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A Compatibility Problem with the MK-148 Electric Delay Primer

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ABSTRACT

The MK-148 electric delay primer (Figure 1) has a bridge-wire beaded with 2-4 mg of a mix containing approximately 90% normal lead styphnate (NLS) in a nitrocellulose binder cured by solvent evaporation. A ferrule or sleeve is potted (Scotchcast #8 epoxy resin) onto the bridge-wire plug.

Additional epoxy resin is used to seal the bridge-wire ferrule to a stainless steel delay body containing, in sequence, an airgap, A-1A, delay mix, A-1A and lead azide. The Scotchcast #8 epoxy adhesive is an amine-cured resin. The amines, all low molecular weight and volatile, are major components of this adhesive and, as such, are specified by the U.S. Navy for use in this item in spite of literature reports of incompatibility with explosives.

This investigation was begun as a result of failure of some MK-148's for contract N60530-81C-0178 to fire after 28-day Temperature and Humidity (T&H) cycling between -65° and +160°F, with a cumulative exposure at the high temperature of approximately 384 hours.

Microscopical study of pure unmilled normal lead styphnate (NLS) and milled NLS beads showed extensive chemical attack during exposure to the vapors of curing epoxy even at room temperature. NLS exposed to epoxy vapors for 16 hours at 160°F was essentially destroyed, as shown by polarized light microscopy and x-ray diffraction.

Amine-cured adhesives are incompatible with lead styphnate; basic lead styphnate is more stable than NLS to amines but would still risk failure in units such as the MK-148. No amine-cured adhesives should be used in ordnance components requiring dependable performance of lead styphnate. (FR)

INTRODUCTION

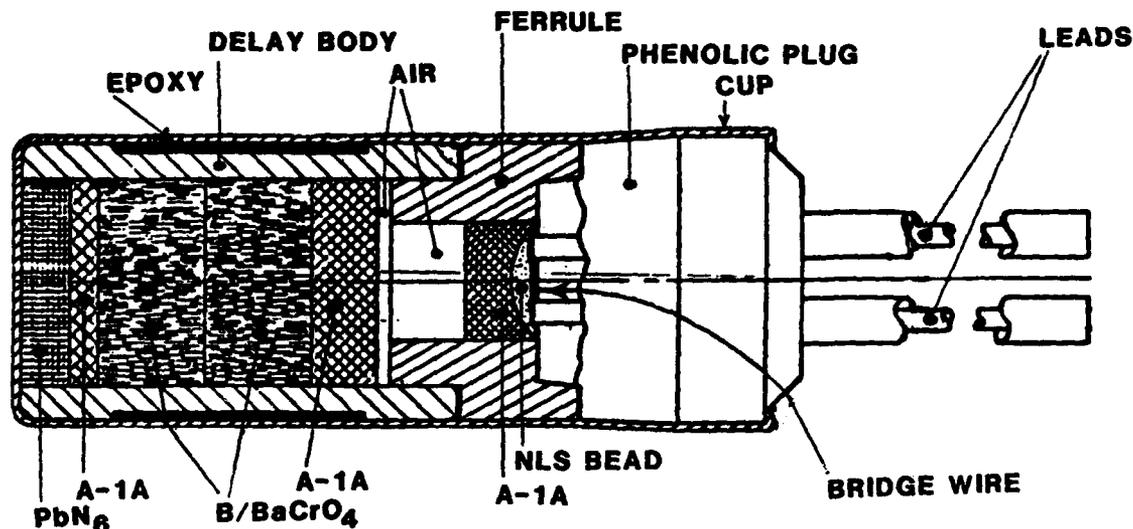
The MK-148 electric delay primer (Figure 1) has a phenolic bridge plug with a nichrome bridge-wire beaded with 2-4 mg of a mix containing approximately 90% NLS in a nitrocellulose binder cured by solvent evaporation. A ferrule or sleeve is potted onto the plug. A charge of A-1A is pressed into the ferrule over the bead.

This subassembly is sealed with Scotchcast #8 to a stainless steel delay body containing an airgap, A-1A, Boron delay mix, A-1A and lead azide.



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PRIMER MK-148

Figure 1. Schematic drawing of MK-148.

This delay body has a reduced outside diameter creating an elongated groove filled with the epoxy resin. The assembly is then pressed into an outer cup, and the cup is crimped over the plug. The phenolic plug is a press fit slightly stretching and bulging the cup when it is pressed home, effectively sealing the unit with approximately 30 mg of freshly mixed fluid epoxy inside. The resin is a mixture of half and half "Parts A & B". Part B is largely amines, the major component being diethylene triamine.

In the Lot Acceptance Test (LAT) 87 units (previously subjected to Transportation & Aircraft Vibration but not Temperature & Humidity Cycling, T & H) were test fired, 1/3 each at hot, cold and ambient temperatures, all gave excellent functioning, delay and output results. The 2 of 8 units subjected to T & H cycling and which fired, also gave good delay and output data.¹

Circuit checks and Thermal Transient tests made of all the LAT samples before test firing showed all within specification resistance values and essentially unchanged by environmental conditioning on bridge wire to bead contact.

The four T & H samples which failed to fire showed open circuits after seeing the test pulse, indicating the wire had "burned" but failed to ignite

the NLS bead. Helium leak tests indicated that none of the failed units were leakers.

In view of this the failures must be associated with something built into the primer, enhanced by the prolonged high temperature storage, since failures to fire did not occur with the balance of the lot acceptance samples. These had not been so heated, and we doubted the degradation resulted from the low temperature part of the T & H cycle.

Careful dissection of the styphnate beads from two of the failed primers disclosed a charred tunnel through the bead resulting from burn out of the nichrome bridge wire, such that had the material in contact with the bridge wire been NLS it would have fired. The uncharred bead material was found to be dark brown rather than the lemon yellow usually associated with NLS beads.

LITERATURE STUDY

The following quotation from Blay and Dunstan (1970)² is appropriate:—

"Many ingredients of pyrotechnics and primary explosives are fairly reactive compounds and as such are susceptible to chemical incompatibility with the other materials with which they have to be used.....

"The need for compatibility testing applies not only to materials and explosives which are used in actual contact, but also to materials and explosives which are used in assemblies where no impermeable barrier exists to protect the explosives from vapor contamination. Primary explosives and, in some instances, pyrotechnics are particularly likely to be affected because of the small amounts which are used, often in proximity to much larger quantities of other materials from which reactive vapors and water may be evolved during storage."

Several reports in the literature describe methods for disposal of lead styphnate. All involve use of alkali, usually sodium hydroxide or sodium carbonate.^{3,4}

A study of the literature yielded evidence that epoxy resins can cause decomposition of lead styphnate. Epoxy resins consist of two parts that, when mixed, harden to a permanent chemically crosslinked solid. Part A is a stable monomer capable of condensation polymerization when catalyzed by the addition of amines (Part B). The amines, so used, are low molecular weight and volatile, e.g., diethylene triamine; the literature documents conclusively that even ammonia will decompose lead styphnate. The general literature shows that the low molecular weight amines used in Part B of Scotchcast #8 are far more basic than ammonia. Diethylene triamine, for example, is 100 times more basic than ammonia.

Through the years the Navy has insisted, in the preparation of NOL 130 for use in their stab primers and detonators, on the use of basic lead styphnate, whereas the Army permits the use of normal lead styphnate.

This preference by the Navy is based upon the NOL findings reported⁵ in NOLM 10614, by Ward & Graff, in which they demonstrated that normal lead styphnate while initially more vigorous as an ingredient in NOL 130, was poor after exposure to ammonium ion, whereas NOL 130 made with basic lead

styphnate remained good to excellent, when similarly exposed.

The British have encountered the same problem with normal lead styphnate exposed to amines which are, like ammonia, strongly basic.^{6,7} They have in their designs prohibited the incorporation of amines in contact, even in the vapor phase, with most explosives.

More specifically, NAVORD Report 4359⁸ states: "Amine type curing agents may or may not produce systems that are compatible with explosives, and are generally incompatible in the uncombined state. In the case of the crosslinked resin, it is believed that this explosive instability is due mainly to the presence of uncombined amine, but it may also be due to the amino groups in the crosslinked polymers. This instability of explosives in the presence of amines is due to the basicity of the amine as well as the strong oxidation reduction potentials setup between the nitro groups of the explosive and amino groups".

NAVORD Report 6023⁸ associates the incompatibility of amines (in epoxy resins) with explosives to their basicity.

EXPERIMENTAL:

These findings suggested a study of the effect of Scotchcast #8 epoxy on the normal lead styphnate used to produce the MK-148 and on the NLS beads. Part B of the epoxy mix has a strong ammoniacal and amine odor. Moist litmus held in the vapor over Part B immediately turns blue indicating high alkalinity. NLS, examined microscopically, shows large, well-formed, clear yellow crystals (Figure 2). On exposure to the mixed resin, even at room temperature the crystals become dark; the molecules are destroyed. When this is repeated at 160°F the changes are much more rapid. In a few hours the crystals are no longer identifiable microscopically as NLS (Figure 3).

These decomposed NLS crystals were compared optically with crystals from a failed MK-148 bead. Both show very fine, dark, formless, almost amorphous particles. This suggested further tests to characterize and compare these samples. First, a standard hotstage procedure was used to determine the thermal stability of several NLS samples: 1) pure NLS, 2) NLS heated 2 hours in mixed epoxy vapor, 3) NLS heated 16 hours in mixed epoxy vapor, 4) NLS from a failed MK-148 bead and 5) NLS from an unfired but T & H tested MK-148 (Figure 4). These curves measure the amount of NLS remaining at the temperatures shown during 10°C/min. heating. The intensity of the light transmitted by the crystals of NLS when viewed between crossed polars is a measure of the amount of NLS remaining in the sample. The 16-hour sample and the two beads contain less NLS at the start and hence disappear entirely at a lower temperature. The appearance of these samples at the end of a heating experiment in epoxy vapor is shown in Figure 5. The epoxy exposure tests carried out a little longer than above result in a similar product.

To confirm these changes by a different method we used powder x-ray diffraction (XRD). Four samples chosen from those shown in Figure 4 were x-rayed with results summarized schematically in Figure 6. The results show that the crystals are destroyed and that new unidentified compounds are formed.



Figure 2. Clear well-formed NLS crystals as used in MK-148.



Figure 3. NLS after exposure to epoxy resin at 160°F for two hours during cure.

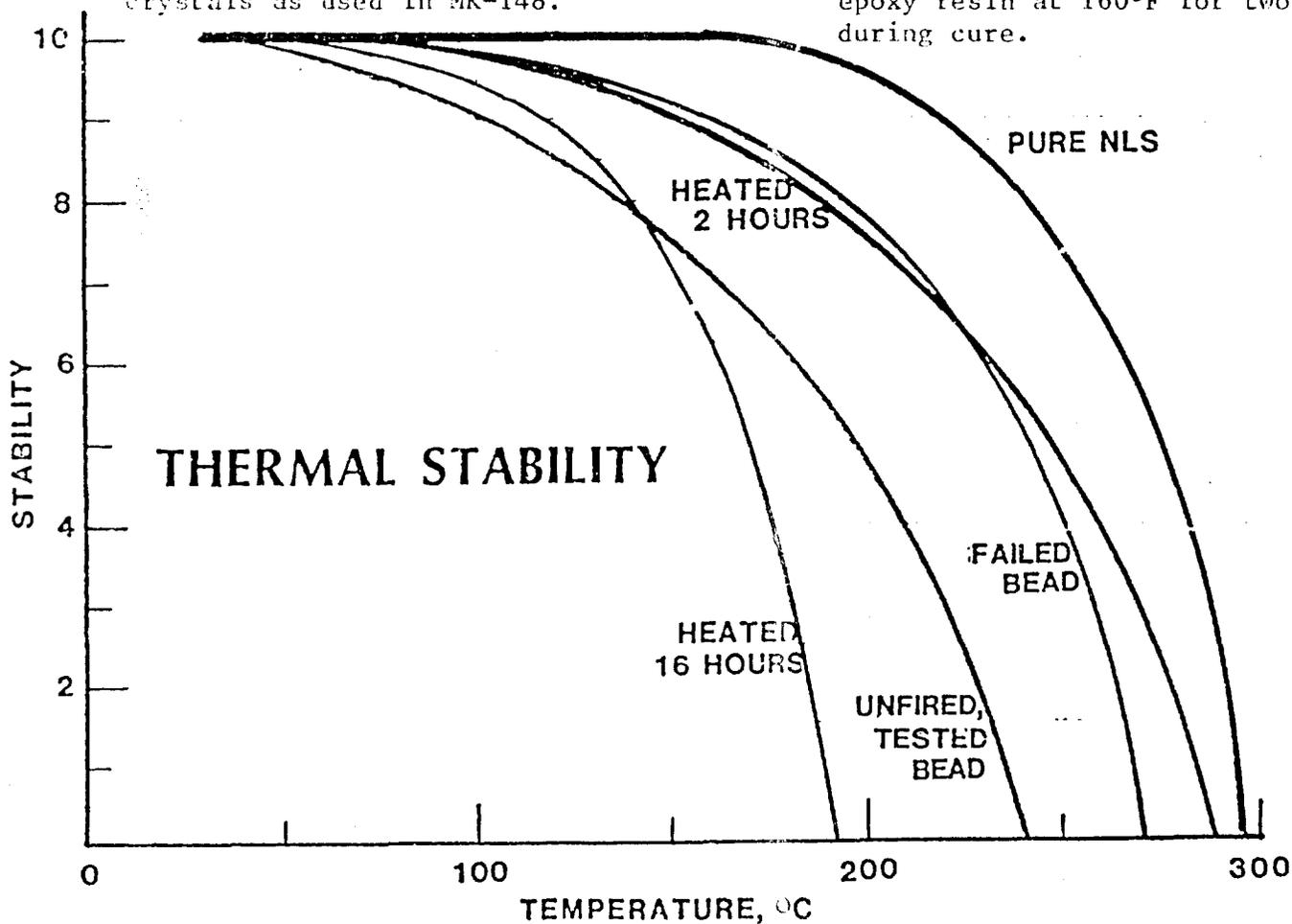


Figure 4. Thermal stability of NLS.



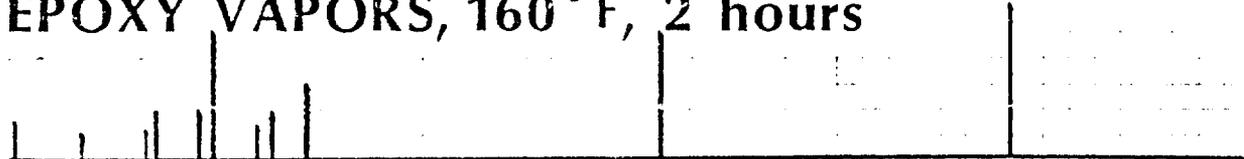
Figure 5. Final appearance of NLS after stability test.

X-RAY DIFFRACTION DATA

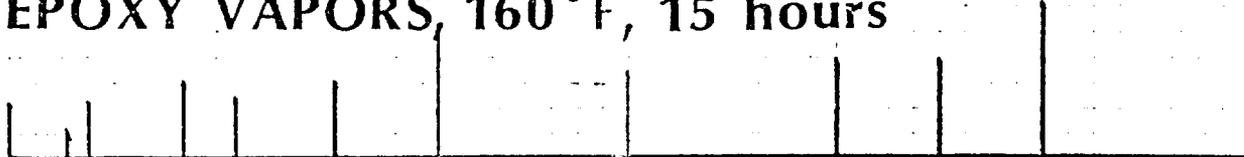
LEAD STYPHNATE (PURE)



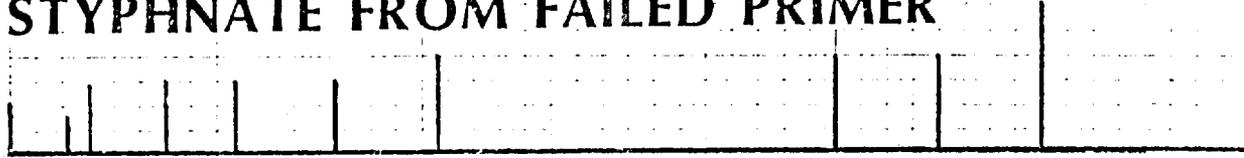
EPOXY VAPORS, 160° F, 2 hours



EPOXY VAPORS, 160° F, 15 hours



STYPHNATE FROM FAILED PRIMER



3 4 5 6 7 8
d-SPACINGS

Figure 6. Powder XRD data.

We also compared basic lead styphnate (BLS) with NLS in terms of stability to epoxy. BLS is far more stable.* Beads of BLS/lacquer and NLS/lacquer were prepared according to specifications and applied to glass microscope slides. The bottom surfaces could then be readily studied microscopically during exposure to mixed epoxy vapors at 160°F. Figure 7 shows the NLS bead with strongest decomposition near the outer surface where the amine concentration was highest. (The pattern in the center is the result of diffusion of amine along an air path between the glass slide and the NLS.) The same conditions (4 hours exposure) had relatively little effect on the BLS bead (Figure 8).



Figure 7. A bead of NLS/lacquer cast on a microscope slide and exposed to epoxy vapors.

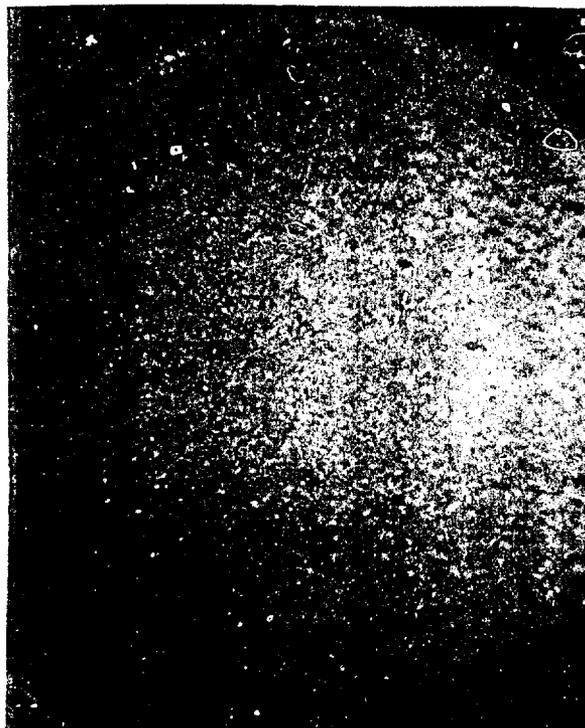


Figure 8. Here a basic LS bead was prepared and tested.

CONCLUSIONS

We can conclude that amine (low temperature-cured) epoxy resins should not be used with styphnate-loaded devices. Even though the high temperature and humidity test cycle is "killing" the MK-148 we cannot conclude that epoxy would be suitable if that test were eliminated or rendered less traumatic. The effect of the amines in the epoxy will still act on, and decompose, the NLS over a longer period of time at room temperature.

Basic lead styphnate is more stable to amine vapors than NLS but, again, we could not recommend its use in the bridge-wire bead. All compo-

*The black and white figures shown here are much less effective in showing the decomposition of the NLS and BLS in Figures 2, 3, 5, 7 & 8. BLS appears in black and white to be less stable (darker in color) than the original color photomicrograph. Anyone wishing to have a set of color prints of these five figures should send a check for five dollars to one of the authors (WCM).

nents showing any degree of chemical incompatibility with lead styphnate should be replaced.

We recommend that Scotchcast #8 resin be replaced with a styphnate-compatible adhesive.

ACKNOWLEDGEMENT

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