ABSTRACT

Corrosion testing of mixed-metal and cyclic-stressed aluminum test assemblies are standard requirements for corrosion-inhibiting aerospace sealant materials qualified to MIL-PRF-81733 and SAE AMS3265. Historically, these methods have been unreliable for differentiation between sealants with hexavalent chromium and non-hexavalent chromium inhibitors. Moreover, sealants without corrosion inhibitors have frequently met corrosion performance requirements, resulting in false positives (Type I error). The prevalence of these Type I errors underscores the necessity for a more discriminating test method.

The present study compares the legacy test methods with three alternative test methods, and explores each method's ability to differentiate between hexavalent-chromium corrosion-inhibited, non-hexavalent chromium corrosion-inhibited, and non-inhibited sealants. The three test methods include an X-scribed 3 x 6 inch (7.62 x 15.24 cm) flat panel exposed to neutral salt fog per ASTM B117, a modified cyclic stressed specimen exposed to low temperature stress followed by SO2-Salt fog per ASTM G-85 A4, and a galvanic couple specimen exposed for 14 days in a 3% by weight salt solution. The specimens from the three alternative test methods simulate damaged test surfaces, while the legacy test methods represent more pristine conditions.

The results of this study suggest that the alternative test methods satisfactorily differentiate between the three sealant types. Future directions include revisions to both the MIL-PRF-81733 and SAE AMS3265 specification test requirements to substitute the proposed test methods.

INTRODUCTION

The Naval Aviation Enterprise uses corrosion inhibitive sealants where there is risk for the corrosion of an aircraft's structure. Corrosion inhibitive sealants employ chemical species in their formulations to prevent corrosion, including hexavalent chromium-containing chromates and non-chromate corrosion inhibitors. MIL-PRF-81733 contains the performance requirements for corrosion-inhibitive aerospace sealants. However, the performance tests in the current revision of the specification inadequately differentiate between chromate corrosion inhibitive (CCI), non-chromate corrosion inhibitive (NCCI), and non-inhibitive (NI) sealants. Test data from the three test methods in the specification inaccurately suggest that non-inhibitive sealants perform equally to inhibitive sealants. Potential safety issues arise, given the possibility that an inferior sealant may qualify to a specification designed to qualify products that are used to protect structural and safety-critical assemblies. Therefore, there exists a need to validate and implement more stringent test requirements in a revision to the MIL-PRF-81733 specification. (1)

This study compares three experimental corrosion test methods with the two legacy methods in several accelerated corrosion experiments. The three experimental test methods include “X”-scribed, galvanic cell, and a three-piece
cyclic stressed assembly. The three experimental test methods are chosen to simulate conditions that a sealant will experience on an airframe during service. The legacy test methods are a mixed metal assembly and a five-piece cyclic stressed assembly. The legacy test specimens lack artificial damage, which lowers the probability for moisture intrusion and corrosion.

The X-scribed specimens introduce artificial damage in a coating of sealant to evaluate the protection of the basis metal in a corrosive environment. The galvanic cell pairs a substrate, partially-coated with sealant, to a dissimilar material in an aqueous salt solution. Three-piece cyclic stressed assemblies feature faying surfaces, fastener holes, and a butt-joint. The three-piece assemblies are mechanically-cycled at sub-freezing temperatures and exposed to a corrosive environment. The legacy mixed metal assemblies contain two dissimilar materials that are electrically isolated by sealant in a faying surface. The legacy five-piece cyclic loading specimens are assembled and tested similarly to the three-piece variant but are fully encased in sealant. The data obtained from the methods are examined for differentiation between four CCI sealants, four NCCI sealants, and one NI sealant. The intent of this experiment is not to suggest that the CCI sealants are superior to NCCI sealants. The objective is to select corrosion test methods that reliability differentiate between sealants in both inter-group (CCI vs. NCCI vs. NI) and intra-group (NCCI-1 vs NCCI-2) contexts.

**TEST SETUP**

*Specimen Preparation*

Aluminum alloy 7075-T6 (AA7075-T6) was selected as the material for each of the test methods. 3 x 6 x 0.040 inch (7.62 x 15.24 x 0.102 cm) test panels were used for the X-scribed and galvanic panels, and 4 x 6 x 0.040 inch (10.16 x 15.24 x 0.102 cm) test panels were used for the mixed metal legacy method. The test panels were purchased from a metal supplier, and the 3 x 6 inch (7.62 x 15.24 cm) and 4 x 6 inch (10.16 x 15.24 cm) panels were from the same material lot. For the 3-piece cyclic assemblies, 2 x 4.5 x 0.25 inch (5.08 x 11.43 x 0.635 cm) and 2 x 4 x 0.25 inch (5.08 x 10.15 x 0.635 cm) panels were roughly cut using a band-saw and then drilled, reamed, and cut to size with a Computer Numerical Control (CNC) mill. The legacy 3-piece assemblies were prepared identically, but included 2 x 2 x 0.185 inch (5.08 x 5.08 x 0.470 cm) pieces. All materials were cleaned per AS5127C with solvent using AMS3819 Class A wipes. (2) (3)

*Sealant Selection*

Polythioether and polysulfide sealants were acquired as samples from four sealant manufacturers. For the chromate corrosion inhibited (CCI) sealants, four MIL-PRF-81733 Type II-2, Class 1, Grade A sealants were selected. For the non-chromate corrosion inhibited group, three MIL-PRF-81733 Type II-2, Class 1, Grade B, and one MIL-PRF-81733 Type II-2, Class 2, Grade B sealant was selected. For the non-inhibited sealant one AMS 3277 Type II-2, Class B, sealant was chosen. Follow-on testing included one additional NI and NCCI sealant, which conformed to the AMS3277 and MIL-PRF-81733 specifications, respectively. (4)

<table>
<thead>
<tr>
<th>Sealant Identifier</th>
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<tr>
<td>NI-4</td>
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**Table 1. Selected Sealants**

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Mixed Metal Assembly Test Method

For each sealant tested, twelve 4 x 6 x 0.040 inch (10.16 x 15.24 x 0.102 cm) bare AA7075-T6 panels were cleaned per AS5127C. Two holes were drilled and reamed in the AA7075-T6 panels, and then the panels were chemically conversion coated with a MIL-C-5541 Type II, Class 1A coating (60-80 mg/ft²). The dissimilar materials included AMS4377 magnesium, chemically treated with AMS-M-3171 Type I solution, Alclad, AS4/3501-6 composite, and AMS4911 TiAl₆V₄ titanium alloy panels cut to 2 x 3 x 0.040 inch (5.08 x 7.62 x 0.102 cm), drilled and reamed to align with the drilled 4 x 6 inch (10.16 x 15.24 cm) plates. The Alclad and composite were abraded with 320-grit non-woven mats by hand, and cleaned per AS5127C. The titanium alloy was abraded with a 150-grit non-woven mat by hand and cleaned identically to the Alclad and composite. Spacers were cut from a 0.007 inch (0.018 cm) thickness polyester film to control bond-line thickness. Sealant was extruded from a pneumatic sealant gun onto the AA7075-T6 substrate and dissimilar metal pieces and smoothed with a sealant applicator tool. The nylon fasteners were inserted, the polyester spacers were added, and the two panels were mated and hand-tightened. A sealant applicator tool and plater’s tape masking were used to create a 0.125 ± 0.015 inch (0.312 ± 0.04 cm) fillet around the 2 x 3 inch (5.08 x 7.62 cm) dissimilar material. The test specimens were cured for two weeks, placed in ASTM G85 Annex 4 for four weeks, and then inspected for corrosion in the fillet, faying surface, and fastener regions with internally-developed rating criteria. (5) (6) (7) (8) (9)

3-Piece Cyclic Stressed Assembly Test Method

For each sealant tested, four assemblies were prepared. Each assembly was made from two 2 x 4.5 x 0.25 inch (5.08 x 11.43 x 0.635 cm) and one 2 x 4 x 0.25 inch (5.08 x 10.15 x 0.635 cm) AA7075-T6 treated with a MIL-C-5541 Type II, Class 1A coating (60-80 mg/ft²). Prior to chemical conversion coating, the pieces were roughly cut from 0.25 inch (.635 cm) plate AA7075-T6 on a band-saw, then drilled, reamed, and cut to size with an CNC mill. Four and eight 0.25 inch (.635 cm) holes were drilled into the 2 x 4.5 inch (5.08 x 11.43 cm) and 2 x 4 inch (5.08 x 10.15 cm) specimens, respectively, with the former bearing four 0.5 inch (1.27 cm) diameter, 100 degree countersinks on one side. The edges of each panel were de-burred with hand tools. The pieces were abraded with 320-grit non-woven mats by hand, and cleaned per AS5127C. The individual pieces were chemically conversion coated with a MIL-C-5541 Type II, Class 1A coating (60-80 mg/ft²), dried for 24 hours at ambient temperature and humidity, and stored until sealant application.

The conversion coated specimens were cleaned per AS5127C prior to sealant application. Sealant was applied to the faying surfaces of the assemblies with a pneumatic sealant gun and sealant application tool. The shanks of MS24694-S99 cadmium-plated, 0.25 inch (.635 cm) steel screws were coated with approximately 0.020 inch (.051 cm) of sealant, and inserted into the fastener holes. AN315-4R cadmium plated steel hexagonal nuts were used and polyester film spacers were cut from a 0.007 inch (0.018 cm) thickness sheet with an inner diameter of approximately 0.28 inch (.71 cm) and an outer diameter of approximately 0.30 inch (.762 cm) The spacers were placed over four of the fasteners to control bond-line thickness. An extra line of sealant was applied near the butt-joint surfaces to ensure adequate squeeze-out into the butt-joint upon assembly. Each fastener was hand-tightened, and excess squeeze out was cleaned from the exterior of the assembly with wipes wetted with solvent.

After a two-week sealant cure, the assemblies were cleaned with solvent, primed with MIL-PRF-23377 Type I, Class N primer, and sprayed with MIL-PRF-85285 Type I topcoat. The organic coatings were cured for a minimum of two weeks at ambient temperature and humidity, then select edges and fastener heads were scribed. (10) (11)

The cyclic specimens were loaded into mechanical test frames and cycled 250 times at -65°F (-53.9°C) for the polysulfide specimens, and -80°F (-62.2°C) for the polythioether specimens. The test specimens were loaded randomly into plastic test racks at 15 degrees from vertical, and placed in acidic salt fog cabinets compliant with ASTM G85 Annex 4 for up to six weeks. Half of the specimens were removed at four weeks and the balance at six weeks. The specimens were mechanically stripped with non-metallic scrapers, and the remaining sealant was stripped using a methylene chloride paint stripper. The specimens were evaluated for corrosion with a rating criteria developed by a Hexavalent Chromium Free Sealant Evaluation Team in an industry-government consortium project. (12)

A total of 36 assemblies were prepared for nine sealants.

5-Piece Cyclic Stressed Assembly Test Method

The 5-piece assemblies were prepared identically to the 3-piece assemblies with several distinct changes: (1) The inclusion of two 2 x 2 x 0.185 inch (5.08 x 5.08 x 0.470 cm) panels that were drilled and reamed with four 0.25 inch
(0.635 cm) holes then inserted between the faying surfaces (2) The use of MS24694-S103 cadmium-plated fasteners (vice -S99) (3) A brush-on overcoat of the entire assembly with ~0.020 inch (0.051 cm) of sealant, vice organic primer and topcoat (4) An X-scribe on the surface of one fastener head panel. The specimens were tested and evaluated identically to the 3-piece specimens.

A total of 36 assemblies were prepared for nine sealants.

**Galvanic Cell Test Method**

For each sealant tested, nine 3 x 6 x 0.040 (7.62 x 15.24 x 0.102 cm) inch bare AA7075-T6 panels were abraded with 320-grit non-woven mats by hand, and cleaned per AS5127C. The AA7075-T6 was not processed with chemical conversion coatings. The dissimilar materials included Alclad (7075-T6 clad with 1000 series aluminum), AS4/3501-6 composite, and AMS4911 TiAl6V4 titanium alloy cut to 3 x 6 x 0.040 inch (7.62 x 15.24 x 0.102 cm). The Alclad and composite were abraded with 320-grit non-woven mats by hand, and cleaned per AS5127C. The titanium alloy was abraded with 150-grit non-woven mats by hand and cleaned identically to the Alclad and composite. 0.020 inch (.051 cm) vinyl thickness tape was cut into 0.50 x 3 inch (.051 x 7.62 cm) strips and applied to the AA7075-T6 substrate such that 0.50 inch (1.27 cm) strips of exposed substrate were created. Sealant was extruded with a pneumatic applicator gun, and applied to the test panel at a 0.020 inch (0.051 cm) thickness with a sealant application tool and custom fixture. After 24 hours, the tape was removed to reveal six evenly-spaced, 0.50 inch (1.27 cm) width, 0.020 inch (0.051 cm) thick sealant strips on the substrate panel. After a minimum of 14 days to cure, the dissimilar material was affixed to the AA7075-T6 substrate with binder clips and then sealed along the 3 inch (7.62 cm) edges with the appropriate sealant. After a minimum of 14 days to cure the edge sealant, the completed assemblies were electrically connected with a copper alligator clip and placed in 3% by weight (W/W) sodium chloride solution, made from deionized water and food-grade salt. The three triplicate panels for each dissimilar material were placed in the same container for each sealant for two weeks. As the salt solution evaporated, the solution level was maintained with daily additions of deionized water to the test vessels.

The test specimens were removed from the salt solution at the conclusion of the two-week immersion. The dissimilar material was separated from the AA7075-T6 substrate, and the substrate was photographed before and after the removal of the sealant strips. Test specimen corrosion was rated with internally-developed rating criteria. The test specimen was divided into two regions for analysis: sealant-covered and exposed aluminum substrate.

A total of 90 test specimens were prepared for the salt solution immersion.

**X-Scribed Test Method**

For each sealant condition, six 3 x 6 x 0.040 inch (7.62 x 15.24 x 0.102 cm) bare AA7075-T6 panels were abraded with 320-grit non-woven mats by hand, and cleaned per AS5127C. There was no chemical treatment of the AA7075-T6. Sealant was extruded with a pneumatic applicator gun, and applied to the test panel at a 0.040 inch (7.62 x 15.24 x 0.102 cm) thickness with a draw-down bar and custom fixture. After a two-week cure in ambient lab conditions, the test panels were engraved with a 3 mm (0.020 inch) scribe using a rotary engraving machine. Once scribed, the test specimens were affixed to a plastic test rack at 15 degrees from vertical in a random order. The plastic racks were placed in neutral salt fog cabinets conforming to ASTM B117 for up to eight weeks. (13)

The test specimens were removed once each week and photographed, and either returned to the cabinet, or removed, stripped of their sealant, and photographed. Prior to sealant removal, the test panels were evaluated using ASTM D1654 for undercutting away from the scribe and an internally-developed rating criteria for corrosion of the scribe region. (14)

A total of 60 test specimens were prepared for the initial round of testing. An additional 12 (NI-3 and NI-4) specimens were created for follow-on testing, and 14 specimens (NCCI-5) were prepared by manufacturer “2,” and tested alongside NI-3 and NI-4.

**RESULTS AND DISCUSSION**

**Mixed Metal Assembly Test Method**

Figure 1. and Figure 2. display the corrosion results for the legacy mixed metal assemblies for the fastener and field regions, respectively. Figure 3. shows an example of the visual change in the conversion coating that was unique to the polythioether sealants. Figure 4. displays the corrosion results for the area under the fillet. The purpose of this test method is to assess the sealants’ ability to prevent ingress of moisture and corrosion into the sealant-covered area, and its ability to isolate and protect dissimilar metal interfaces. The fastener, field, and fillet regions
were evaluated by the same visual inspection criteria. A rating of “1” indicated no detectable change to the conversion coating. A rating of “2” equated to the lightening of the conversion coating shown in Figure 3. A rating of “3” specified there was minor pitting (fewer than 10 pits) in the area of observation. A rating of “4” indicated there was major pitting (greater than 10 pits) in the area of observation. A rating of “5” described white corrosion product in the area of observation.

The results for each sealant’s four mixed metal pairings appear on the same graphic. The fastener region was defined as the area up to 0.125 inch (0.32 cm) diameter from the fastener holes, the field region was defined as the area of the faying surface, and the fillet region was defined as the 0.125 inch (0.32 cm) width area under the fillet seal. No corrosion or pitting was observed for any of the sealants in the fastener or field areas. A visual lightening of the chromate conversion coating was observed on polythioether NCCI-4, NI-1, and NI-2 test articles, as shown in Figure 3. It was unclear whether the lightening of the conversion coating related to the progression of corrosion, or an unrelated phenomenon. Pitting of the AA7075-T6 substrate was observed in fillet region for all of the sealants tested and for each dissimilar material, as indicated in Figure 4. Pitting under the fillet provided minimal differentiation between the three sealant groups, CCI, NCCI, and NI. This test method failed to differentiate between the three sealant groups.
3-Piece Cyclic Stressed Assembly Test Method

The assembly, exposure, and inspection of the 3-piece assemblies was completed, but the data is not shown. The purpose of this test method is to assess the sealants’ ability to prevent ingress of moisture and corrosion into the sealant-covered areas, which include faying surfaces, fillets, butt joints, fastener holes, and countersinks. The unique aspect of this test method is the mechanical cycling of the specimens at low temperatures to simulate in-service airframe conditions. A preliminary review of the data (not shown) suggested this test method failed to differentiate between the three sealant groups.

5-Piece Cyclic Stressed Assembly Test Method

The assembly, exposure, and inspection of the 3-piece assemblies was completed, but the data is not shown. The purpose of this test method is the same as the 3-piece assemblies. A preliminary review of the data (not shown) suggested this test method failed to differentiate between the three sealant groups.
**Galvanic Cell Test Method**

Figure 5. and Figure 6. display the galvanic cell assemblies’ corrosion test results for the “sealant-covered” and “exposed substrate” regions, respectively. This test method addresses the ability of the sealant to prevent ingress of moisture and corrosion into the sealant-covered area, and its ability to protect surrounding areas of exposed substrate to simulate damage occurring at dissimilar metal joints. The sealant-covered and exposed aluminum regions were evaluated by the same visual inspection criteria. A rating of “5” specified the absence of corrosion, “4” equated to discoloration of the aluminum substrate, “3” indicated pitting, “2” designated uniform corrosion, and “1” indicated corrosion product build-up.

The data in Figure 5. for the sealant-covered region suggest that the Alclad dissimilar pairing affords little differentiation between sealants, likely due to the small potential difference between the AA7075-T6 and the Alclad materials. However, the composite and titanium pairings reveal a hierarchy in performance, with some sealants performing better than the intra-group and inter-group competitors. Similarity in the performance of several CCI sealants and NI sealants within titanium and composite specimens was attributed to poor adhesion of the CCI-1 and CCI-4 sealants in the test. Excluding CCI-1 and CCI-2, which lost adhesion, the observed trend was that the CCI sealants performed better than the NCCI and NI sealants, while the NCCI sealants outperformed the NI sealants. The data from the sealant covered region suggest differentiation between the three sealant groups with this test method.

![Figure 5. Corrosion Ratings for Galvanic Cell Sealant-Covered Regions](image)

Data from the exposed substrate region in Figure 6. suggest that CCI sealants outperform the NCCI and NI sealants for each dissimilar material pairing. There is some differentiation between the NCCI and CI sealants, though the clearest delineation was between CCI and NCCI/NI sealants.

The human factor of subjectivity in the corrosion rating system creates disagreement between the visual and recorded data. The visual data shown in Figure 7. suggest that the galvanic cell test method is able to differentiate between the three sealant groups. In Figure 7. the CCI sealants exhibit reduced corrosion and loss of substrate relative to the NCCI and NI sealants. A careful visual inspection of the panels allows the operator to quickly distinguish between the groups of sealants. This test method also enables the operator to rank sealants within a group. For example, CCI-1 and CCI-2 outperformed CCI-4.
Figure 6. Corrosion Ratings for Galvanic Cell Exposed Aluminum Regions

Figure 7. Galvanic Cell Visual Corrosion Test Results
**X-Scribed Test Method**

Figure 8. and Figure 9. display the corrosion rating data for the X-scribe test specimens’ “scribe” and “field” regions. Figure 10., Figure 11., and Figure 12. depict the visual corrosion data for each sealant group. From left to right in each visual data image, the four panels represent 168, 504, 672, and 1344-hour duration test articles. The scribe region was defined as the area within the 0.118 inch (3 mm) width scribe, and the field was defined as the area covered by the sealant, excluding the scribe and the 0.25 inch (0.635 cm) border around the panel to eliminate edge-effects. The rating criteria for the scribe in Figure 8. assigned a rating of “1” as a free of corrosion or staining, a “2” indicated staining without corrosion product build-up, a “3” designated trace to moderate corrosion product, a “4” specified considerable coverage of the scribe with corrosion product, and a “5” indicated corrosion product overflowed from the scribed area.

Figure 8. indicates that differentiation between CCI and NCCI/NI sealants was possible at 168 hours. Similarly, differentiation between NCCI and NI sealants was obtained at 168 hours. Instances in which the panel appears to “recover” are addressed with the fact that one or more panels were used for each time interval without reuse. The extension of the test duration beyond 168 hours resulted in the progression of undercutting into the field region in NCCI and NI panels. In this study, undercutting was defined as the propagation of corrosion beneath the sealant surface.

![Figure 8. Corrosion Ratings for X-Scribed Panel Scribed Areas](image)

Figure 9. displays the corrosion rating data for the X-scribed specimens field regions throughout the test duration. For the sealants tested, undercutting originated from the scribed area and progressed outward. The criteria from ASTM D1654, Table 1 were used to measure the distance of the corrosion creepage away from the scribe. A rating of “10” indicated there was no undercutting (corrosion creepage) beneath the sealant. A rating of “0” specified that the undercutting extended more than 0.63 inch (16 mm) away from the scribe. Ratings between “10” and “0” represented undercutting of various distances away from the scribe. (14)

In each time interval, the CCI sealants outperformed the NCCI and NI samples. Differentiation between CCI and NCCI/NI and between NCCI and NI was achieved at 168 hours. NI-1, NI-2, NI-4 and NCCI-4, all polythioether sealant chemistry, displayed severe adhesion failure, which resulted in greater undercutting away from the scribe. However, NI-3, a polythioether sealant, performed similarly to the NCCI sealants. NI-3 and NI-4 specimens were produced from the same sealant, but NI-3 did not use the manufacturer-recommended adhesion promoter. The visual corrosion data in Figure 12. suggest that the sealant performed significantly better without its adhesion promoter.
Figures 10-12 display the visual corrosion data for each sealant group. The panels are arranged in sets of four for each sealant. From left-to-right, the panels represent the test exposure durations of 168, 504, 672, and 1344 hours in ASTM B117 neutral salt fog exposure. Figure 10 contains the CCI sealants, Figure 11 contains the NCCI sealants, and Figure 12 contains the NI sealants. The common degradation mechanism was general corrosion of the scribe, followed by undercutting away from the scribe beneath the sealant layer.

The visual data in Figures 10-12 suggest that the CCI sealants resisted general corrosion and undercutting better than the NCCI and NI sealants. NCCI-4 in Figure 11 and NI-1 and NI-2 in Figure 12 are polythioether sealants that displayed poor adhesion, which may have contributed to their poor resistance to undercutting. In Figure 12., NI-3 and NI-4 are the same sealant, but the corrosion performance is much worse for NI-4. The manufacturer-recommended adhesion promoter was used for the NI-4 specimens.
CONCLUSIONS

This study explored three alternative sealant corrosion test methods to replace or update existing test methods in MIL-PRF-81733 and AMS3265. The mixed metal specimens did not corrode in the fastener or fay surface regions; this test method was inadequate to assess the corrosion inhibiting sealants. A preliminary review of the data (not shown) from the 3-piece and 5-piece cyclic stressed assemblies indicated these two test methods could not differentiate between the three sealant groups. The X-scribed and galvanic cell alternative test methods successfully discerned chromate corrosion inhibited (CCI), non-chromate corrosion inhibited (NCCI) and non-inhibited (NI) sealants. Moreover, the X-scribed and galvanic cell tests offer simple test article designs, and require equipment or simple salt solutions that are readily available at current test labs. The X-scribed and galvanic cell test methods’ ability to differentiate between and within the three sealant groups will support revisions to MIL-PRF-81733 and AMS3265. Revisions may include the substitution of the galvanic cell and X-scribed test methods for the mixed metal and 5-piece cyclic stressed assembly methods. Additional efforts to improve the corrosion rating criteria are warranted. Image-based, quantitative data collection may minimize subjectivity in future studies. (15).
REFERENCES