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SOLVENT REMOVAL OF EC-2273 POTTING COMPOUND FROM F-4 AIRCRAFT ELECTRICAL COMPONENTS

By
Porter W. Erickson
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14 MAY 1971

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A safe, fast, and effective solvent system has been developed for the removal of EC-2273 encapsulant. This material, which is now showing signs of failing, is the potting compound used in several hundred components of a large number of Navy F-4 aircraft.

The preferred solvent system consists of a mixture of N-methyl-2-pyrollidone (M-Pyrol), benzyltrimethylammonium hydroxide and methanol. Virgin, as well as deteriorated, EC-2273 is cleanly removed from a typical electrical connector simply by immersion of the component in the solution for a period of a few hours.

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SOLVENT REMOVAL OF EC-2273 POTTING COMPOUND FROM F-4 AIRCRAFT ELECTRICAL COMPONENTS

A practical, safe, economical and effective solvent depotting system designed for Naval Aircraft Rework Facilities (NARF) use has been developed. This permits the rapid removal of deteriorating EC-2273 potting compound used in the electrical connectors and relays of a large number of our F-4 aircraft.

This system could, perhaps, be refined further, particularly in regard to washing methods for the optimization of electrical insulation properties of depotted components. However, present requirements in this regard are met by the proposed solvent system. The system is now ready for practical demonstration in the field.

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The mention of any trade names or manufacturers in this report shall not be construed as an official endorsement or approval of such products or companies by the United States Government.

GEORGE G. BALL
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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INTRODUCTION

This Laboratory undertook a task for the Naval Air Systems Command in FY 1970 to investigate a serious materials problem with the F-4 aircraft. The problem involved the deterioration of a proprietary potting compound used in many of the electrical components of these aircraft.

During the period from about 1958 to 1965, a potting compound called Pro-Seal 777 had been used. In 1963, after several years of presumably satisfactory performance, it was discovered that this material occasionally changed and reverted from a solid to a viscous, sticky gum. Since the electrical insulation properties of the resin were also dangerously deteriorated, these electrical connectors were, for some years, either replaced or depotted and then repotted on a per case basis. By 1965, a very large number of such failures was beginning to show up, especially in aircraft operating in humid tropical climates. Meanwhile the defective material, Pro-Seal 777, was being replaced by a more reversion resistant proprietary material known as EC-2273 (a 3M product). Then, in 1965, a decision was made to use this compound in all future new F-4 construction.

This change in potting materials, however, was not the end of the problem. In 1968, 1969 and 1970, the Air Force discovered occasional failures even with this improved material. Certain blocks of aircraft, such as "Block-29," had a relatively large number of documented cases of reversion. The pattern of failures was very similar to that experienced with the earlier use of Pro-Seal 777, and it seemed as if history was about to repeat itself. In late 1969, EC-2273 was, in turn, replaced by a reversion resistant polysulfide in all new F-4 construction. Thus the Navy, in July 1969, had every good reason to expect on the basis of previous experience an oncoming massive and costly repair bill on all F-4 aircraft having EC-2273 potted electrical components. On the basis of the data available at that time, it was predicted that the problem would peak in, perhaps, 1972 or 1973.

In view of this time factor, the Navy had the opportunity to investigate methods of blunting or possibly avoiding the costly depotting-repotting route used with the Pro-Seal 777 and which had failed in the earlier F-4's. The cost of complete reworking of these aircraft was over $40,000 per plane and involved an excess of 2,000 manhours (NARF experience).

The McDonnell Aircraft Company, builder of the F-4, had also become involved in finding solutions to the problem. A more detailed history of potting compounds for the F-4, which was compiled by McDonnell, is given in reference (a).

Our task for the Naval Air Systems Command involved two approaches to the EC-2273 problem. These consisted of (1) a search for satisfactory chemical methods of reconstituting the material in situ and (2) an investigation of improved methods of depotting electrical components without removal from the aircraft. This report deals with the depotting study only. NOLTR 71-53 discusses the restoring of EC-2273 in situ.

The main result of this study was the finding of a practical, safe, economical and effective solvent system which cleanly removes even virgin EC-2273 and which can be adapted to NARF use.

EXPERIMENTAL

A screening program was initially set up to evaluate a number of materials and methods for relative effectiveness in the removal of virgin EC-2273. The more promising of the solvents tried, the laboratory depotting specimens used, and the general methods of evaluation are described below. The details of the latter are shown in the appendix.

A. Materials

EC-2273 was provided by the 3 M Company, St. Paul, Minnesota. N-methyl-2-pyrollidone (M-Pyrol) is an industrial grade solvent which was obtained from the GAF Corporation (cost $0.62/lb, F.O.B. Calvert City, Ky.).

Benzyltrimethylammonium hydroxide was obtained from the Marshall Division of Miles Laboratories, Inc., 1127 Myrtle Street, Elkhart, Indiana 46514. This was supplied as a 40% solution in methanol of a cost of $2.00 per pound.

Choline was supplied as a 45% solution in methanol by the Rohm and Haas Co., Independence Mall West, Philadelphia, Pa. 19105 at a cost of $1.28 per pound F.O.B. plant.

Sanitone, a dry cleaning detergent, was supplied by Emery Industries, Cincinnati, Ohio. Soramine CAZ-75, GAFAC RE-610, and GAFAC RM-510, which were solvent soluble detergents, were supplied by the GAF Corporation.

All other solvents used were purchased from chemical supply houses. The grades of these generally met ACS quality standards.

Two-pin electrical connectors, type SCO 6 P22-11P, were obtained from the Bendix Corporation. These realistic models of actual F-4 connectors had neoprene inserts and conformed to applicable military specifications.

B. Specimens

For all screening experiments, cylindrical discs, 1 3/8" in diameter and 1/2" high, were cast and cured to a Shore A hardness of 65 to 68 (see Fig. 1). The total weight of material was about 16 ± 1 g. The end of a 13" long, 0.063" diameter soft steel wire was embedded in the center of the disc to facilitate manipulation and positioning of the specimen in the solvent for the dynamic depotting experiments. Mixing and curing of this two-component system was done according to 3 M recommendations.

The two-pin connectors were filled with potting compound to within 1/8" of the top of the potting boot (Fig. 1).

C. Methods of Evaluation

Two methods of depotting were studied. The first involved maintaining a continuous, controlled rate of flow of the solvent over the specimen which was immersed in the solvent (see Fig. 2). Weight loss was noted at half-hour or hourly intervals. This will be referred to as the "Dynamic Method." In the

second method, the specimen was simply suspended (complete immersion) in the solvent without agitation. Weight loss was noted usually at two-hour intervals. This is the "Static Method." For the most part, electrical connectors were depotted using this latter method.

RESULTS

Table 1 gives a comparison of the relative effectiveness of the two organic bases, BTAH and choline, dissolved in various solvents, to remove the EC-2273 potting compound under dynamic conditions. The data indicate that both bases are very effective when dissolved along with 15% acetone in either of the three solvents--dimethylacetamide (DMAc), dimethylformamide (DMF), or tetrahydrofuran (THF). BTAH was, however, sufficiently better than choline with two of these solvents to merit selection for further extensive study. There was no synergistic effect from mixing 1:1 ratios of BTAH and choline in any of these three solvents.

Table 2 shows selected dynamic weight loss data on similar specimens after 2 hours in 13 different mixed solvent combinations. None of these contained acetone. The only system which exceeded 80% weight removal after 2 hours, other than for the three solvents shown in Table 1, was M-Pyrol.

The storage stability of three BTAH solvent systems was determined. Depotting effectiveness of a THF-BTAH solution was unchanged after 3 days, while the DMF-BTAH solution lost 78% of its activity in this period of time. The M-Pyrol-BTAH system showed no loss in activity even after 13 days.

Early experiments with stable solvent combinations were found to have a much faster rate of mass removal during the first 15 minutes of a run than during the succeeding 15 minutes, which in turn was faster than the following 15 minutes, etc. Data in Table 3, particularly with the THF system, show that the amine base is consumed in the process. The use of trace amounts of "dry cleaning" type detergents (less than 1%) had virtually no effect in promoting the rate of potting compound removal for the several solvent systems tried.

Table 4 shows the depotting results with three different solvent systems, all containing 10% BTAH, under static conditions. The screening specimen (about 16 grams) was completely dissolved by all systems in less than 8 hours. It took 13 to 16 hours, however, to statically depot a freshly potted test connector.

It was found that the electrical insulation resistance of connectors following solvent depotting was in many cases unacceptably low. This could be improved to some extent by extensive methanol and water washing but it never approached the resistance values for new test connectors prior to or after potting. Figure 3 shows megohm resistance versus time on new unpotted two-pin connectors which were suspended in the vapors above several solvent systems. The BTAH has apparently far less deleterious effects on the electrical resistance of the neoprene insert than many of the solvents themselves.

Figure 4 shows similar data obtained when these connectors were immersed in the liquid solvents. Electrical resistance goes down to less than one megohm in a matter of a few days. Bar graph (X) of Figure 4 shows the remarkable recovery

of resistance values for these same connectors which had been washed and air dried for 13 days. Bar graph (Y) of Figure 4 shows that the equilibrium resistance values which were reached in four hours were considerably reduced on these same "recovered" connectors after exposure to 95% R.H.

Single and mixed solvents were investigated for potential as "swell and pick" systems. The best of these investigated at this point (without optimization) is trifluoroethanol and a mixture of M-Pyrol, methylethyl ketone and methanol. Figure 5 shows the deteriorated condition of a potted test connector which had been immersed (statically) in trifluoroethanol for 24 hours. Figure 6 shows a similar connector which had been immersed in the M-Pyrol-methylethyl ketone-methanol mixture for two days. Both solvent systems obviously drastically degrade virgin EC-2273.

DISCUSSION OF RESULTS

Several solvent systems had been investigated earlier both here and elsewhere for the removal of the ester based polyurethane potting compound, Pro-Seal 777. As far as was known, all of these solvents contained a caustic such as potassium hydroxide (KOH) which presumably hydrolyzed ester linkages in the material. Connectors depotted with these systems were, however, extremely difficult to restore electrically and very extended washings with methanol, water, and even detergents were required to bring the insulation values up to minimum requirements for repotting. In at least one case, the solvent was itself badly deteriorated by the caustic.

EC-2273 also contains hydrolyzable linkages which had to be exploited in its removal. The chemical stability of this 3 M product was, however, very much greater than that of Pro-Seal 777. No solvent could be found in the early investigations which dissolved the virgin material without the use of alkali.

Benzyltrimethylammonium hydroxide is one of the strongest organic bases known. It was first tried as a component in place of KOH in methylene chloride, similar to that used in depotting Pro-Seal 777. This system was effective if used immediately after mixing. It was soon found, however, that the interaction between this solvent and the organic base was also such as to completely destroy its depotting effectiveness in a matter of hours.

Meanwhile, the Naval Air Development Center (NADC) had investigated to some extent the effectiveness of another organic base, choline, in certain solvents (ref. (c)). The first part of the EC-2273 depotting investigation was, therefore, a search for effective solvents which were compatible with these bases.

Table 1 shows dynamic weight loss data on screening specimens (virgin condition) after 120 minutes with three different solvents which are well known for good solvent properties. The data show that DMAc, DMF, and THF with 15% acetone, 10% BTAH and 10% methanol (volume basis) all dissolve about 90% of the material in two hours. With the exception of DMF solvent, choline is about 10% less effective than BTAH. A 1:1 mixture of BTAH and choline in these three solvents showed no synergistic effect. The BTAH-choline systems were almost equivalent to BTAH alone with these solvents.

Discussions with Commander Barboo of the Navy Bureau of Medicine and Surgery and Mr. Ralph Wands of the Advisory Center on Toxicology of the National Research Council regarding NARF use of the above solvents were discouraging. It was pointed out that DMF is very hazardous to personnel and THF has a flash point of -14°C. DMAc is also very costly and probably not available in large enough quantities for general NARF use.

A decision had been made on the basis of data in Table 1 to extend the study of BTAH with a number of other available solvents. Acetone was not added in this case. The results in Table 2 show that M-Pyrol, while not as fast as the three solvents tried earlier, dissolved about 80% of the potting material in a period of two hours. M-Pyrol is generally free from the objections cited above. It is a widely used industrial solvent, is relatively nontoxic, has a flash point of 204°F, and is available in tank car lots at 50 cents per pound.

An early problem in the depotting of Pro-Seal 777 had been the rapid deterioration of the solutions after mixing the components. Stability of the depotting solvent systems is very important for at least two reasons. It is desirable, certainly for NARF use, to prepare amounts up to, perhaps, several hundred gallons which could be used in smaller quantities as needed. The components of the system should, therefore, be chemically compatible with each other so as not to deteriorate in depotting effectiveness. Storage stability should certainly be greater than one month. The methylene chloride-methanol-KOH system used earlier for removing Pro-Seal 777 had to be freshly mixed to be effective. A more important reason that a solvent system should not self-destruct with time is that even freshly prepared solutions rapidly lose depotting effectiveness. Static depotting requires several hours for the average electrical connector. Mass depotting of aircraft connectors with a pumping device, such as used at NARF, Cherry Point in 1969, involved large volumes of solvent (perhaps 50 gallons). An unstable system even freshly prepared is very unsatisfactory in such instances because most of the active material is destroyed by the solvent before it has an opportunity to act on the potting compound.

Table 3 gives data which show that the effectiveness of the BTAH-DMF system rapidly deteriorates with time. This is clearly evident when compared to the data from the BTAH-THF system which is also shown. The specimen in each case was replaced with a fresh one at 30-minute intervals to minimize the effect of reducing specimen surface area as it was consumed. The greatly reduced weight loss rate after 60 minutes with the THF-BTAH system can be attributed to the BTAH being consumed in the chemical destruction of the potting compound. It was later shown that a BTAH-M-Pyrol system is stable at room temperature for two weeks. BTAH-THF is stable for 3 days and probably very much longer, since this cyclic ether (THF) would be expected to be very resistant to hydrolytic scission. The BTAH-DMF system lost 78% of its effectiveness in 3 days. Dimethylformamide is apparently easily hydrolyzed.

One thing to be borne in mind here, however, is that even a BTAH solvent system is not stable for long in air. BTAH is a strong base and will rapidly absorb carbon dioxide to form carbonate and thus destroy itself for depotting use. All depotting with these systems must, therefore, be done in such a fashion as to generally exclude exposure to the atmosphere.

Several solvent soluble detergents with and without traces of water were investigated for effectiveness as components of the solvent system in speeding up depotting rate (ref. b). There were instances when some slight improvement was noted; but generally the benefit was not worth the complication of additional components in the system. This area was, however, not investigated enough to rule out discovery of any possible future benefit, particularly in regard to later washing for improving electricals between connector pins.

An important consideration in a NARF depotting operation is simplicity. Mass depotting of connectors in situ on an aircraft using a solvent pumping system is not only complicated, but messy and less than totally safe. NARF (North Island) used a static system for Pro-Seal 777 in which the potted connectors were individually immersed for several days in a plastic bag containing the solvent. Swelling and disintegrating of the material occurred to the extent that it could be removed by the "pick" method.

Several of the systems tried under dynamic conditions were evaluated for effectiveness under static conditions. The screening specimens were simply immersed in the depotting solvent in a glass jar and weight loss determined every two hours. Results in Table 4 show that 3 different systems will totally dissolve a 16 gram specimen in less than 8 hours. No effective system was found that did not contain BTAH. Atlas 62, which is used by NARF (North Island) for removing Pro-Seal 777, was totally ineffective without BTAH. It did, however, swell EC-2273 but did not render the material suitable for "pick" removal.

To completely depot a connector statically, however, it took up to 16 hours. With no help whatever (agitation or probing), it takes very long to dissolve away the last traces of potting compound buried within the wire bundle. Figure 7 shows such a connector with visible potting material remaining within the wire bundle at the base. It is estimated that most F-4 connectors can be completely depotted in 3 days by this system without any interim attention.

It is very important that the electrical resistance values across circuits of depotted connectors be at an acceptable level before repotting. A tentative requirement now is 10 megohms in a 95% RH equilibrium environment.

The new unpotted two-pin connectors had insulation values up to 100,000 megohms. After potting, the values would still be about 50,000 megohms. In contrast, it was soon found that depotting virtually ruined connector insulation values. This was similar to the experience at NARF (Cherry Point) with proprietary solvents containing KOH when depotting Pro-Seal 777. Washing systems and procedures, often very complicated, using methanol, water, acetone, methylene chloride with and without detergent were tried.

It had been predicted that connector clean-up would be far simpler using non-KOH systems. At first it was suspected that BTAH, being a strong organic base, would, even in trace amounts, become an electrolyte in moist environments. Several experiments were carried out to gain an understanding of this problem with "washing". The best system to date involved alternate methanol-water washing as shown in the appendix.

Figure 3 shows selected unusual and unexpected results. New unpotted connectors were suspended in the vapor above the liquids shown and the pin-to-pin resistance

was measured daily. The reason for operating in the vapor phase was to avoid effects of possible unknown ionic contaminents in the solvents. Resistance was reduced only slightly in a BTAH-methanol atmosphere over a 20-day period. However, M-Pyrol, DMAc and DMF vapors resulted in greatly degraded electrical insulation values. With DMF, the resistance dropped from 100,000 to 10 megohms in only 4 days. The resistance with M-Pyrol dropped also but was still about 50 megohms after 20 days.

Figure 4 shows the devastating effects on electrical insulation values when new unpotted connectors were immersed in M-Pyrol, Atlas 62, and Acrylite B. Atlas 62 is used by NARF (North Island) to depot Pro-Seal 777. Acrylite B is a proprietary material which has been promoted for removal of Pro-Seal 777 because the depotted connector can be restored electrically by a simple water washing. In a matter of minutes the resistances dropped to a fraction of the original values and after 6 days were too low to be measured on the megohm meter. The connectors were then removed, rinsed with methyl alcohol and placed in a ventilated hood at room temperature for 13 days. Bar graph X shows a remarkable recovery of electrical insulation values with Acrylite B being best, M-Pyrol next and Atlas 62 last, but the latter still had a pin-to-pin resistance of almost 100 megohms. These connectors were then placed in a 95% R.H. environment. The resistance values at equilibrium all dropped but the connector exposed to M-Pyrol still retained a resistance value of about 50 megohms and was clearly the best one. The connector treated with Acrylite B gave about a 10 megohm resistance and Atlas 62 was down to about 2 megohms. These measurements were made across the connector terminals without removal from the high R.H. environment.

Later experiments showed that all of these liquids have very low resistivities. Enough of these solvents penetrating the neoprene insert presumably results in the poor insulation values. Apparently the neoprene recovers its electrical properties as the solvent escapes and presumably would improve even more with a longer drying period.

It should be mentioned that M-Pyrol does not noticeably swell a neoprene insert even after several days. It does, however, gain measurable weight upon immersion which it is slow in losing under ambient conditions.

Several solvent systems were investigated for "swell-and-pick" use. Many BTAH containing systems which dissolve virgin EC-2273 greatly swell the material. In most cases, however, the swollen material was a very tough rubber and could not be broken up by "pick" methods. Solvent swollen EC-2273 was even frozen in liquid nitrogen and then plunged into boiling water to promote cracking and breaking up of the material. Some cracking did occur but upon warming to room temperature the bulk of the mass was still a tough rubber. Atlas 62, methylene chloride, THF, and DMF are all good swelling agents.

The most promising "swell-and-pick" system tried so far was trifluoroethanol. This had been previously studied by McDonnell Aircraft (ref. d). After 4 days immersion, virgin material on a connector was very deeply cracked. Figure 5 shows the condition after 2 days. The material was then quite easily removed with a spatula within a period of minutes. The material at the center which was least accessible to the solvent, still resisted removal, but it was no longer rubbery, so that it still could be broken up. It is estimated that a longer immersion (perhaps 10 days) might convert all of the material to a condition

that it could be removed with a stiff brush. The crudely cleaned up connector after being given a methylethyl ketone (MEK) rinse and dried had a pin-to-pin resistance of 300 megohms. This rose to 80,000 megohms after 3 hours in open air and maintained a 15,000 megohm resistance in a 95% R.H. equilibrium environment (3 days). The neoprene insert showed no signs of swelling. The other "swell-and-pick" system tried was the M-Pyrol-MEK-methanol mixture. After 4 days immersion, some of the material on a connector could be hand removed (with difficulty). Figure 6 shows the condition after 2 days. This system did not noticeably swell the neoprene insert either. This was clearly not as effective in terms of speed as trifluoroethanol.

A limitation on NARF use of trifluoroethanol could be its toxicity. The Advisory Center on Toxicology reports (ref. e) that the LD_{50} vapor concentration (inhalation) is only 1.6 mg/100ml. (LD_{50} means that this vapor level kills 50% of the experimental animals at a standard condition.) In view of this the M-Pyrol based system, even though more sluggish, probably merits further study.

The McDonnell Aircraft Corporation has also studied "swell-and-pick" systems for EC-2273 particularly for material reverted to a Shore A hardness of, perhaps, five (ref. f). Material reverted to this degree is quite easily removed by "pick" methods when frozen to liquid nitrogen temperatures or by using hot trichloroethylene to further soften it. An advantage with their methods is that the electrical resistance values are not degraded by the depotting operation. Their system does not, however, work on virgin or partially reverted material. These methods are excellent for depotting an occasional "runner on a per case basis in the field. The methods would not be satisfactory for use in a general aircraft rework job, since most connectors would not be reverted to a sufficient degree for the system to be effective.

CONCLUSIONS AND RECOMMENDATIONS

A satisfactory solvent system based on commercial BTAH solutions and M-Pyrol is effective in depotting a typical connector in less than 3 days under static conditions. Methanol-water washes of the depotted connectors followed by two weeks of air drying should result in acceptable electrical properties for repotting. An expected objection to the use of this system is the offensive odor of BTAH.

Trifluoroethanol is also effective as a "swell-and-pick" system. A four-day immersion of a connector detriorates even virgin material such that the potting compound can be removed by spatula (with some difficulty). It is estimated that after a 10-day immersion the material could be removed with a stiff brush. The electricals are not degraded by this process, thus avoiding troublesome electricals clean-up. This system presents, however, considerable toxicity hazards.

It is recommended that the BTAH-M-Pyrol solvent system be introduced to the NARFs on a trial basis using spare parts potted with EC-2273. This would provide valuable training in the use of the method. More investigative work should also be done to optimize the "swell-and-pick" system based on trifluoroethanol in event this would be approved for NARF use.

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f. Ref. d, Weekly Report, Period ending July 3, 1970, p.2

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TABLE 1

COMPARISON OF EFFECTIVENESS OF BTAH AND CHOLINE BASE IN DEPOTTING EC-2273 UNDER DYNAMIC CONDITIONS[a]

BASE	SOLVENT	SPECIMEN WEIGHT LOSS (%) AFTER 120 MINUTES
BTAH[e]	DMAc[b]	88.5
BTAH[e]	DMF[c]	91.2
BTAH[e]	THF[d]	98.0
CHOLINE[f]	DMAc	74.5
CHOLINE[f]	DMF	90.6
CHOLINE[f]	THF	86.5
BTAH/CHOLINE (1/1)	DMAc	85.9
BTAH/CHOLINE (1/1)	DMF	91.3
BTAH/CHOLINE (1/1)	THF	98.6

a. Solutions consisted of 60% solvent, 15% acetone, 10% base and 15% methanol (Volume basis)

b. Dimethylacetamide

c. Dimethylformamide

d. Tetrahydrofuran

e. Benzyltrimethylammonium hydroxide

f. 2-hydroxyethyltrimethylammonium hydroxide

TABLE 2

COMPARISON OF SEVERAL SOLVENTS CONTAINING 10% BTAH IN DEPOTTING EC-2273 UNDER DYNAMIC CONDITIONS[a]

SOLVENT (75%)	SPECIMEN WEIGHT LOSS (%) AFTER 120 MINUTES
37.5% Tetrahydrofuran +37.5 Dimethylacetamide	95.5
Tetrahydrofuran	94.2
Dimethylformamide	88.5
M-Pyrol	82.4
Dimethylacetamide	76.1
Nitrobenzene	71.7
Monochlorobenzene	61.7
Toluene	59.5
Bromobenzene	56.2
1,4-Dioxane	53.0
Dichlorobenzene	46.8
Butanol	35.9
Benzylalcohol	29.5

a. Each system also contained 15% of Methanol (Volume Basis)

TABLE 3

LOSS OF BTAH DEPOTTING EFFECTIVENESS[c]

SOLVENT COMPOSITION[a]	%WEIGHT LOSS ON SUCCEEDING FRESH[b] SPECIMENS AFTER 30 MINUTES			
	1st Spec.	2nd Spec.	3rd Spec.	4th Spec.
60% THF, 10% BTAH, 15% Acetone, 15% Methanol	48	47.7	33.5	28.7
30% THF, 30% DMF, 10% BTAH, 15% Acetone, 15% Methanol	45.8	33.5	28.7	24.5

a. Volume percent

b. Partially dissolved specimen replaced with a new specimen after 30 minutes to minimize effect of changing surface area.

c. Dynamic conditions.

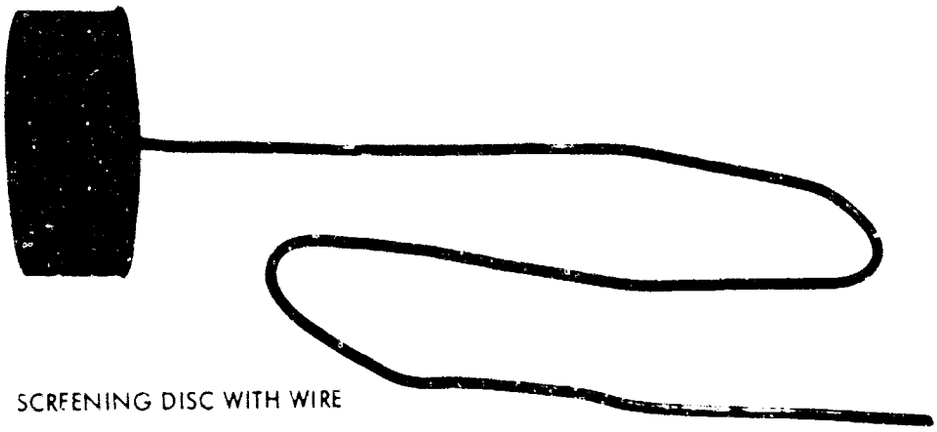
TABLE 4

STATIC DEPOTTING

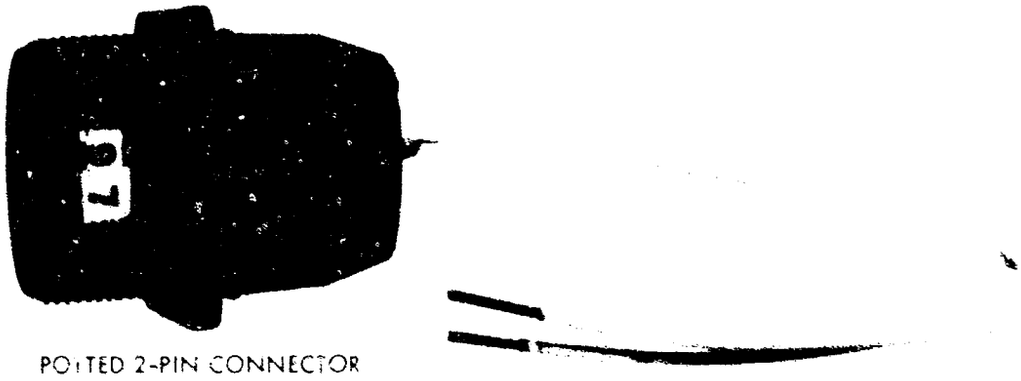
SOLVENT SYSTEM[a]	DEPOTTING TIME (HOURS)[b]
75% M-Pyrol, 15% Methanol, 10% BTAH	7.6
70% M-Pyrol, 15% Methanol, 5% Acetone, 10% BTAH	7.3
75% Atlas 62, 15% Methanol, 10% BTAH	6.1

a. Volume percent

b. Time to totally dissolve disc specimen



SCREENING DISC WITH WIRE



POTTED 2-PIN CONNECTOR

FIG. 1 DEPOTTING SPECIMENS

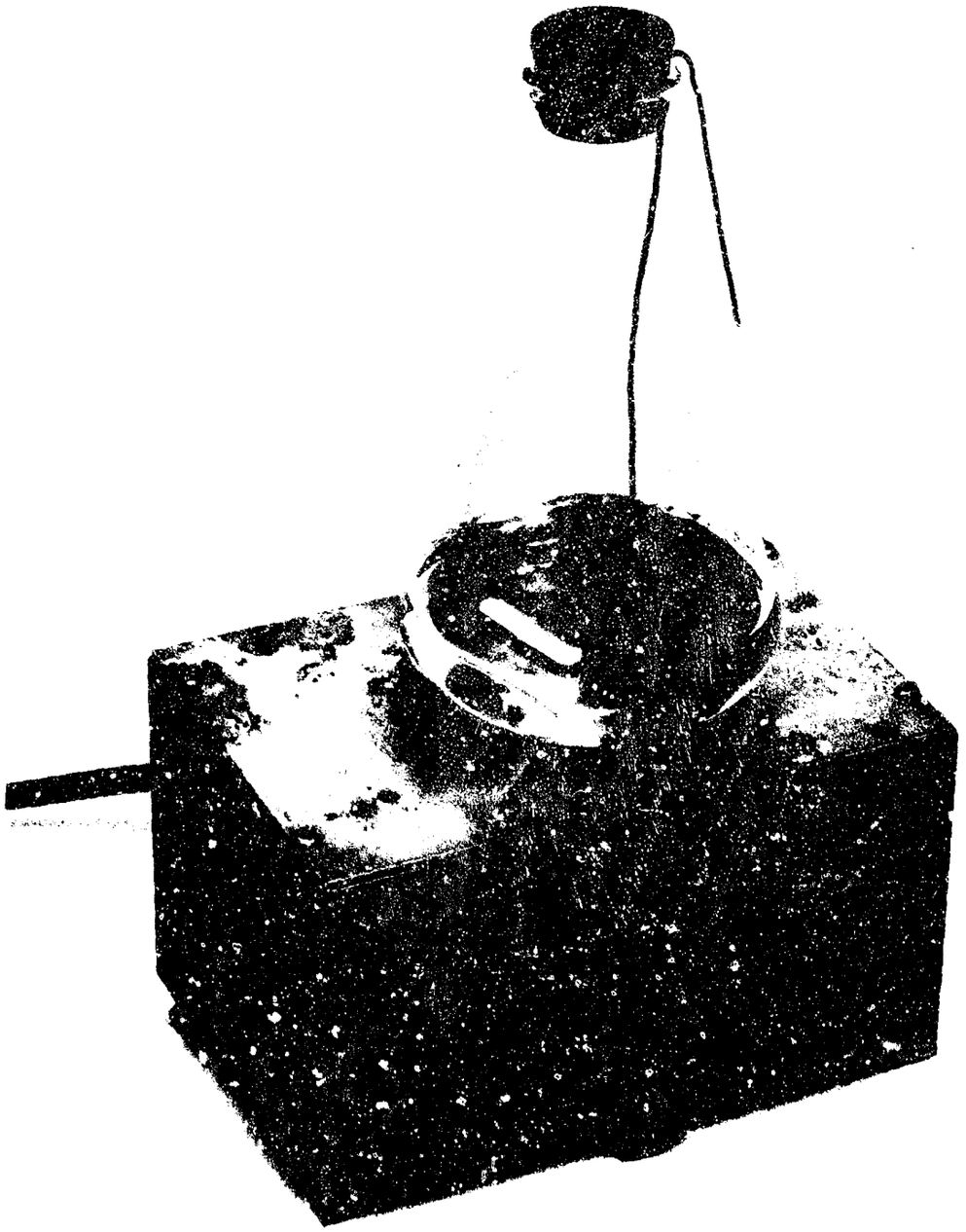


FIG. 2 DYNAMIC DEPOTTING APPARATUS

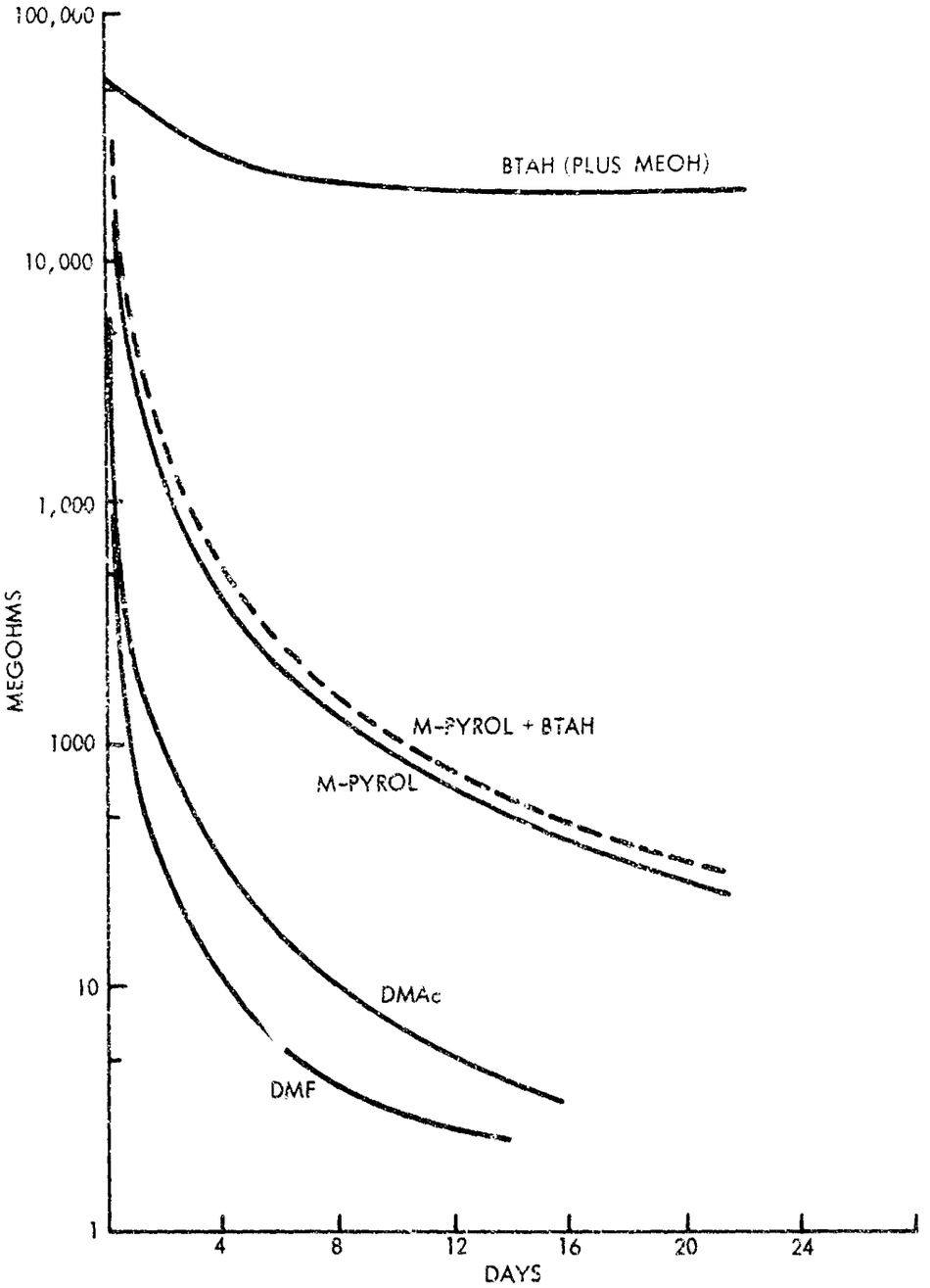


FIG. 3 EFFECT ON ELECTRICAL INSULATION PROPERTIES OF NEOPRENE CONNECTOR INSERTS WHEN EXPOSED TO SOLVENT VAPORS

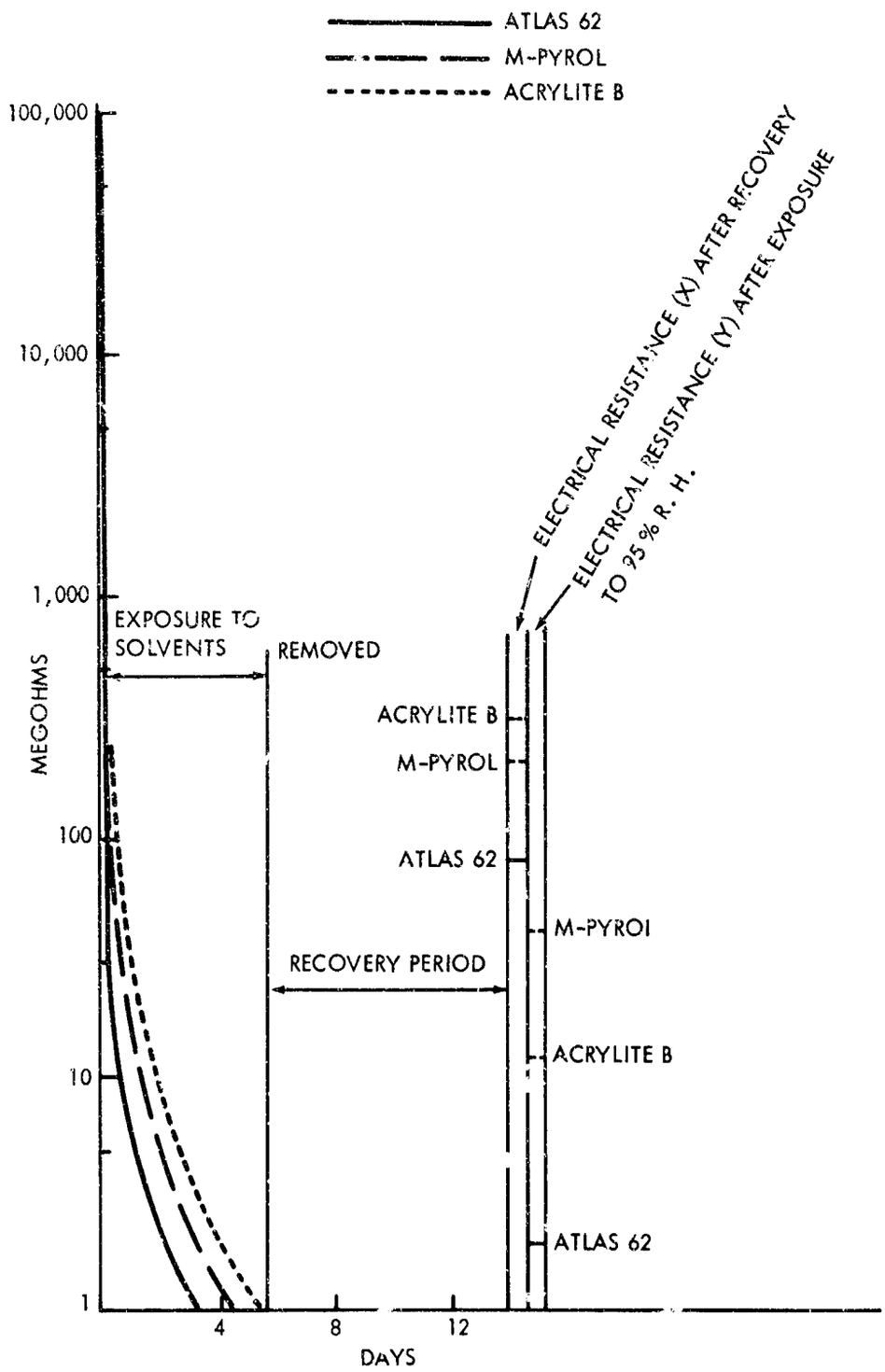


FIG. 4 EFFECT ON ELECTRICAL INSULATION PROPERTIES OF NEOPRENE CONNECTOR INSERTS WHEN IMMERSED IN VARIOUS SOLVENTS

FIG. 5 EFFECT OF 24-HOUR EXPOSURE TO TRIFLUOROETHANOL

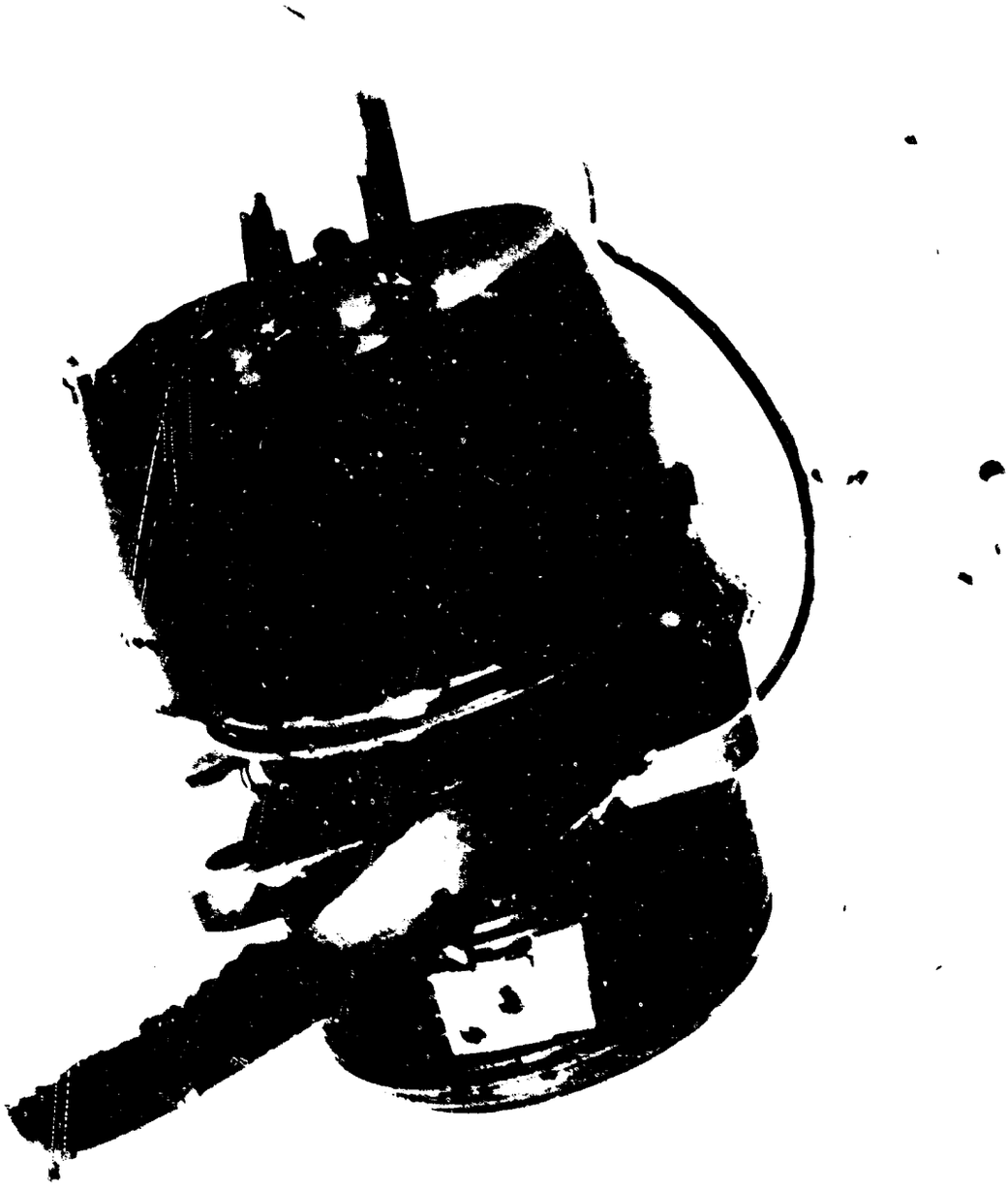


FIG. 6 EFFECT OF 2-DAY EXPOSURE TO M-PYROL-MEK MIXTURE

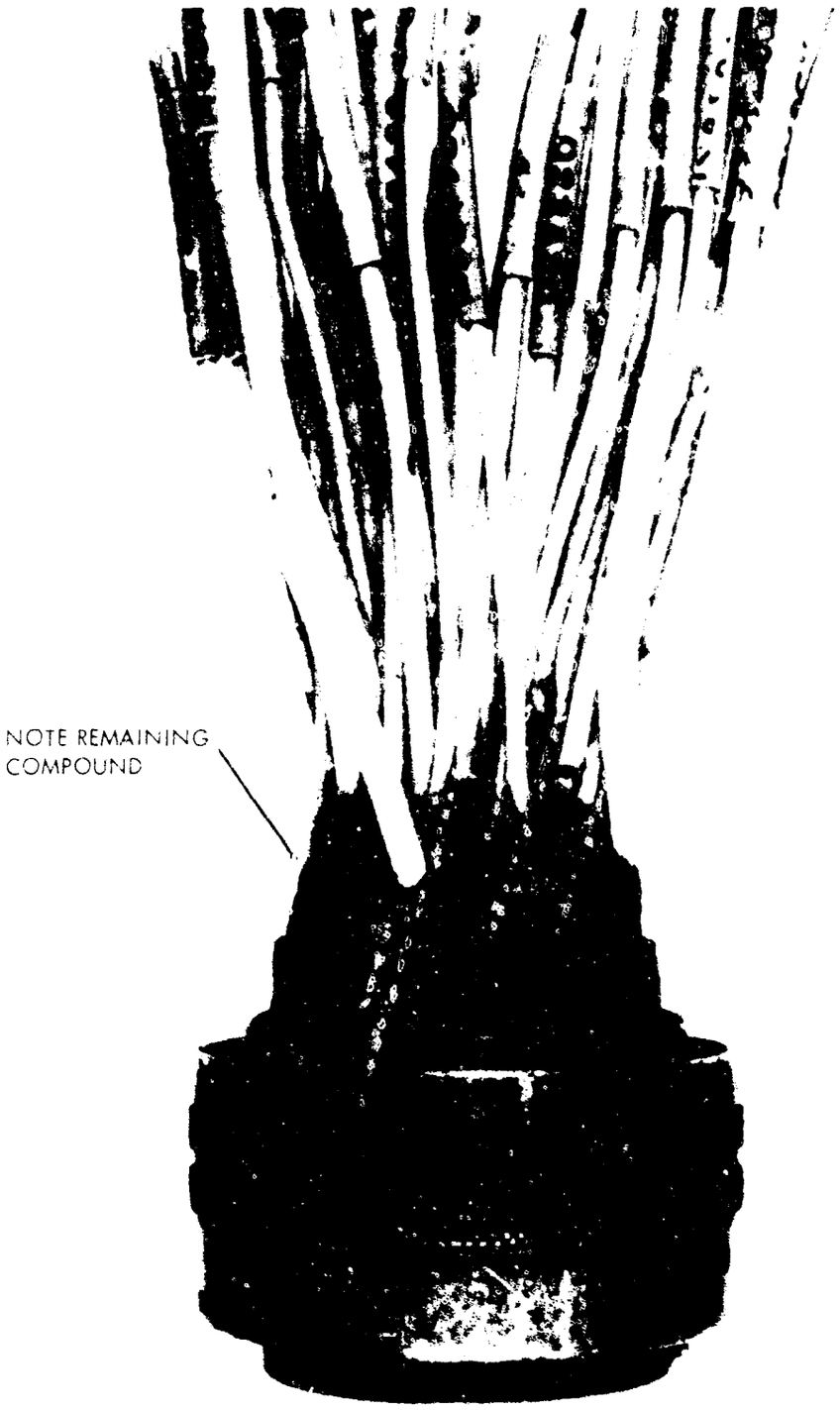


FIG. 7 INCOMPLETELY DEPOTTED CONNECTOR AFTER 19 HOURS OF STATIC DEPOTTING

APPENDIX A

EXPERIMENTAL DETAILS

A. DEPOTTING TEST SPECIMENS

The screening specimens were cast in "Cerro" metal molds. These specimens were discs, 1 3/8" in diameter by 1/2" high, and weighed about 16 ±1 g. EC-2273 Part A, which was kept frozen in storage at -60°F, was warmed up to room temperature. Part B, which was stored at about 40°F, was heated to about 150°F. A quantity of each of Parts A and B, in the ratio of 1 to 5 by weight, were weighed out in a glass beaker in sufficient amount to make about 20 specimens. The components were then mixed by hand without undue air entrapment using a spatula. The inside walls of the beaker were carefully "spaded" with the spatula to achieve a very thorough mixing of the entire batch. The mixture was then subjected to about 10mm Hg of vacuum and degassed until the foam broke. Immediately following this, the material was poured into the molds which had previously been coated with 3 M's No. 1711 mold release.

Curing was carried out in an oven at 225°F for 2 1/2 hours. Prior to setting up, the end of a 13" long, 0.063 diameter soft steel wire was embedded into each specimen. This facilitated later handling and positioning of the specimen for the dynamic depotting experiments. The cured specimens had a Shore A hardness of 65 to 68.

B. POTTED CONNECTORS

Standard two-pin electrical connectors (male and female), type SCO 6P22-11P, manufactured by the Bendix Company, were similarly potted with EC-2273. The rear of the neoprene insert had been previously coated with 3 M's EC-11945 primer to promote bonding between the potting compound and the insert. Parts A and B of the primer were mixed in a 1:1 ratio and applied as a coating, which was cured for 8 hours at 72° ±1°F. The polyethylene potting boot (which served as a mold) was repositioned on the connector base and filled to within 1/8" of the top. The potted material was then cured as above.

C. DYNAMIC DEPOTTING PROCEDURE

For the BTAH-solvent depotting experiments, 50 ml of BTAH and 150 ml of the solvent were placed into a one liter Erlenmeyer flask. A 1 1/2" long Teflon enclosed bar magnet was inserted into the liquid. The flask was then placed on a "Mag-Mix" type stirrer, which was specially made for this work and which rotated at a fixed speed of 200 RPM. The weighed screening specimen to be depotted was then placed into the solution. It was positioned firmly on the bottom at the edge of the flask such as not to be thrown around in the liquid by the stirring bar. This was possible because of the wire embedded in the specimen. An approximately four-inch square piece of aluminum foil was then pressed down over the mouth of the flask to minimize exposure of the solution to the atmosphere.

The "Mag-Mix" motor was started, time noted, and the depotting commenced. At 30 or 60 minute intervals (as required), the specimen was removed from the flask, excess solution shaken off and placed for five minutes on "Kim-Wipe" tissue

to drain off. It was then weighed to the nearest tenth of a gram and immediately replaced in the solution. Depotting resumed for another given time interval, as before. The procedure continued until the material was consumed (or for two hours). Weight loss was plotted versus time. The results given in this report are generally those showing weight loss after two hours.

D. STATIC DEPOTTING PROCEDURE

A 200 ml solution of the depotting system sufficient to immerse the specimen was placed in a wide-mouth glass jar. A wire platform to suspend the specimen about 1/2" off the bottom had previously been placed in the jar. This was made from ordinary aluminum standard mesh window screen. The specimen was positioned at about a 30° angle on the platform. It was found that depotting action was more efficient on the top side if the specimen was thus tilted. The wide mouth jar, which was a screw cap type, was now closed and the depotting proceeded statically.

At the end of each two hours, the specimen was removed, shaken, dried and weighed as before. Complete solvation of 16 grams occurred in eight hours or less with the better systems. Some were left overnight where depotting action was slower.

Potted two-pin connectors were also depotted in a similar manner but weight loss was determined at longer time intervals. The rate of depotting was slower and more erratic due to the constantly changing geometry and smaller exposed surface area. The potting boot was, of course, removed before the depotting commenced. Figure 7 shows a 30-pin connector after 19 hours of static depotting where a substantial amount of compound still remains within the crowded wire bundle.

E. USE OF DETERGENTS

Several experiments were carried out where two to six drops of liquid dry cleaning detergents were added to the depotting solvent. Similar amounts of water were also added in some cases along with the detergent to note the effect on rate of depotting, usually with the best systems found at that point in time.

F. CONNECTOR WASHING

Following depotting, a number of washing routines were tried to optimize electrical insulation values between circuits. In general, the freshly depotted connector was immediately subjected to the following:

a. Immersed in stirring methanol for five minutes.

b. Removed, excess solution shaken off, and placed for five minutes in a fresh stirring methanol bath.

c. Removed, shaken off, and immersed in stirring water bath for five minutes.

d. Step "c" repeated once.

e. Step "b" repeated once.

f. Connector placed in a 140°F oven and dried for 30 minutes.

g. Removed and placed on the lab bench for at least 30 minutes to cool to room temperature.

There were variations of the above, such as the addition of detergents to the early water wash solutions. The bath usually consisted of a 500 ml beaker containing about 300 ml of the solvent which was rapidly stirred with a "Mag-Mix." The connector was not permitted to "dry" off after the depositing before washing was started.

G. ELECTRICAL INSULATION TESTS

The electrical resistance across the two-pin connectors was determined using a General Radio Megohmmeter, Type 1862-B. This instrument has a range of 0.5 to 100,000 megohms. All measurements were done at 500 volts.

Measurements were made on the connectors which had equilibrated to lab bench environment (section F, step g) and after equilibration to 96 ±1% R.H. For the latter test, 24 hours prior to the first readings the connectors were placed into a closed desiccator whose R.H. environment was maintained at ambient temperature by a copper sulfate-water mixture (ref. (g)). The connector leads extended through holes in the desiccator cover, which had been drilled for that purpose. The space around the wires was further sealed with molding clay. Resistance measurements were made daily without removal of the connectors from the humid environment.

H. VAPOR EXPOSURE OF CONNECTORS

New unpotted connectors were exposed in the vapor above BTAH (in methanol) and above several solvents for a period of up to 20 days. The electrical resistance was determined daily without removal of the connector from its environment. Data such as shown in Figure 3 were obtained which reflect the effect of vapor absorption on insulation properties of the neoprene insert.

I. EXPOSURE OF NEOPRENE TO SOLVENT

Unpotted connectors were immersed in several solvents. The electrical resistance dropped very rapidly, as shown in Figure 4. The connectors were removed from the respective solvents, washed with methanol and water, and then rinsed with methanol. After drying in air, the resistance was measured. The resistance could not be measured on the connector in the solvent since the resistances of all the solvents were very low--below the lower range of the meter.

After the resistance had fallen below 1 megohm, the connectors were left to "dry" in an air circulating hood for 13 days, when the resistance was again determined.

At this point the connectors were placed in the 95% R.H. environment and the resistance determined as before (Fig. 4, bar Y).