COLLECTION OF ALL COMMENTS RECEIVED WITH MY (LIANE SMITH) FEEDBACK FOR MAJORITY

PLEASE NOTE THAT THIS REPRESENTS THE STATUS OF COMMENT REVIEW CLOSE TO THE END OF THE REVIEW PERIOD (CIRCA JAN/FEB 2008). AFTER THIS SOME FINAL EMAILED DISCUSSIONS DID NOT GET INCORPORATED BECAUSE OF TIME PRESSURE.

I WOULD LIKE TO RECORD MY THANKS TO EVERYONE WHO ASSISTED IN FINDING ERRORS AND ISSUES FOR IMPROVEMENT. THERE WAS A VERY SUPPORTIVE DYNAMIC EXCHANGE OF VIEWS BY EMAIL. THANKS TO ALL.

Proposed change	Reason	Comments rec'd	FEEDBACK
Section 8.2.1, page 24, last line: Delete sentence starting, "If hydrogen exit …."	Discussion within EFC group indicated that this text had been generally ignored and hydrogen exit was not considered to be a critical parameter (or not sufficiently impeded by the presence of CP); internal loading with hydrogen from the H2S was the critical issue affecting cracking risk.	CROLET - Hydrogen exit still is a critical issue (see NACE paper 02036). Most often however, and contrary to what was initially believed, exit is not the reverse reaction of charging, but a totally independent process (i.e. the usual physical degassing, but which is also surface and sulfide dependent). Therefore, the reality is that H exit is not linked to the presence or absence of another charging process on the opposite side. Unfortunately, since I did not attend discussions, I am unable to write a new sentence, but a mere cancellation is not wiser than the old one. BOND - Note the revision in ISO15156-2 just issued and the references given in the ballot if you wish to "substantiate" change. KERMANI - If people ignore the text, that does not imply its deletion – suggest reshaping the sentence allowing user to decide.	Adopt this change on grounds that - H exit is not linked to the presence or absence of another charging process on the opposite side. - ISO 15156 have taken lead on this point too.

Proposed change	Reason	Comments rec'd	FEEDBACK
		MARTIN - No problem with this and will be consistent with the recent changes to ISO 15156 Part 2 Table A.1 changes.	
Section A.3. Test Solutions. Add text- For tests requiring greater pH stability an alternative solution (NACE TM0177-Solution B) may be applied, adjusted to pH 3.5 or pH 4.5 depending upon the production conditions (gas or oil wells). This solution may be preferred for routine testing. Where this solution is selected it shall be identified as "NACE TM0177-Solution B" with the adjusted test pH.	Several labs have found that the EFC solution is not a good basis for testing. pH stability is poor, requiring continuous monitoring and frequent adjustment. The EFC solutions are proposed to be kept, as they may be argued to be most appropriate for specialist qualification testing, but introducing the NACE B solution gives an "easy" option where appropriate. NACE B is considered to be very stable at pH 3.5 and quite stable at pH 4.5.	Scoppio: For tests requiring greater pH stability NACE TM0177-Solution B is recommended, …….. This solution may be preferred for routine testing……………. CROLET - This "where appropriate" shall be stressed more, for not becoming a laxist abandonment KERMANI - This may give flexibility in conducting tests – however, we need to still emphasise that the main solutions "base solutions" – the publication does not stop people to use other solutions – agree with the inclusion of other options subject to user' approval. HASHIZUME - pH 4.5 should be deleted from the sentence. In my understanding, Solution B is just for pH 3.5. BOSCH - TM0177 Solution B is a "real" buffer system with a mixture of an Acid and the salt of this acid, pKa = 3,7. Fewer adjustments should be needed! MARTIN - We would vote against this change at this time. We are unable to support the proposal to make this addition without:- (1)Understanding the issues the 'several labs' have had with the EFC 16 solution chemistry. Whilst we would agree that there can be problems at the lower pH values (where more corrosion	OK ok they are still there. Message is that Soln B CAN be used with sufficient stability if adjusted to pH 4.5, indeed to higher pH up to 5.2 if necessary (others say no buffer needed above 4.6) OK 1) pH drifts too fast 2) need to adjust pH frequently (e.g. daily) 3) adjustment brings greater

Proposed change	Reason	Comments rec’d	FEEDBACK
		occurs and therefore more Fe goes into solution), good pH control can be achieved by employing a suitable volume to surface area ratio appropriate for the test period. (2) Seeing evidence (e.g. from comparison tests) to show that equivalent results can be obtained from the EFC 16 test solution and the NACE Test Solution (which has a much higher concentration of acetates, presumably the reason it is thought the pH is more stable) PS. Has consideration been given to using CO2/bicarbonate buffer systems for higher pH values and acetate/strong acid buffer systems for the lower pH values, in line with Jean-Louis Crolet’s Paper on pH buffers at Eurocorr a few years back. SEE NOTES BELOW TOO	risk of O2 ingress 4) stated to be an issue even with very large SA/Vol ratios THE VARIATION IN PH IS A RECOGNISED SOURCE OF TEST RESULT VARIATION. I don’t have comparative evidence, maybe others do. What is clear to me is that large numbers of test spec refer to a mixture of NACE B soln + modified pH, so we are aligning the text here with the reality of what operators and manufacturers have been widely requesting for several years i.e. the convenience of the stable test solution, with the buffer and procedure to hold it at one pH , not let it drift..
MARTIN - Paragraph A.3 For the condensing water condition why is a 50 g/l NaCl solution used? This would not occur in service.		MARTIN - Change to 1 g/l NaCl (also consistent with EFC 17)	Interesting idea. I THOUGHT COULD BE NICE TO LINE UP WITH EFC17 BUT IT WON’T WORK, E.G. EFC17 A4.4 has no buffer in it (cos CRAs not corroding substantially). Maybe we need to spilt up several levels of environmental test solutions comparable to EFC17?? OR is the

Proposed change	Reason	Comments rec'd	FEEDBACK
			issue of chloride concentration less critical in CS case where less impact on the initiation event?? Are we over-complicating this whole issue?
Annex A A.3		Turnbull - Annex A and test solutions that the EFC requirement for a pH change no greater than 0.1 is not matched by the NACE standard, which allows a limited upward drift. It is difficult to do pH adjustment during a test without an incursion of oxygen and it may be better to tolerate a modest pH increase. In any case the near-surface pH will be possibly elevated as there is no stirring.	THIS IS PART OF THE KEY PROBLEM – PREFER TO ADJUST TO MORE STABLE SOLUTION THAN WIDEN THE PH DRIFT RANGE.
Table A.9 Heading Needs correcting to read: "Acceptance criteria for SSC test methods"	This is an original typing mistake.		OK
Table A.9 Right hand column Add the subscript "^{3}" to "Actual YS" in first 3 rows to be consistent with following 3 rows.	This is an original typing mistake.		OK
APPENDIX 2 Add Section 2.5 Failure Appraisal 4PB specimens should be evaluated for any evidence of cracking including:	Appendix 2 did not have any guidance on failure appraisal, unlike Appendix 1. The proposed	Turnbull - for 4 pt bend specimen (appendix 2) we section at 1/3 and 2/3 positions - not simply mid-width - this is a more conservative approach and we would recommend it. OLSEN -	Adjusted wording of sectioning and examination to accommodate majority of the suggestions. Agree with Mike Billingham about avoiding confusion of HIC

Proposed change	Reason	Comments rec’d	FEEDBACK
i) Surface breaking SSC ii) Sub-surface / surface breaking SOHIC (SZC) iii) HIC / hydrogen cracking. The following evaluation method should be used: i) Initial visual examination @ 10x mag. ii) MPI of stressed test face. iii) Sectioning of the specimens at mid width, with metallographic examination at 100x mag of both cut faces. All cracks identified should be reported, clearly identifying the type of crack and location.	procedure is basically similar to that detailed in section B.4.2.3 of ISO 15156 / NACE MR0175 for the evaluation of 4PB specimens for SOHIC/SZC.	iii) Sectioning of the specimens at indications from MPI or mid width if no indication from MPI. iv) Metallographic examination at typically 100x mag of both cut faces. Any evidence of stress corrosion cracks or SOHIC initiated from the stressed face will be considered as failure when deeper than 500 µm. More shallow features should only be considered as a failure if appearance is very typical for SSC or SOHIC. BOND - iii) Sectioning of the specimens at mid width, with metallographic examination in the unetched condition at 100x magnifcation of both cut faces. The location of any cracks should be confirmed in the etched condition.. BILLINGHAM - *“iii) Sectioning of the specimens at any suspicious features noted in steps (i) and (ii), or otherwise at mid-width… etc”* For practical purposes, perhaps a cut-off limit could be applied to crack size (eg 0.1mm) eg : *“Any stress corrosion cracks or SOHIC extending more that 0.1 mm in the through thickness direction should be considered as cause for failure. ”* HIC: Sections made for SSCC inspection may be different dimensions & orientation from the std HIC test sections. Hence if HIC is found,	evaluation on non-standard HIC specimens, so removed reference to HIC. Made Wade corrections.

Proposed change	Reason	Comments rec'd	FEEDBACK
Any evidence of stress corrosion cracks or SOHIC initiated from the stressed face will be considered as failure. If HIC cracks are detected they should be evaluated as in Annex B.		evaluation of crack ratios CTR CLR CSR is not necessarily unambiguous or equivalent to that in HIC test (what should one use for the width W and thickness T in the formulae ?) My experience is that it is usual to do separate HIC tests and SSCC tests in parallel and to record any HIC type cracking in the 4 pb test for information only rather than a pass / fail evaluation. Occasionally, we have done combined HIC & SSCC tests, but these are one-offs and I think it best to leave people to write their own procedures for these specific cases. As you probably don't want to go into great detail of HIC procedures here, I would suggest omitting the final sentence. There is still the statement that *"all cracks… should be reported…".* WADE - SUGGEST new text be Section 2.4 and existing 2.4 be renamed as "2.5 Reporting – requirements for welded specimens" AND 3rd word below be changed to "welded". CHANGE: "...SOHIC (SZC)" to "SOHIC and SZC" to exclude implied equivalence. Why not: "....examination @ at 10x magnification." ?? AGAIN "magnification" (Note "at" this time). BOSCH - In an SSC test SSC is tested, not HIC, this is tested separately under HIC test conditions. HIC should not be evaluated in an SSC test, in	

Proposed change	Reason	Comments rec'd	FEEDBACK
		particular if thin (i.e. machined, not full wall specimens) are tested. Sectioning is only needed for sub-surface SOHIC evaluation. If SOHIC evaluation is requested, it should be addressed separately. As stated in 2.2 (Method), SOHIC evaluation should require 15 mm min. wall thickness of full wall thickness for a wall thickness < 15 mm. BOSCH - PROPOSED ALTERNATIVE WORDING: 4PB specimens should be evaluated for any evidence of surface breaking SSC cracking. The following evaluation methods should be used: i) Initial visual examination @ 10x mag. ii) MPI of stressed test face. Additional evaluation to sub-surface / surface breaking SOHIC (SZC) may be carried out by the following evaluation method: iii) Sectioning of the specimens at mid width, with metallographic examination at 100x mag of both cut faces. All cracks identified should be reported, clearly identifying the type of crack (SSC or SOHIC, if applicable) and location. Any evidence of stress corrosion cracks or, if applicable, SOHIC initiated from the stressed face	Bosch – clearly most labs and operators are demanding sectioning of samples for SSC as well as SOHIC. Wall thickness issue may be valid in a specific test, if it is not then issue of finding of SOHIC may be challenged, but majority will aim to investigate both in one go.

Proposed change	Reason	Comments rec'd	FEEDBACK
		should be considered as failure. MARTIN - We normally recommend two sections through the width in the test section, at 1/3 and 2/3 through the width to get more information and since our experience is that cracks tend to start at the corners rather than in the middle of the sample. Suggest changing (iii) to:- Sectioning of the specimens at 1/3 at 2/3 width positions, with metallographic examination at 100x mag of all cut faces. Also ask that any corrosion (especially localised corrosion/pitting) is reported. Also change:- Any evidence of stress corrosion cracks or SOHIC initiated from the stressed face will be considered as failure. To:- Any evidence of surface breaking cracks (e.g. SSC) or SOHIC shall be considered as a failure. (Why limit this to the stressed face? Evidence of cracking from the 'back face' is an indication of a testing problem if nothing else and needs to be investigated/corrected).	See proposed revision to focus cutting on indication locations as per Billingham suggestion. OK OK
APPENDIX 3 Swap over the current Section 3.6. Test Duration and Section 3.5. Stress Intensity Factor. Then Rename that	Intended to make all the Appendices cover the topic "Failure Appraisal" for consistency	Turnbull - The section on DCB (Appendix 3) needs updating. Whilst the definition of KISCC is generically reasonable it would be inappropriate to suggest that the DCB test over 14 days necessarily gives that value. I proposed a	Not rec'd any further info from Dave Sponseller. If his ongoing work results in updates to TM0177, that is referenced from EFC16 so no incompatibility.

Proposed change	Reason	Comments rec'd	FEEDBACK
section to "Section 3.6. Failure Appraisal", adding the last sentence, "The K1SSC value shall exceed the acceptance criteria defined for the design or based upon the general guideline in Table A.9"		definition for KISCC in the context of DCB testing that Dave Sponseller included in his draft document. The emphasis was on using the nominal KISCC from DCB as a way of ranking steels without suggesting necessarily that a particular steel will not fail in service if the K value is below the DCB determined value. BOND - For: "The K1SSC value shall exceed the acceptance criteria defined for the design or based upon the general guideline in Table A.9" should we add reference to the concern that K_{1SSC} may result in a smaller allowable flaw size than workmanship criteria? KERMANI - new section will be 3.5 however, clarify "The K1SSC value shall exceed the acceptance criteria defined for the design (BY WHO?) or based upon the general guideline in Table A.9" BOSCH - The KISSC value strongly depends on the steel grade tested and on the wedge thickness used. Unless no clear limits for KISSC dependent on steel grade and wedge thickness are given, the reference to Table A.9 might be misleading. Common acceptance criterion for C110 is 27 MPa m^(1/2), whereas for C90 and T95 it is 33 MPa m^(1/2). My concern with the reference to Table A.9 in the text is that acceptance criteria might be generated which could not be fulfilled by modern standard steels such as C110. Could it be useful to add a footnote to Table A9	Turnbull comment suggests that a change to paragraph 3.1 is applicable (and maybe longer test time??). Bond comment useful to add to the new 3.6.

Proposed change	Reason	Comments rec'd	FEEDBACK
		indicating applicability of 35 MPa m^(1/2) for steels up to 95 ksi SMYS and possibly to add a second value (27 MPa m^(1/2)) for higher strength steels up to 110 ksi SMYS?	OK?? ANY OBJECTIONS??
ANNEX B Section B.8 Acceptance Criteria Change CTR limit from 3% to 5% Change CSR limit from 1.5% to 2%	To bring EFC 16 text in line with ISO 15156	MARTIN - OK but is there any technical basis for this change? Presumably the 3 and 1.5 values came from somewhere?	My recollection is that the criteria for HIC acceptance are all fairly arbitrary. EFC picked the tighter values after discussion with all the steelmakers in the group at the time, but ISO chose different values. EFC is now coming in line for consistency.
ANNEX C Add sentence at end of first paragraph: "Note that actual pH values in highly concentrated chloride environments may be lower than the estimates indicated in these figures. It is recommended to simulate the field condition as closely as possible, based on the water sample analysis and partial pressures of acid gases, rather than use pH values derived from these figures or theoretical models".	There has been some discussion on this and the opinion was that the graphs had been established for low chloride environments. I am not certain that is true (since the captions do refer to "formation water"), and anyway, they really are very approximate guides (which the text already says). I suggest only to add the further sentences	CROLET - "Note that actual pH values in highly concentrated chloride environments (> 0.5 M NaCl) may be lower than the estimates indicated in these figures. It is recommended to simulate the field condition as closely as possible, based on the water sample analysis and partial pressures of acid gases, rather than use pH values derived from these figures, or overly simplified theoretical models, or check that your pH calculations explicitly include the dependence of the acetic acid pK_a upon chloride concentration ". WADE - My understanding is that these figures were intended to be conservatively 'low' by 0.25 – 0.5 of a pH unit. A caution about extreme salinities may be appropriate but they are probably OK for 'normal' formation waters. Application to formation waters is implied by the inclusion of Ca & bicarbonate – otherwise where	Add Crolet - (> 0.5 M NaCl) Add Wade corrections. Since text is discussing the graphs there is no need to enter into the theoretical models issues as Wade suggests.

Proposed change	Reason	Comments rec'd	FEEDBACK
	indicated as further warning, unless anyone has firmer guidance to suggest.	do these come from? Any uncertainty should be resolved by reference to J-L C. Not withstanding the above, the proposal would be more grammatical as: It is recommended that ~~to simulate~~ the field conditions be simulated as closely as possible, based on the water ~~sample~~ analysis and acid-gas partial pressures ~~of acid gases~~, rather than by use pH values derived from these figures or theoretical models. ALSO SUGGEST last three words be deleted as opening a wider issue not related to the figures. KERMANI - These figures are very good approximate and in the absence of pH determination models serve a very good purpose. They already include the approximation and addition of a sentence unnecessary. As you know pH is affected by three main buffer systems (CO_2/HCO_3^-, H_2S/HS^- and HAc/Ac^- or other organic acids) – these are only catered by a very few pH calculation models – if you change the graph for one parameter, others may also affect the outcome. In any event higher Cl content does not necessarily affect the pH significantly - as you know higher Cl content affects CO_2 solubility. MARTIN - Assume the intention is to test under these simulated field conditions rather than just measure the pH under these conditions and then use the pH in the tests? Would need to change to '….It is recommended to simulate the field conditions **in the SSC tests** as closely as possible…'	Point taken - Better to just simplify to basic clarification that these graphs are approximate guides and avoid this potential confusion.

Proposed change	Reason	Comments rec'd	FEEDBACK
		We would not agree to this change at present and until we understand the issues better (NB. not sure I would necessarily agree with the Recommendation, which would tend to push people towards autoclave testing and hence '4PB tests' rather than 'constant load tests' at ambient pressure say which have bee used 'traditionally' to generate domain diagrams for carbon and low alloy steels). Can you provide more details? I believe the original source of the information in Annex C was Jean-Louis Crolet and would value his opinion on this aspect of chloride.	
DESAMAIS - Appendix 1 section1.4		**DESAMAIS - Change Ra<0.2mm to 0.2µm; I guess it is an original typing mistake**	OK

WADE –
Table A.9, p32: the entry in column 1, last row has a typo/formatting error that should be corrected. – DONE

Proposed change	Reason	Comment

Page 1, Definitions, CHANGE definition of CSR to “Crack **sensitivity** ratio”. Also change elsewhere as necessary, eg twice on p 43 – but I do not have an ecopy to search.	As defined in TM0284-2003 (Section 7, p8)	ISO 15156-2 also appears ‘wrong’ unless there is a reason for ‘Crack surface ratio’. I recall that no-one liked the term “sensitivity” because it implies that CSR is most sensitive measure of HIC (which it is not, its probably the most arbitrary). I will highlight to Stein but suggest leave unchanged.
p19 5th para: “....even trace elements of H_2S.....” REPLACE “elements” with “amounts” ALTERNATIVELY: “....even traces ~~elements~~ of H_2S.....”	typo or similar	DONE
p24, 1st line below table: “(@ 22 HRC)” DELETE spurious “@” character	typo carried over from 1st ed.	Occurred twice in 1st ed, now once. DONE
p30, Section A.3. Test Solutions 2nd word – “basis” CHANGE to “base”. Is repeated elsewhere, eg in subs-paras A & B on same page. 4th line – “HCl” – final character is ‘#1’ - CHANGE	“basis” is a noun so use as an adjective is ungrammatical. Typo or similar	DONE
p32, Table A.9 “σ_{th}” is not defined (as far I can see). ADD definition and x-reference to Section A.9.	To ensure no misunderstanding.	ADDED REF TO SECTION 1
p47, RESTORE legend defining meaning of dashed/continuous/dotted lines in Figs C3 – C5. This was in 1st ed but lost in 2nd ed. ALSO add units to criteria (that were missing from 1st ed). These are: meq L^{-1}.	Correct error of omission in 2nd ed.	DONE

p55, Reference 3 - ??update to 2nd ed??	Update	
Reference #6 REMOVE numerical character from 3rd name, REPLACE as "Truchon".	Typo	
Reference 14: ??Condensate Lme??? – presumably "Line" – REPLACE. Also ADD spaces between numbers on last line.	Typos	
More generally, the references are inconsistently formatted, eg: #7 & #14: "perform", "Perform". #9: no punctuation after title, elsewhere varies between comas & periods. The whole page needs careful proof reading!	Typos	

BOND - Are all the acronyms already defined previously in the text inc MPI?? – CHECK - DONE

THOMPSON - wonder if we should address the issue of averaging of HIC test results when applying the acceptance criteria for CTR, CLR, and CSR. Last time I spoke with Chris Fowler about this, I think he said that averaging of the 3 examined faces per test block was not unusual and should be permitted, but this is not addressed in any of the existing standards. Sytze asked the 15156 MP about this last year as I recall, and its come up for us a few times before. DONE – THIS IS COMMON PRACTISE

FOWLER - Industry standard is to average the three cut faces from one coupon.

SWIDZINSKI - We have experienced some unexpected failures of welded specimens tested Per EFC 16 Appendix 2 (4-point Bend).

A detailed investigation revealed that the failures were NOT due to any metallurgical effects. However, what was found was that the samples had variable cross section (height) resulting in them being trapezoidal in cross section. The current EFC narrative says that the samples should be "generally of uniform thickness". I CANNOT FIND THIS TEXT ANYWHEREbut added in other section). I believe that we need to tighten up on this.

Our investigations were quite conclusive in that samples with faces > or equal to 2 degrees all failed whereas those with near parallel faces passed.

NACE TM 01 77 is invoked by ISO 15153. Method B (Bent Beam) Section 9.2.1 stipulates that the height and width of the specimen should not vary by more than 5 thou (0.18 mm). I would strongly urge adoption of similar tighter tolerances for the 4-point bend specimens described in EFC 16. [CONVERSION OF 0.12MM = 4.7 THOU SO SUGGEST TO TAKE THAT TOLERANCE]

FOLLOWING SECTION IS MORE DISCUSSION OF THE A.3 SECTION ON TEST SOLUTIONS

STEIN OLSEN comments to A.3 Test solutions.

We are writing that the basis solution of 50 g/L NaCl simulates the majority of produced waters. Well, my experience is that the chloride content is much higher for formation waters and much lower for condensed water. So, the statement is not correct, however, it has no significance for testing of C-steel, and this is also copied into table B.1 in ISO 15156-2 Annex B. To prevent any confusion, this should probably not be changed.

You are proposing an option to use NACE Solution B, but adjust it to pH 3.5/4.5 (or may be actual calculated pH). The reason is that we need a stronger buffer to stabilize the pH. If you look into NACE TM0177-2005, the pH of test solution should be between 3.4 and 3.6 with 1 bar H2S, and there is not offered any option to change this pH. So if we are proposing a Solution B with adjustable pH and a CO2/H2S mixture, this is something new and not well defined. How should the pH be adjusted, by changing the concentration of Acetic acid or by adding HCl/NaOH? (OTHER LABS SUGGEST HCl/NaOH IS OK)

Personally, I have limited experience in testing of C-steel, but we have undertaken testing with regular pH-measurements and regeneration of buffering power, which complicates testing and is a potential source for oxygen contamination. A stronger buffer could help, but we have to be sure that what we are proposing is correct.

Jean-Louis presented something on this some years ago in Budapest. (See minutes from EFC meeting below). He commented that the buffering capacity of the “EFC solution” depends strongly on the actual pH, and that above pH 4.6, the buffering strength is low. I am not sure how correct that is, but if so, a NACE solution B adjusted to a high pH will probably also have limited buffering capacity.

The whole idea with the “EFC approach” is to simulate more accurately the actual conditions. But by adding more organic acids to the solution, the conditions will probably be more corrosive and very different from what we have in real systems. Jean-Louis is suggesting use of NaHCO3 for higher pH-values. I do not think we should go for that.

My conclusion is therefore that we should just use the wording as it is, and not offer any other buffering system before we really understand the strength and the effect of the buffer on corrosion. SEE ADDITONAL INFO FROM J-L BELOW.

MINUTES FROM EFC WP13 MEETING IN BUDAPEST.

Consequence for testing of C-steel - EFC 16

JL Crolet:

- NaAc buffer has limited capacity for pH> 4.6 OK
- Avoid the need for pH measurements with the solution saturated with the test gas !
- For high pH (pH> 4.6) there is a limited need for buffer as iron sulphides limits the corrosion rate !
- Recommendation:

Use the 4 g/l NaAc buffer for pH < 4.6 BUT THERE IS A LOT OF EVIDENCE (NPL, CAPCIS, BODYCOTE,CSM) THAT THIS IS INSUFFICIENTLY STRONG TO BUFFER THE SOLUTION ON ITS OWN.

Use NaHCO3 for pH>4.6 and calculate the required concentration (natural buffer) SINCE THIS SECTION OF TEXT IS CONCERNED WITH PH3.5 AND 4.5 DON'T NEED TO ADDRESS IN THIS PUBLICATION.

Proposal to WP for EFC 16:

- Normally testing undertaken at ambient temperature
- If you follow EFC as it is today, including pH adjustment with the test gas, it is basically OK

Discussion:

- Don't we need a strong buffer at high pH ? Probably not due to protective sulphide scales.
- Should we avoid the need for pH adjustment with the solution saturated with the test gas ? For checking and adjustment during the test period, pH must be checked with the test gas anyway for high pH !!!!

Recommendation:

- No immediate need to change the EFC 16!

- JL Crolet’s proposal is better than the existing procedure!
- Must be discussed during the next meeting

DENT - Chris and myself have looked at the comments from Stein and we recognize the problem of just changing the solution. However the problem is that the solution A at a pH of 3.5 is not stable in the range of 3.4 – 3.6 when testing carbon steels due to the amount of corrosion. We do not see the same problem with the higher pH 4.5 levels.

One option when using solution A for carbon steels would be to relax the pH restriction and set the initial pH to 3.4 – 3.5 (as is the case now), but to allow the pH to increase up to a maximum of 4.0. Therefore the solution would remain unchanged and the pH would then be consistent with the requirements in NACE TM0177 and NACE MR0175/ISO 15156. If the pH was required to be maintained within a range of +/-0.1 for the duration of the test this would need to be specified and the method of maintaining the pH agreed between the laboratory and the client.

We agree with Steins general views regarding the metallographic examination, HOWEVER we feel that setting an acceptance limit on the depth of any indications (i.e 0.5mm) following metallographic examination,
is easy to interpret although it could lead to clear SSC being accepted. Chris will be giving you a call to discuss this point.

BILLINGHAM,

Comments on Proposed Revision & Stein Olsen Comments.

Section A.3. Test Solutions.

The acetic acid / acetate buffering system is most effective around pH 4.8 where there are similar concentrations of acid and salt species (pKa for acetic acid is ~ 4.76). At ph 3.5 the buffering capacity is less than at pH 4.5.

Our experience with carbon steel samples is that EFC “A” in particular typically needs some adjustment during a 30 day test to maintain pH 3.5, whereas EFC B at ph 4.5 is more stable. The ph control practice & frequency is a possible source of test variation. NACE B type solution is more pH stable than the EFC type solutions as one would expect from the higher levels of buffering species, but acetic acid / acetate is inherently not an ideal buffer system for ph 3.5. At higher ph, the NACE B acetate buffer seems to be stable up to at least ph 5.2 in our experience [ie contrary to the comment quoted by Stein ?] . [LIANE _ J-L is saying not to use above 4.6 and not reqd’ but we only cover up to 4.5 so no prob.

Both EFC and NACE B solutions are commonly specified for testing flexible pipe wires for example, and also for SSCC tests on conventional rigid pipe. Often the solutions are adjusted to other pH ranges for specific applications. We generally use NaOH or HCl for adjustment rather than acetic acid. This keeps the total acetate content constant. I don ‘t think clients & end users in general have a strong preference for one or the other solution type, indeed I have seen specifications which are vaguely worded so as to allow either EFC or NACE type solutions (along the lines of “ 50 g/l NaCl adjusted to pH xxx”) .

There is a more general issue that the corrosion rates of carbon steel in some conditions are known to be affected by the total amount of acetate species (beyond the affect of pH): eg SPE papers 100412 & 100669, SPE Corrosion Control 2006 . This could potentially affect hydrogen generation and hence severity of the SSCC test. Corrosion rate data appears to be limited to production water type conditions with much lower amounts of acetate than in these artificial test solutions. My feeling is that a really large difference between NACE B and EFC solutions at the same pH as regards SSCC test severity would have been noticed by now, but there may well be some effect. More consideration needed ?

Summary

- Agree that NACE B is experimentally more convenient as regards pH control than EFC type solutions, in particular EFC ”A” at pH 3.5. therefore (other factors being equal), we would prefer to use NACE B in practice.
- Both NACE B and EFC type solutions are being used commonly, often adjusted to other ph values
- Practices with regard to initial ph adjustment and ph control do vary; my preference is for using HCl / NaOH to adjust NACE B solution (as specified in EFC 16 at present) in case the concentration of acetate is a significant factor.
- Nevertheless, perhaps the effect of acetate concentrations on corrosion should be considered more thorouoghly before making a change.

CROLET COMMENT –

Also most important, I do not see the recommendations I made in Budapest in 2003 both at WP 13 and ISO, and later developed in the NACE paper 04140 , namely :

1) Prohibition of any artificial buffering with an acetate buffer above pH 4.5, since it is first strictly useless (no pH drift in natural conditions, and nothing better with an acetate buffer outside its domain of efficiency). The main point, however, is that it becomes highly misleading if the bothering procedure is not strictly followed. (pH adjustments mandatorily under gas pressure, otherwise a negative pH shift will immediately occur when applying acid gases).

2) Below pH 4.5, possibility of a pH adjustment directly on the mother solutions, i.e. not yet under acid gas pressure., since the above mentioned pH shift is then negligible. Many people already did that "illegally", including me, but if this does not matter below 4.5. On the contrary, such "approximate" procedure can become extremely false above 4.5, whereas paradoxically, artificial buffering in itself is then useless.

In Budapest, the ISO committee swore that this would be incorporated into ISO 15156 as soon as re-edition will allow it, and it seems to me that EFC 16 cannot do less.

15 years ago actually, Liane and the WP 13 committee had refused this distinction of 2 pH domains only "for the sake of simplicity". Since then, however, I brought proves that this is absolutely mandatory. Like for CO2 corrosion and API vs Bonis' rules (NACE paper 05272), LIANE there is no overlap between the successive buffering domains, and when CO2/H2S are the leading acids, using HAc is not only useless, but also highly tricky when not supported by quantitative assessments, even though calculations are still less easy.

In passing, I am also surprised to see no reference to my key note paper is Nice LIANE CHECKED, whereas my CORMED 2 software is apparently the only one able to predict what will happen during such tests, or design tailor made variants for matching specific issues like minimal chloride.