel	homogénéité du polissage / présence de rayures (partiellement rebouchées ou non) ?	
		Polissage électrolytique	Adéquation de la méthode avec le type de matériau	Eventualité de piqûres locales ou zones non dissoutes (précipités essentiellement) suivant la procédure REX : Sur nuance inoxydable, l'électropolissage est déterminant sur la réussite de l'essai (un acier inoxydable cassant après qq dizaines d'heures ne cassera peut être pas s'il est électropoli). REX : Signaux d'Emission Acoustique répartis de façon plus homogène après électropolissage par rapport à polissage mécanique (test réalisé sur 2 échantillons)
		Stockage	Décrire les conditions de stockage avant dégraissage (lieu, durée)	Dessicateur, protection anti-rayures/chocs ?
		Transport de l'échantillon	sachet, protection anti-rayures/chocs	
		Mesure de la section de l'échantillon	Décrire la méthode : Calcul de la section réelle. Noter la valeur	Ovalisation de l'éprouvette +/- 0.02 mm ? Utilisation d'un calliper ou projecteur de profil pour la mesure du diamètre mini ?
		. Type de solvant (iso-propanol, alcool, acetone, autre)- Grade	Nettoyage de l'éprouvette avec un coton , puis nettoyage bac US avec de l'alcool/eau puis de l'acétone	
	Roughness	**Fréquence des vérifications ?**	Privilégier Rz et Ra plutôt que Ra seul	Dépend du surfaçage des éprouvettes après usinage **Pertinence du Ra (0.81 µm dans NACE et 0.2 µm dans EFC) – Privilégier le Rz.** Impact de la couche écrouie suite à usinage et polissage (couche de Beilby).
	Cleaning/Decreasing	. ASTM F2165 ou autres		
		. Environnement	décrire	
		. Durée	Préciser la durée de stockage entre dégraissage et essai.	
	Storage		Moyens de protection des éprouvettes	

Solution preparation

Solution preparation	Action	Check list	Recommandations	Comments
	Gas (H_2S or CO_2/H_2S – N2)	Quality, grade, supplier	Specify O2 content (ppm (v) if known)	CO_2 / H_2S vs. N_2 / H_2S comparison ? pH "drift, shift". **N2 or CO2 degassing ? – better with CO2 20 mn degassing (see TM0177) does not seem to be enough ? O2 concentrations max and min values ? (gas quality dependant)** NACE TM0177 : Inert Gas nature specified N2 or Ar. Degassing : faster with CO2 compared to N2 (BP tests) **NACE does not specify maximum O2 concentration depending on gas quality (O2 residual content)** CO2 / H2S better pH buffer (carbonates presence) Note : use an oxygen scavenger e.g. ammonium sulfite ? Impact on hydrogen absorption, solution chemistry change ? Note : visible pollution increases corrosion by twice
	Containers storage	Temperature / Storage time before use	Specify	Mixtures concentrations variations (seasonal ambiant temperature change e.g. colder in winter ???)
	water	Quality, supplier	Deionised water ASTM D1193 type (IV) other ?	Replacement frequency of ion exchange resins deionised vs. Distilled water
		pH	Measure water pH before solution preparation	pH meter calibration frequency, buffer solutions types (single use,…) No impact for NACE solution A but influence on cracking in pH buffered solution by strong acid use Note : solution pH (NaCl added) measurement with pH paper reads 7.0 whereas pH meter equipped with electrode gives more alkaline values 8.9 – max 9.3) ??? Cationic membrane electrode which does not differenciate Na+ et H+ ?
		Storage time and conditions before use	Specify	
	NaCl	Quality, grade, supplier	NaCl (quality, grade) ?	
		Storage	Storage time and conditions	
		Quantity added to water (NaCl weight/H2O weight ratio)	5% weight NaCl (TM0177)	
	Units / concentrations	**wt.% vs. g/L**	HACE TM0177 specifies wt.% NACE MR0175/ISO 15156 uses wt.% but refers to NACE TM0177 and EFC 16-17 EFC uses g/L European end-users specifications use g/L	5.0% weight corresponds roughly to 51 g/L,… Which unit has to be used ???
	Acetic acid CH_3COOH	. Quality, grade, supplier . Storage . CH3COOH quantity in solution (CH3COOH weight) . Storage of prepared solution NaCl + CH3COOH	glacial see NACE 0,5% CH3COOH (TM0177)	Solution A - NACE TM0177 "pH should expect to range between 2.6 and 2.8". Lab experience 2.6 or 2.7. See NACE Sol. A : Test solution pH before or after H2S saturation but before contact with a test specimen is expected to range between 2.6 and 2.8. (H2S pK superior to 2.6 => pH increases after saturation ???)

Test launching

Test launching	Action	Check list	Recommandations/examens	Commentaires
	Assemblies for the stress application (proof rings or other)	. Type, capacity maxi-mini, supplier		Constant load vs. proof-ring/spring loaded. REX : constant load more severe than proof-ring
		. Calibration - validation (method)	Calibration frequency - Method	Force sensor, strain gauges, tensile testing machine/compression
		Stress measurement	Comparator, gauges?	**« Bending issue » : API WG 1055 « Bending stress error »**. Russ Kane/Bruce Urband. + Articel NACE 2009 concerning bending
	Impact of the constraint vs. Corrosion speed			+ Articel NACE 2009
	Applied stress			Stress applied: 90% AYS: take account of the residual stresses and possible local plasticities due to defects. + specification in ISO 15156/MR0175"
	Sample fixing	. Description	Faire un schéma et une description précise de l'accrochage des échantillons	
		Specific sample fixing preparation (screw, torsion, ...)		
		Auto-centering system		
		Stress application- Slip or sliding?		
		Zero Reference		
		Jauge calibration		
		Influence of sample size		
		Galvanic couplings	No galvanic coupling. How to explain?	
	Sealing of the samples/test cell	. Description, efficacity, duration . Silicone grade (or other) used	Describe precisely how the sealing of the cell is guaranteed	Permeation of the tubings Remark : VITON absorbs rapidly CO2 and deteriorates
	Test cell	. Material, form, volume, thickness	Glas, plexiglass or PMMA	Different configurations (different labs) : - Inox + PFA (Teflon) - Viton - Inox + Glas + Teflon - Polypro +soft "polymer" - Inox + Teflon/Viton REX: Viton doesn't resist 720 hours crushing when using peristaltic pompes
		Supplier	Give precise information about the volume (H, diam) , thickness and the supplier	
		Cleaning	Clien nicely the cell before each test	
	Test: Fit-For-Purpose	pH adjustement during the tes	O2 polution possible if no precautions taken pH drift off- impact of vol. Surfaces ratio (standard: approximately 30 mL/cm2)	REX : comparison between test with pH 2.7 at the beginning and with pH 3 adjusted (constant) . pH 3.0 seems more severe than the Nace A solution pH 2.7 ?
	Buffered solutions	Limits the pH drift off	Impact of Hac vs. HCl or NaAc vs. Other buffer ?	Utilisation of different buffers : formic acide (short carbon chains, pK more acide). Perchloric acid (test without Cl-). Attention to the oxydation force of perchlorate (potential could be modified) . cf. article Eurocorr 2009 Eq : particular case for the supermartensitic SS : Impact of Ac- on Super 13% Cr ? Discussions concerning the impact of NaAc on the supermartensitic SS (cf. buffer with 0.04 wt.% instead of the usual 0.4 wt.%).
	Test duration NACE TM0177 Method A = 720 h.			Question : what is the origine of the 720 hours duration. A rapid statistic analyse shows the ruptures to be mainly in the duration of about 2 weeks (conditions Sol.A + 1 bar H2S)

WP 5: Important parameters to be checked

Sampling / machining of the specimen

- Machining tools (« rapid steels » vs. carbide or ceramic tools) still in line with NACE criteria, *i.e.* « in machining operations, the final two passes should remove no more than a total of 0.05 mm (0.002 in.) of material »?
- Manual mechanical polishing (long./transverse) vs. automated polishing
- Electrolytic polishing vs. mechanical polishing
- Roughness (R_z + R_a instead of only R_a) – frequency of the checking – 0.81 µm in NACE Std. instead of 0.2 µm in EFC 16

Solution preparation

- CO_2 / H_2S vs. N_2 / H_2S comparison? pH “drift, shift”.
- N_2 or CO_2 degassing? -> better with CO_2
- 20 mn degassing (see TM0177) does not seem to be enough? O_2 concentrations max and min values? (gas quality dependant)
- NACE does not specify maximum O_2 concentration depending on gas quality (O_2 residual content)
- Units/concentration: wt. % in NACE vs. g/L in EFC 16

Test launching

- Stress measurement - « Bending issue » *cf.* API WG 1055 « Bending stress error » or torsion effect

WP 5: To be cont'ed...

- **Need to share this work with EFC members and to discuss a potential update of the Std. / guidelines**
- **Publish a short paper on the topic summarizing the important parameters to be controled**
- **Organize « one-day seminar » on HE (2011)**
- **Open for collaboration and exchanges**

WG6**: Corrosion under insulation**

WG leader: (F. ROPITAL, IFP Energies nouvelles)

Main activities**:**

- to discuss EFC guide n°55 (share of REX)

WG7**: External corrosion**

WG leaders: (Sophie LAMBINET & Patrick LAMBERT, Trapil)

Main activities**: to edit guidelines on:**

- Selection of external coating

- Application procedures

WP 7: External coating of buried or immersed pipes

The aim of the document is to choose the best candidate coating in order to externally protect buried or immersed pipes and structures

- Part 1 is dedicated to general features allowing the reader to acquire a good knowlegde of the different kinds of external coatings.
- Part 2 is designed from an application point of view to help choosing the right product best suited to each case.

WP 7: External coating of buried or immersed pipes

Part 1

1-Object

2-Application area

3-Aim of external coatings and required properties

4-Main reference norms

5-Different kinds of stress undergone by external coatings during the instalment phase and in service

6-Different kinds of coatings Directory, in plant or on site applied

7-Annexes

- **-Historical review of coating**
- **-Corrosion phenomena**
- **-Cathodic protection theory**
- **-Essential properties of coatings**

WP 7: External coating of buried or immersed pipes

Part 2

1-Object
2-Typical characteristics summury chart
3-In plant built
3-1.Duty-related criteria choice
3-2.Surroundings-related criteria choice
3-3.Circuit configuration-related criteria choice
4-On site built
4-1.Duty-related criteria choice
4-2.Surroundings-related criteria choice
4-3.Circuit configuration-related criteria choice
4-4.Repairing kits
5-On site applying recommendations
6-Annexes
-Security, health care and regulations
-Surface duty requirements definitions guide
-Dielectric checking

WG8**: Internal inspection**

WG leader: (Didier CARON, GDF Suez)

Main activities**:**

- To share REX on inline inspection (ILI) tools

- To discuss normative guidelines

Objectives

- **Share transmission pipeline operators and ILI services compagnies experiency**
- **Analyse normative and guideline documents**
- **Sum up performances and using fields of Well and Pipeline Inspection tools**

Accomplished work

- Introduction to TRAPIL, NDT, ROSEN compagnies and their inspection tools
- Introduction to the transmission pipeline inspection policy of GRTgaz
- Introduction to the Guidelines from GESIP about pipeline transmission integrity management
- Introduction to the ANSI/ASNT ILI-PQ-2005 standard for the qualification and certification of ILI operators and analyst and background field from Rosen experiency on that standard
- Introduction to the long range guided wave inspection

WG9**: High temperature**

WG leader: (François DUPOIRON, Total Petrochemicals)

Main activities**:**

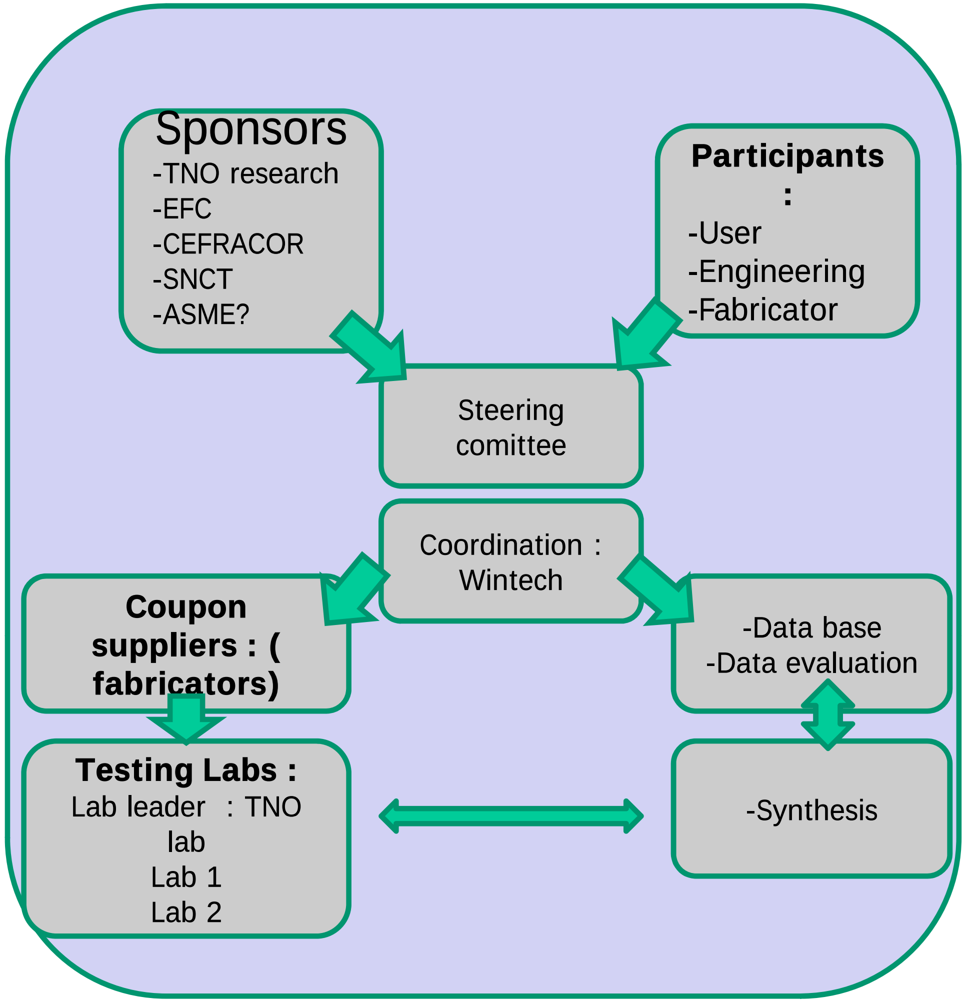
- Return of experience exchanges
- « Forum » between users (Petrochemical, Refinery , Chemical industries) , Research center , producers , fabricator, engineering
- Works on specific topics: Stress relaxation cracking

JIP in preparation

Alloy manufactures: Industeel, Outokumpu , Special metals , Haynes , VDM Krupp , Sumitomo, Sandvik , DMV , Metrode Boehler Thyssen ...

- Boiler makers: Verolmes , ATC , ACM ...
- Engineering: Technip , Fluor , Shaw (Badger) , Heurtey , Hude , Areva...
- End users: Total , BASF , Bayer , Dow Chemicals , Exxon Mobil, Shell , EDF, RWE
- Notified bodies on RBI: DNV, Bureau Veritas, TUV, AFIAP
- Independent research centers: TNO , BIL (Institut de soudure Belge) , EWI , DECHEMA ...

WP 9: High Temperature Corrosion



Summary

- **Some WG shall soon propose guidelines**
  - Possibility to share with EFC to end-up with EFC documents

- **Other topics of common interest**
  - H_2S testing methods
  - *etc.*

- **For more informations:**

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