



Results of A-2 Schjeldahl Connector  
"Green Crud" Investigation

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ABSTRACT

This ATM presents the results of the investigation into the Array A-2 "green crud" problems. A green substance had been found under the KYNAR sleeving at each of the soldered terminations of the Schjeldahl jack connections on the A-2 central station.

Briefly, the analysis demonstrates that the presence of this green substance, or residue, does not represent a degradation in the functional integrity of the ALSEP experiment interface connections; it is strictly a cosmetic effect. This green crud results from a chemical reaction with metallic oxides in flux residue which gets trapped under the insulation of the wire, and not from an attack of the wire at the soldered connection. In addition, these residues are nonconductive, noncorrosive, and will not propagate.

This apparent problem was not related to the failure of the P/J 35 connector at KSC (Reference: ALSEP Failure Analysis Report A-24).

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INTRODUCTION

Following the cold test failure at KSC, of the PSE connector P/J 35, sub-pack I of the Array A-2 ALSEP was returned to Bendix Aerospace Systems Division, Ann Arbor, Michigan for failure analysis and repair. Prior to initial rework operations, all Schjeldahl experiment interface connectors on the sub-pack were thoroughly inspected. This inspection revealed the presence of a green substance under the KYNAR sleeving at each of the soldered terminations. An investigation was initiated to identify the green substance and to determine whether or not the functional integrity of the ALSEP experiment interface connections had been compromised.

University of Michigan Analysis

Dr. R. D. Pehlke, a metallurgist associated with the University of Michigan was contacted for assistance in the evaluation of this problem. A sample of the green crud and KYNAR sleeving from the A-2 sub-pack and a similarly contaminated connector from the prototype C sub-pack were given to Dr. Pehlke for analysis.

At the University of Michigan, the KYNAR sleeving was removed from the connector and the soldered terminations were examined at 40X using a binocular microscope. Nearly all of the junctions exhibited the green-colored deposit. The deposit was spongy, non-adherent to any of the metals and appeared to be associated with the teflon insulation of the manganin wire. At several of the junctions the insulation was split and peeled back; green crud was found as far as one-half inch from the stripped end of the insulation. There was no evidence of chemical attack on the surface of the teflon or the KYNAR which were in contact with the green crud, and the deposit did not adhere to either material.

A contaminated pin was removed from the connector, mounted, and a polished cross-section prepared for the soldered junction. This polished section was examined at magnification up to 600X. The surfaces of the manganin wire and the solder were smooth and showed no pitting or other evidence of corrosive attacks; neither did the gold plated beryllium-copper terminal. For comparison, a cross-section of manganin wire which was not in contact with the green crud was prepared and examined at 600X. The wire sections were comparable and showed that the manganin in contact with the green crud was not reduced in cross-section, however the surface oxide layer had been removed.



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The green deposit was not electrically conductive; resistance measurements made with needle probes across several pieces of the residue (approximately 1/16 inch diameter particles) indicated resistances in excess of 20 mega-ohms. Visual examination of the distribution of the residue on the wire and on the KYNAR sleeving indicated a non-contiguous deposit which would further inhibit conduction.

Two samples of the KYNAR sleeving, one with the green deposit and one without the deposit from bonded stores were prepared for x-ray diffraction analysis. The x-ray patterns were identical and indicated a definite crystalline substance; however since only one sample contained green crud, the pattern must be attributed to the KYNAR sleeve. This result indicates that the green deposit is amorphous, or a non-crystalline substance.

A piece of the KYNAR sleeve taken from the contaminated A-2 connector was prepared for electron microprobe examination. Element identifications were taken at three areas analytically surveyed. The elements identified were: chlorine (strong), copper (strong), as well as manganese, nickel, tin and lead. The microprobe does not provide an indication of the presence of low atomic number elements from about sodium (11) down, and hence, the presence or absence of carbon, nitrogen, oxygen, hydrogen and flourine are not discernable. An uncontaminated sample of the KYNAR was also examined; no radiation peaks were present indicating that the previously identified elements were in the green deposit.

Discussions with Kester Solder Company

The Kester Solder Company was also contacted for assistance in identifying and evaluating the formation of the green deposits at the soldered terminations. Kester was very co-operative and provided much information relating to the circumstances surrounding our problem.

Kester has been studying the actions and effects of flux and flux residues for over 50 years. These green deposits result from a reaction between metallic oxides removed from the surfaces to be soldered and the acids present in the flux residue. These reactions take place while the residue is still in an ionized state, that is, prior to complete evaporation of the residue solvents.



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A properly soldered electrical connection is the result of a chemical alloying action between the metals of each surface and the solder. For the proper alloying action to take place, each surface must be clean and oxide-free. If the surfaces haven't been previously cleaned and given a protective coating (e. g. a thin gold plating), an activated rosin flux capable of reducing and coagulating the surface oxides is applied. Then when the solder is applied the molten solder pushes a "pool" of flux along the surface, removing the oxides and enabling the alloying action.

Fluxes for soldering electrical connections contain natural rosins or resins which can be dissociated into active ionic constituents that are capable of chemical action. By themselves they are inert and incapable of reducing surface oxides. The ionic activity is induced by the application of heat when soldering with flux-cored solders or by dissolving the rosins in an ionizing agent such as isopropyl alcohol, which is the case with liquid fluxes such as Kester 1544.

To speed the fluxing action of ionized natural rosins, a catalytic activator is added. The activated rosin is a homogeneous, single-entity proposition with properties that parallel and duplicate those of the original rosin; the increased activity is due to a reaction by the activating agent on the polymerized molecules of the rosin. The agent does not directly enter the reduction reaction and cannot be isolated as a different reaction in the fluxing process. Without this activator, the action of the ionized natural rosins is too slow to adequately reduce surface oxides. In the Kester "44" fluxes (which includes the 1544 liquid flux), the activating agent is ammonia-free, amine-chloride.

All liquid fluxes, including core fluxes which are in a molten state ( $\sim 100^{\circ}\text{C}$ ), are corrosive and electrically conductive as long as they are in a liquid state. This is due to the ionizing power exerted by the "solvent" in dissociating the rosins into active ionic constituents. Once the solvents have evaporated, the resinous residue is inert, non-corrosive, non-conductive and can not propagate. For further reactions to occur, the residue must be reactivated. Water will not react with flux residues and by itself will not induce further activity; however, addition of isopropyl alcohol will induce activity as will the addition of more liquid flux. Temperatures sufficient to liquify the rosins in the residue may also reactivate it.



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The green crud formation is caused by excessive use of liquid flux and failure to successfully remove all flux residues after soldering operations. The flux residue remaining after soldering is readily wicked up under the teflon insulation on the wire, especially when diluted with isopropyl alcohol while attempting to remove excess flux and flux residues. These trapped residues do not dry out immediately and the reaction continues, producing the cosmetic effect. To remove excess flux, Kester recommends using anhydrous organic liquids which will dissolve the residue enabling the removal of excess flux and flux residue.

Studies at North American - Downy

At North American - Downy, an investigation was undertaken to attempt to identify the nature of similar green colored deposits. In their opinion they are an organic complex and include chlorine and metallic ions. The presence of this deposit causes only a cosmetic effect, since once the solvents have evaporated, the remaining residue is inert.

Washing soldered junctions with alcohol has been traced to be a part of the problem leading to the formation of the green-colored residue. Flux residues dissolved in the washing solution will readily leak up under the teflon covering of the wire under capillary action; under the teflon, the rate of solvent loss is severely retarded. These trapped solutions remain active and will form the green deposits. If access to air is provided, or if the soldered junction is placed in an evacuated chamber, the volatile solvents are readily evaporated.

In standard soldering procedures, isopropyl alcohol is used as the washing agent. This alcohol is highly deliquescent, readily absorbs water, and promotes chemical reactions in the flux residue. At North American, the use of tri-chloro-ethane was explored. It was found that this solvent readily dissolves the flux if used immediately, and the solutions trapped under the wicking action do not lead to the formation of green deposits. It's solvent action on aged residues is not as good as that of isopropyl alcohol.

At North American, they feel that the most feasible approach to avoid the formation of green deposits is to use tri-chloro-ethane and immediately wash the soldered junctions. Evacuating the junctions will remove all solvents from under the teflon covering, but this procedure is difficult to use in standard soldering operations.



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CONCLUSIONS

The green residue is a result of chemical reactions taking place in flux residues trapped under the teflon insulation of the manganin wire. Excessive application of liquid flux during the soldering operation, coupled with the isopropyl alcohol applied when attempting to remove the flux residue, provides the necessary conditions for these reactions. The diluted flux residue is readily wicked up under the teflon insulation under capillary action, preventing the solvent evaporation. Until the residue has dried out, it remains in an ionized state and capable of further chemical reaction.

The natural rosins in the liquid flux contain carboxylic acids, principally abietic:  $C_{19}H_{29}CO-OH$ . These acids will react with metallic oxides to give ionic-metallic salts and water. With the residue in an ionized state, organic and hydroxyl radicals, ionized atomic hydrogen, oxygen, and metallic ions will be present. These constituents combine as the solvents evaporate, forming the green-colored, complex metallic organic residue.

To prevent this problem from recurring, the flux residues must be prevented from wicking up under the insulation. This is difficult to accomplish in actual practice, but to minimize the effects:

1. Keep solder flow approximately one wire diameter's distance from stripped edge of insulation when tinning wire.
2. Do not attempt to solder to non-tin-dipped connector terminals.
3. Try soldering with just flux-cored solder and no externally applied liquid flux.
4. If (3) above is unsuccessful, to minimize the flux residue, just touch the end of the wire with flux when tinning.
5. Remove flux residue with tri-chloro-tri-fluoro-ethane, or any approved anhydrous organic solvent.
6. If feasible, place soldered junction in a hard vacuum to remove trapped solvents.



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After evaluating the information gathered and the results of the analyses performed by Dr. Pehlke at the University of Michigan, it can be stated that the presence of this green-colored residue does not represent a degradation in the functional integrity of the ALSEP experiment interface connections. And further, if the proper precautions are taken during the assembly of interface cabling the incidence of green crud can be significantly reduced, if not eliminated.