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# Space Product **Assurance**

Determination of the susceptibility of metals to stress-corrosion cracking

> **ECSS Secretariat ESA--ESTEC Requirements & Standards Division Noordwijk, The Netherlands**



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### Foreword

This standard is one of the series of ECSS Standards intended to be applied together for the management, engineering and product assurance in space projects and applications. ECSS is a cooperative effort of the European Space Agency, National Space Agencies and European industry associations for the purpose of developing and maintaining common standards.

Requirements in this standard are defined in terms of what must be accomplished, rather than in terms of how to organise and perform the necessary work. This allows existing organisational structures and methods to be applied where they are effective, and for the structures and methods to evolve as necessary without rewriting the standards.

The formulation of this standard takes into account the existing ISO 9000 family of documents.

This standard has been prepared by editing ESA PSS-01-737, reviewed by the ECSS Technical Panel and approved by the ECSS Steering Board.





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### Scope

This document defines the preferred way to determine the susceptibility of metals and weldments to stress--corrosion cracking by alternate immersion in 3,5 % sodium chloride under constant load.

The results obtained from test programmes made according to this specification are used to classify alloys, weldments and their individual heat treatment conditions. When sufficient stress--corrosion data exists the alloy designations may be submitted for inclusion into the various tables contained in ECSS-Q-70-36.



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# Normative references

This ECSS Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these apply to this ECSS Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.







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### Definitions and abbreviations

#### 3.1 Definitions

For the purposes of this standard, the definitions given in ECSS-P-001 apply. The following terms and definitions are specific to this standard and shall be applied.

#### **Residual strength** (see 6.1 e)

Apparent UTS based upon nominal cross-sectional area

#### 3.2 Abbreviations

The following abbreviations are defined and used within this standard.





### $\boldsymbol{\varLambda}$

### General

This specification covers constant-load stress--corrosion testing of metal specimens taken from welded or unwelded material. It is primarily intended for testing aluminium and ferrous alloys. The tests are carried out under alternate immersion conditions in 3,5 % sodium chloride over a thirty-day exposure period.

Unstressed control specimens are exposed to the same environment to provide a basis for comparison in assessing stress--corrosion susceptibility of materials that survive thirty days in the stress--corrosion test. This susceptibility is assessed by tensile tests, to compare the residual strengths of the exposed specimens, stressed and unstressed, and by metallographic examination of microsections from stressed and unstressed exposed specimens, to distinguish between stress--corrosion and intergranular-corrosion or pitting occurring independent of stress.

The requirements for the apparatus used for carrying out the test are set out in subclause 5.3 - Conditions for test apparatus. Details of the preferred apparatus are given in annex B but may be varied provided that the requirements set out in subclause 5.3 are met.

Some wrought alloys commonly show greatest susceptibility to stress--corrosion when stressed in the short transverse direction. When such materials are to be subject to short transverse stressing in service, stress--corrosion tests shall be carried out under tensile stress applied in the short transverse direction. The test method covered by this specification is suitable for such tests provided that the dimensions of the material to be tested will provide short transverse specimens with a gauge length not less than 10 mm long. For other material short transverse stress--corrosion tests shall be carried out using C-ring specimens, bent-beam specimens or "tuning fork" specimens as described in ASTM G38-73(1995)e1, ASTM G39-90(1994)e1, or DIN 50908.



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### Preparatory conditions

#### 5.1 Types of samples

- a. Samples shall be tested in the form of tensile test specimens which may be either turned bars or special specimens machined from flat material as specified in subclause  $5.2$  - Details of test specimens.
- b. Test specimens shall be produced so that the direction of the test stress appropriately represents the direction of stress in service. When stress--corrosion tests are being carried out on wrought aluminium alloys for general classification purposes, the stress--corrosion test specimens shall, where possible, be cut so that the stress will be applied in the short transverse direction.
- c. For tests on welded material the welds shall be made according to the specification laid down for the equipment or component to which the stress--corrosion tests relate, and the test specimens shall be cut so that the weld is situated at the centre of the gauge length. In tests using flat specimens the surface of the sample is machined to remove the weld bead - giving a smooth surface for the full gauge length of the specimen.
- d. The specimens subjected to stress--corrosion tests shall be cut from material in the same condition (heat treatment, or room temperature ageing after solution treatment or welding) as the components that will be used in service.
- e. The material designation, actual composition, grain size and orientation and heat treatment condition shall appear on all documentation relating to the test specimens.

#### 5.2 Details of test specimens

Where the shape and size of the material to be tested permits, a turned specimen of the type shown in Figure 1 is preferred. The diameter of the parallel end pieces and the form of thread can be made to suit the shackles of the tensile test machine and the stress--corrosion apparatus but should not be less than 10 mm diameter. The gauge length of 50 mm shown in Figure 1 is suitable for unwelded material and for most weldments; for welded specimens it shall extend at least 10 mm beyond the heat affected zone on each side of the weld. The plain parallel portion of the test piece between the threaded end and the shoulder is provided to facilitate sealing the bottom of the surrounding cell to the test piece and to provide latitude in adjusting the solution level within the cell to come above the gauge length but below the top shackle.



When specimens are to be taken from material less than 12 mm thick, flat specimens of the type shown in Figure 2 shall be used. The specimen is machined to a thickness of 5 mm giving a cross section of 50 mm<sup>2</sup> in the gauge length. The gauge length requirements shall be as for the machined specimens shown in Figure 1. The dimensions of the end sections shown in Figure 2 are suitable for use with 10 mm diameter shackle pins in the tensile test machine and stress-corrosion jigs. They may be altered to suit the test apparatus available but it is essential that the width of the end section shall be at least 20 mm greater than the diameter of the shackle-pin hole; otherwise failures may occur across the centre line of the shackle-pin instead of in the gauge length.

The specimens shall be machined to a finish of 0,5 micron CLA or better, the edges of flat specimens being slightly radiused by careful abrasion with fine silicon carbide paper (1 200 grade).

**NOTE** Polishing papers that contain oxides of iron shall not be used for this purpose as they can accelerate pitting during exposure of the specimen to the saline solution.

When short transverse specimens are required, the dimensions shown in Figure 1 and Figure 2 will usually have to be reduced. This is permissible provided that the gauge length of the modified specimen is not less than 10 mm. The dimensions shall also provide a minimum length of 10 mm between the gauge length and threaded portions of turned specimens and a minimum of 10 mm all round the shackle-pin holes of flat specimens.

#### 5.3 Conditions for test apparatus

#### 5.3.1 Preferred test apparatus

Details of the preferred design of test apparatus are given in annex B. This utilizes the compression of a precalibrated spring to apply the desired axial tensile stress to the specimen under test. The specimen is surrounded by an open-topped cell which is flooded with 3,5 % sodium chloride solution for ten minutes in each hour. Failure of the specimen is automatically recorded by the operation of a microswitch beneath the lower specimen support plate.



#### Notes:

- 1. Remove all sharp edges
- 2. The specimen should be fully symetric
- 3. Sample identification by 1 to 9 Letter height is  $1,5$  and location is marked  $\Theta$
- 



#### Figure 1: Preferred turned stress-corrosion test specimen





#### Notes:

- 1. Remove all sharp edges
- 2. The specimen should be fully symetric
- 3. Sample identification by 1 to 9 Number height is 3,5 and location is marked
- 4. t; Thickness typically 5 mm. However could vary according to product thickness and testing device loading capability
- 5. Dimensions of drawing in mm and not to scale



### **Figure 2: Preferred flat stress--corrosion test specimen**

#### 5.3.2 Alternative test apparatus

Alternative designs of apparatus may be employed provided that the following conditions shall be satisfied:

- a. The characteristics of the spring shall be such that slight yielding or creep of the specimen during the thirty-day test will not significantly alter the load applied by the spring. This is achieved in the preferred test apparatus by employing springs requiring a compression of about 50 mm to produce a typical desired load. Other springs may be used which require a compression of between 30 and 70 mm to produce the desired load.
- b. The load shall be applied to the specimen through shackles which provide automatic alignment so that the applied tensile stress is axial with respect to the specimen.
- c. Provision shall be made for automatic recording of the time of failure of the stress--corrosion specimen.
- d. The dimensions of the cells surrounding the stress--corrosion specimens and the unstressed control specimens shall be such that the surface of the sodium chloride solution is above the top of the gauge length of the specimen, but below the top shackle, when the cell is flooded and the gauge length shall dry out during the fifty-minute interval between successive ten-minute periods.
- e. Suitable arrangements shall be made to seal the bottom of the cell to the specimen below the gauge length or to the lower shackle and to protect the shackle against contact with the solution.
- f. A separate reservoir containing 1 litre of the 3,5 % sodium chloride solution shall be provided for each pair of specimens (one stress--corrosion specimen and one unstressed control).
- g. The arrangement for alternately flooding and draining the cells surrounding the specimens may be a mechanical device which automatically raises and lowers the solution reservoir or a pneumatic device as in the preferred apparatus.





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### Test method

#### 6.1 Procedure

- a. Nine flat or turned tensile test specimens as detailed in subclause  $5.2$  "Details of test specimens" - are required for the test. Three are used to determine the initial tensile properties of the material. Three are loaded in tension to the required stress (see subclause  $7.1$  - "Test stress") in a spring loaded test jig which provides an axial constant load. Detailed requirements for the test jigs are given in subclause  $5.3$  - "Conditions for test apparatus". Each stress-corrosion specimen is surrounded by a cell which is flooded with 3,5 % sodium chloride for ten minutes in each hour. A timing device is connected to a microswitch operated by the displacement of the jig which occurs when the specimen breaks.
- b. The third set of three specimens is used for unstressed controls. These specimens are supported vertically in individual cells of the same type as those which surround the stress--corrosion specimens, the cells being flooded with the same solution and at the same times as the stress--corrosion specimen cells.
- c. The  $3.5\%$  sodium chloride test solution is made to ASTM G44-94 "Standard" Practice for Evaluating Stress Corrosion Cracking Resistance of Metals and Alloys by Alternate Immersion in 3.5% Sodium Chloride Solutionî. One litre of solution is provided for each pair of specimens and is replaced after seven, fourteen and twenty-one days.
- d. When any of the three stress--corrosion specimens fails it is removed from the test rig, washed with warm water and dried in a stream of warm air. The corresponding unstressed control specimen is taken out at the same time and similarly washed and dried. Both are then stored in a desiccator until all three stress--corrosion specimens have failed or the thirty-day test period has been completed.
- e. When the tests have been completed the unstressed control specimens corresponding to any stress--corrosion specimens that failed during the test should be tensile tested, according to subclause 6.1 g., h. and i., to determine their residual strength. Comparison of the residual strength of the unstressed control with that of the specimens tested initially and with the stress applied in the stress--corrosion test will indicate to what extent the failure of the stress- corrosion specimen was due to stress--corrosion cracking and to what extent it was due to other forms of corrosion occurring independently of applied stress.



- f. If none of the stress--corrosion specimens fails during the thirty-day test period two of them, and their corresponding unstressed controls, are tensile tested and submitted for metallographic examination. If any stress--corrosion cracking has occurred the residual strength of the stress--corrosion specimens will be less than that of the corresponding unstressed controls. The third stress--corrosion specimen and its unstressed control are used for metallographic examination without tensile testing. (see annex A case A).
- g. If one of the three stress--corrosion specimens fails during the thirty-day test, tensile tests are carried out at the end of the test period on the corresponding unstressed control, on one of the two unfailed stressed specimens and on its unstressed control. The third stress--corrosion specimen and its unstressed control are used for metallographic examination without tensile testing (see annex A case B).
- h. If two of the three stress--corrosion specimens fail during the thirty-day test the remaining unfailed stressed specimen and its unstressed control are used for metallographic examination without tensile testing. Only the unstressed control specimens for the stress--corrosion specimens that failed are tensile tested (see annex A case C).
- i. If all three of the stress--corrosion specimens fail during the thirty-day test they are submitted to metallographic examination. The first two unstressed control specimens to be withdrawn are tensile tested and then used for metallographic examination. The last unstressed control specimen to be withdrawn is not to be tensile tested, but will be metallographically examined (see annex A case D).
- j. Axial microsections across the full thickness are prepared from all the stress- corrosion and unstressed control specimens. From those which failed during the test, or were broken in subsequent tensile testing, the microsection shall extend from the fracture surface for a distance of at least 20 mm. For welded specimens the total length of the microsection shall extend at least 5 mm into the parent metal beyond the heat affected zone. Two adjacent microsections may be prepared if necessary to cover this length. For unbroken specimens, the longitudinal microsection shall extend from one side of the centre of the gauge length for a distance of at least 10 mm or (for welded specimens) to a point 5 mm beyond the heat affected zone. The microsections are mounted and polished for metallographic examination as specified in subclause 7.2.
- The operations to be carried out during and at the end of the thirty-day test period shall be set out in tabular form in annex A for examples in which different numbers of stress--corrosion specimens fail during the test period.
- l. The criteria for assessment of stress--corrosion susceptibility from tests carried out according to this specification shall be set out in subclause 7.3.

## 7 Acceptance criteria

#### 7.1 Test stress

- For unwelded material the stress-corrosion test load is calculated from the average 0,2 % proof stress value provided by the three specimens tensile tested initially. To enable classification of materials according to subclause 7.3 tests shall be carried out at 75 % of the 0,2 % proof stress. Materials that have been characterised to possess a medium or low resistance to stress-corrosion cracking may be further tested to establish their stress--corrosion threshold. When these materials are loaded in the short transverse direction in service under conditions where the load can be controlled, tests at 50 % of the proof stress or even lower values may be called for.
- b. For welded specimens the stress--corrosion test load should not exceed 75 % of the proof stress of the welded test bar but, since yielding in welded test bars occurs usually in the weld bead, the proof stress for such specimens depends upon the ratio of weld bead width to gauge length. The selection of the test stress should, therefore, be related to the nominal proof stress of the parent metal and to the design stress - making due allowance for possible assembly stresses. It is best expressed in absolute units rather than as a percentage.

#### 7.2 Metallographic examination

- a. The longitudinal microsections, taken as described in subclause 6.1 j., shall be mounted, ground and polished by normal metallographic procedures and shall be examined initially at a magnification of X50 for evidence of stress- corrosion cracking. When they exist, the maximum depth of pits shall be recorded. Stress--corrosion cracks can initiate from these pits.
- b. In aluminium alloys stress--corrosion cracks generally follow intergranular paths. Any apparent cracks shall be examined in greater detail at X500 magnification to establish whether they are true stress--corrosion cracks running through virtually uncorroded metal or tensile cracks running through areas of intergranular corrosion.
- c. The characterisation of a crack as due to stress--corrosion is facilitated by comparison of microsections taken from the specimens tested, stressed and unstressed. Any cracking found in the unstressed control specimens examined after tensile testing is probably not due to stress--corrosion, and similar cracking observed in the corresponding stress--corrosion specimens cannot be considered to be due solely to the stress-corrosion test.



#### 7.3 Assessment of stress-corrosion susceptibility

The materials tested shall be classified as showing resistance to stress--corrosion; either:

- high:
- moderate; or
- low.

#### 7.3.1 Class 1

Alloys or weldments show *high* resistance to stress-corrosion if:

- a. none of the three stress--corrosion specimens fails in the thirty-day test. Any failure is disregarded if the tensile strength of the unstressed control specimen removed from test at the time of failure of the stress--corrosion specimen does not exceed the stress--corrosion test stress; and
- b. the average tensile strength of two of the three stress--corrosion specimens after the thirty-day test is not less than 90 % of that of the unstressed control specimens; and
- none of the three stress-corrosion specimens shows evidence of stress-corrosion on metallographic examination at X50 magnification.

#### 7.3.2 Class 2

Alloys or weldments show *moderate* resistance to stress--corrosion if:

- none of the three stress-corrosion specimens fails in the thirty-day test. Any failure is disregarded if the tensile strength of the unstressed control specimen removed from test at the time of failure of the stress--corrosion specimen does not exceed the stress--corrosion test stress; and
- b. the average tensile strength of the two stress--corrosion specimens after the thirty-day test is not less than 90 % of that of the unstressed control specimens; and
- c. metallographic examination at X50 magnification shows evidence of stress- corrosion in any of the three stress--corrosion specimens.

#### 7.3.3 Class 3

Alloys or weldments show *low* resistance to stress-corrosion if:

- any of the three stress-corrosion specimens fails in the thirty-day test at a test stress below the tensile strength of the unstressed control specimen removed from test when the stress--corrosion specimen fails; and
- b. the average tensile strength of the stress--corrosion specimens after the thirty-day test is less than 90 % of that of the unstressed control specimens; and
- c. metallographic examination at X50 magnification shows evidence of stress- corrosion.



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### Quality assurance

#### 8.1 General

The measurement and test equipment used shall be calibrated using standards traceable to national standards and used in an environment controlled to an extent to ensure valid measurements. Any nonconformance on equipment, material or test specimen shall be recorded and reported in the test report.

#### 8.2 Test report

On completion of the test a test report shall be prepared with the following data and information:

#### 8.2.1 Material

- $\bullet$  material specification, form, actual composition and condition (e.g. grain orientation, heat treatment);
- material identification code (e.g. manufacturers' batch number);
- for welded material, details of weld process, filler metal composition and post-weld heat treatment or natural ageing period are also required.

#### 8.2.2 Specimens

- type;
- $\bullet$  dimensions;
- grain size; and
- grain orientation.

#### 8.2.3 Test conditions

 $\bullet$  stress level.



#### 8.2.4 Results

- $\bullet$  Lives of stress-corrosion specimens;
- $\bullet$  0,2 % proof stress and tensile strength of all specimens subjected to tensile testing;
- $\bullet$  Metallographic observations for the:
	- stress-corrosion specimens; and
	- $\bullet$   $\;$  unstressed control specimens.

#### 8.2.5 Assessment

Classification of material tested as showing high, moderate or low resistance to stress-corrosion.



### Annex A (normative)

## Treatment of specimens during and after thirty-day test period

#### A.1 Case A - No stress-corrosion specimen fails



#### A.2 Case B - One stress-corrosion specimen fails





### A.3 Case C - Two stress-corrosion specimens fail



### A.4 Case D - All three stress-corrosion specimens fail





### Annex B (informative)

### Preferred design of test apparatus

- 1. Figure B-1 is an exploded sectional assembly drawing of the preferred apparatus for testing according to this specification. The tubular frame which forms the central member is a 350 mm length of 100 mm o.d. by 3 mm wall steel tube with a window 200 mm long by 125 mm wide machined in each side.
- 2. The bottom of the tubular frame is fitted with a bottom closure plate carrying a 125 mm length of 20 mm studding held by a securing nut at the bottom and screwing into a specimen outer shackle at the top. The opposite end of the outer shackle has a vertical slot 10 mm wide by 55 mm deep which engages with a tongue at the bottom end of the inner shackle. A 10 mm diameter steel pin holds the two shackles together and permits slight movement between them to maintain axial loading.
- 3. For use with threaded specimens of the type shown in Figure 1 the top of the inner shackle is drilled and tapped 12 mm. For use with the flat type specimen shown in Figure 2 the top of the inner shackle is slotted to receive the end of the specimen and a 10 mm diameter hole is drilled through to accept a shackle pin which passes through the hole in the end of the specimen.
- 4. The top end of the tubular frame is fitted with a top closure plate with a plain central hole in which a 500 mm length of 20 mm studding is a sliding fit. The length of studding carries a keyway which engages with a grub screw in the top closure plate to prevent the studding turning in relation to the plate during loading. The lower end of the length of studding screws into the outer member of a pair of shackles similar to those at the bottom of the jig.
- 5. The compression spring which provides the load to be applied to the specimen is located between the top closure plate and a spring end plate which is a sliding fit on the studding. Both plates have spigots to locate the spring and the ends of the spring are ground flat to seat squarely on the two plates.
- 6. The spring is compressed by means of a tensioning nut which operates against a thrust race. Between the thrust race and the spring end plate is a cross piece supporting two hook bolts the lower ends of which engage in the windows in the sides of the tubular frame. These are provided to limit the expansion of the spring when the specimen breaks.



- 7. The compression spring shall be selected to suit the load that is to be applied to the test specimen. For testing high and medium strength aluminium alloy specimens of the dimension shown in Figure 1 and Figure 2, springs with ten turns of 15 mm diameter steel having a total free length of 150 mm and an outside diameter of 105 mm have proved satisfactory. These springs produce a load of 20 kg for each 10 mm of compression and require a compression of about 50 mm to provide a typical desired load.
- 8. A tensile test machine is used to plot a calibration curve of load against compression for each spring. The calibration curves are used to calculate the compression required to apply the desired load to the test specimen. Centre punch marks are made on the top and bottom turns of the spring and the compression measured by locating the points of a pair of dividers in these marks.
- 9. The test specimen is surrounded by an alternate immersion cell details of which are given in step 14 to 16.
- 10. To use the stress--corrosion test jig the parts are assembled with the exception of the top and bottom inner shackle. The specimen is fitted to the shackles and the alternate immersion test cell fixed in place. If flat specimens are used the part of the lower shackle which comes within the cell is coated with a suitable sealant (**Warning**: avoid sealants which may produce corrosive chemical species during curing, e.g. acetic curing silicones). The specimen, with the inner shackles and alternate immersion cell in place, is fitted into the test rig and located by inserting the pins which secure the inner shackles to the outer shackles. The tensioning nut is screwed down to take up the slack and a check is made to see that all the parts of the jig are correctly positioned so that the specimen will be under axial load when the spring is compressed.
- 11. The specimen is loaded to the required stress by compressing the spring to the appropriate length measured between the centre punch marks upon it. This may be done by holding the top closure plate by means of a peg spanner fitted into holes drilled in the plate for that purpose and screwing down the tensioning nut against the thrust race. It is more convenient however to use a simple hydraulic jack type of loading device acting between the bottom closure plate and the spring end plate to compress the spring to the desired extent and then screw down the tensioning nut to retain the spring in that position when the loading device is removed. After loading the specimen the nuts on the restraining hook bolts are screwed down finger tight.
- 12. After loading, the jig is suspended vertically by passing the upper part of the 500 mm length of 20 mm studding through a hole in a suitable frame as shown in Figure B-2 and fitting a nut above the suspension plate of the frame. The frame incorporates a tray which is situated about 10 mm below the bottom closure plate of the stress--corrosion jig and has a central hole through which the lower securing nut of the jig passes freely. A microswitch is attached to the tray so that this movement will alter the switch condition when the specimen breaks and the bottom closure plate drops. The microswitch is connected to an electrical timer which is started at the commencement of the thirty-day test period and stops automatically when the specimen breaks, thus recording its life.
- 13. The stress-corrosion tests are carried out in triplicate three similar unstressed control specimens being tested at the same time. The unstressed controls are supported vertically in spare inner shackles similar to those used in the stress--corrosion jigs and are surrounded by similar alternate immersion cells, but their top ends are free.
- 14. The alternate immersion cell comprises a transparent rigid plastic tube 60 mm i.d. fitted into a moulded PVC lower end cap which forms a seal onto the lower, plain end-portion of the specimen if threaded-end specimens are used. If flat specimens are used it is difficult to get a satisfactory seal on the specimen itself; the seal is then made on the inner shackle. The parts of the shackle within the PVC end cap and the gap between the end of the specimen and the slot in the shackle shall then be sealed with a suitable compound.
- 15. A 5 mm diameter tube moulded into the bottom of the end cap is connected to a flask containing 1 l of 3,5 % sodium chloride the pH of which is adjusted to between  $6.4$  and  $7.2$  as specified in ASTM G44-94 "Standard Practice for Evaluating Stress Corrosion Cracking Resistance of Metals and Alloys by Alternate Immersion in 3.5% Sodium Chloride Solutionî. Each flask serves two specimens, a stress--corrosion specimen and its unstressed control.
- 16. A pneumatic device operated by an electrical timer pressurizes the flask at fixed intervals causing the sodium chloride solution to flood into the cells surrounding the stressed and unstressed specimens for ten minutes in each hour. The maximum liquid level in the cell is set to come above the gauge length but below the bottom of the top inner shackle.





Figure B-1: Cross-section of stress-corrosion jig



Figure B-2: Support frame for stress-corrosion jig







**Note:** The originator of the submission should complete items 4, 5, 6 and 7.

This form is available as a Word and Wordperfect-Template on internet under http://www.estec.esa.nl/ecss/improve/

