Lawrence Livermore Laboratory

STEMMING OPTIONS AND THEIR EFFECT ON CONTAINMENT

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STEMMING OPTIONS AND THEIR EFFECT ON CONTAINMENT

ABSTRACT

Stemming plans can be divided into either layered or continuous forms. There are marked differences between standards for these forms. The materials used in the continuous plan must meet more stringent specifications; as a consequence, they are more expensive. Both plans have been successful since Baneberry. The general philosophy for both plans is based on the following procedures:

- Provide a plug sufficient to match the overburden density of earth.
- Contain radioactive gases at the lowest possible depth.
- Minimize the generation of noncondensable gases (such as CO_2).
- Minimize gas flow rates during the early post-detonation stages when cavity pressure is high.
- Be compatible with the experiment and its diagnostics.

All commonly used materials reach the necessary overburden density when emplaced without large voids. The bulk density of granular stemming material is a function of particle size, water content, consolidation time, and rate of emplacement. The slower the rate, the shorter the time to reach a given density. No material completely consolidates during practical time limits of interest, but larger grain sizes approach consolidation faster than fines.

Overton sand at the lower (experimental) end of the stemmed column provides a cavity-sealing material relatively free of CO_2 -forming compounds at the cavity radius during cavity formation.

If applied rigorously, LLL standards and specifications will provide an emplaced permeability of 1.5 to 2.0 D, which approximates earth permeability at the Nevada Test Site (NTS). This low permeability allows cavity pressure to decay to ambient before a gas front advances any significant distance.

Low-yield conditions may allow stemming to fall into the cavity. Coal-tar-epoxy (CTE) plugs are redundantly emplaced at appropriate elevations both as support platforms for the stemming and as gas seals.

If bridging is considered an undesirable circumstance, then the tendency to bridge is a measure of undesirability. Large grain size, high moisture, and high stemming rates all increase the chance of bridging.

Pipe motion and stemming loads are directly affected by the choice of stemming material. The material's density, cohesion, internal angle of friction, and consolidation time can influence both the time and position of pipe closure due to ground shock, and the pipe response below the pinhole assembly.

Slumps (rapid consolidation) have often been associated with cable failure. Recorded data do not support this conclusion. Cables do not generally fail as a direct result of slumping unless a slump or consolidation occurs during plug solidification when a plug slips.

Cables are a potential channel of flow through the stemming. They must be gas-blocked both internally and by stemming the cable-bundle interstices -- the latter currently done through the use of fan-outs.

INTRODUCTION

The development of an acceptable containment plan for each nuclear explosion requires an understanding of the phenomena involved, a data base on materials and geology, and a clear idea of what containment is supposed to do. Over a period of 17 years, LLL has developed containment procedures that include a stemming plan or stemming philosophy described as follows:

- Provide a plug sufficient to match the overburden density of earth.
- Contain radioactive gases at the lowest possible depth.
- Minimize the generation of noncondensable gases.
- Minimize gas flow rates during the early post-detonation stages when cavity pressure is high.
- Be compatible with the experiment and its diagnostics.

This philosophy will possibly lead different groups to different, acceptable solutions for stemming requirements as a result of trade-offs within these general procedures. The purpose of this report is to present reasons for the adoption of LLL's continuous stemming plan (Fig. 1).

This report is a synopsis of many internal reports and many years of experience. Some of these reports are noted as references; Appendix C is a detailed listing of relevant LLL documents.

Fig. 1. LLL standard continuous stemming plan for pinex experiment.

BRIEF HISTORY

The present-day method of backfilling emplacement holes has evolved from the practice of simply filling them with the material removed during drilling. This evolution has been prompted by increased concern for the containment of radioactive products. Figure 2 is a chronology of important containment advances.

The first formal stemming plans used during the period from 1962 to 1965 used pea gravel as backfill with grout or concrete plugs in the emplacement hole. From 1965 to 1967 many variations in the layering of materials were used, which included layers of pea gravel, overton sand, NTS fines, and, on some occasions, grout. By 1967 the use of grout plugs was discontinued because of temperature effects on equipment and cables during exothermic curing, and the layered stemming plan became more formalized. This plan used layers of NTS fines between layers of pea gravel. The emplacement hole also contained layers of overton sand at the top and near the bottom. The hole was filled with 10-20% overton sand, 30% NTS fines, and 60% pea gravel. This plan was used until 1971. Between 1967 and 1971 the use of epoxy and tar plugs for stemming platforms and sealing was begun. In 1970 LLL commenced the practice of fanning-out cables at specific depths to allow stemming material to fill the voids between cables.

In 1971 the stemming plan was modified to the basic form used today. Cables were gas-blocked in the epoxy plugs to prevent gas leaks through cables. Also LLL mix was introduced as a low-permeability backfill material. This mixture contains 5 to 7.5% flyash (pozzmix), and the remainder is Area 1 material. Area 1 material, which ranges in size from half-inch stones to silt, is native material obtained from the playas of the NTS Area 1.

STEMMING MATERIALS AND STANDARDS

Contractors prepare the emplacement stemming materials according to LLL specifications.¹ The granular materials are tested for conformance to specifications both in stockpile and at emplacement. The moisture content in stockpile is particularly monitored (NS 76-5-5, Ref. 1), and the temperature at emplacement is recorded.

Appendices A1-A3 contain NS 76-7-4 ("Backfilling Emplacement Holes"), NS 76-7-5 ("Stemming Procedure"), and NS 76-3-2 ("Standard for Polymer Plugs").

Fig. 2. Chronology of important containment advances.

These documents represent current practice, but they are continually upgraded as required. Appendix D contains material specifications currently in use.

Polymeric CTE plug materials are selected for specific performance required (gas seal, platform, etc.) and then mixed immediately prior to use. Table 1 identifies some of these formulas and describes their final state of hardness. The pea gravel in the CTE mix is a convenient heat sink and filler that is already available as a stemming material.

Basic testing on these polymeric plugs is conducted on a gallon sample per batch basis. Gel time, shear strength, and flexural properties are determined for each sample. Before stemming can be resumed, a "go/no go" gel time and shear-adhesion test is made and reported.

The stemming plan prescribes waiting periods between the stemming and plug-pouring operations to ensure consolidation of stemming material prior to pouring plastic plugs. A waiting period after the pour allows the plastic to achieve the minimum acceptable structural capability before the next stemming phase begins.

STEMMING PLANS AND POROUS FLOW

The development of LLL's stemming plan closely followed increasing concern for leakage. Until Baneberry, major developments resulted from specific needs demonstrated at previous events. Since Baneberry, both LASL and LLL have developed different stemming plans; both have prevented any leakage to date. A major difference between the two plans is the effective permeability of the column stemming. Figure 1 shows the LLL plan. Figure 3 shows the LASL plan.

From a containment viewpoint, a measure of stemming effectiveness is the pressure at any station along the stemming columns as a function of time as a result of cavity-pressure forced flow. Pressure at any station depends on rate of flow through the porous stemming and can be calculated. The stemming material's effectiveness in restricting flow can be determined either by testing or by solving the appropriate analytical equations.²⁻⁷ The relative effectiveness of different plans can be judged by evaluating and comparing their constituent particle properties, particle size, distribution, packing, moisture content, and density. If the material properties are known, porous-flow calculations can be made that describe the pressure-flow relationship with time at known stations. The accuracy and corrections of these

TABLE 1. Modified epoxy material and formula requirements for stemming plugs.

Material	Formula Identification													
	LAE-59	LAE-59M	LAE-59MT	LAE-59MY	LAE-59P	LAE-59Y	LAE-59Z	LAE-60	LC-204	LC-204H	LC-204P	NB-104	NB-104.	
	Formula, wt%													
MEL 71-500060D	29.630	31.915	25.478	25.210	39.344	23.762	26.374	33.802	30.372	32.424	30.341	29.925	30.000	
MEL 76-500068	61.728	66.489	74.310	73.529	49.180	69.307	65.934	56.338	63.275	60.794	63.211	62.344	62.500	
MEL 76-500069	0.247				0.328	0.198	0.219	0.282	0.253	0.270	0.253	0.249	0.250	
MEL 76-500070	6.173	1.170		0.924	8.197	4.950	5.495	7.042						
MEL 76-500071									5.315	5.674	5.310	6.983	7.000	
MEL 74-500067	2.222	0.426	0.212	0.336	2.951	1.782	1.978	2.535	0.760	0.811	0.860			
MEL 76-500072									0.025	0.027	0.025	0.499	0.250	
Density, mg/m ³)	1.17	1.18	1.18	1.18	1.16	1.18	1.17	1.17	1.15	1.15	1.15	1.15	1.15	
MEL-76-001296	As Required													
Cured form	Hard	Soft	Soft	Soft	Very hard	Medium hard	Hard	Hard	Medium hard	Hard	Medium hard	Flexi-ble	Flexi-ble

Fig. 3. LASL standard layered stemming plan.

calculations for uncased holes, of course, also depends on the local geology -- in particular the in situ permeability, porosity, moisture content, and chemical composition.

Simple one-dimensional calculations solving the isothermal porous flow equation for noncondensable gases,

$$\frac{\partial}{\partial x} \left(\rho_l \frac{\partial P}{\partial x} \right) = \frac{c \mu \partial P}{K \partial t} ,$$

using a perfect gas and a 200-m cased emplacement column with constant driving pressure of 3.8 MPa for LLL and LASL permeabilities, give the results shown in Fig. 4. For points both 40 and 80 m up the column, the pressure front arrives nearly twice as fast in the LASL stemming.

Pressure vs time is plotted in Fig. 5 for one node of a two-dimensional calculation of porous flow done on an uncased 330-m emplacement column with decaying source pressure and with earth permeability as the variable parameter. As a measure of flow potential, these curves indicate the LLL plan with earth permeabilities of either 0.5 or 7 D is more desirable than the layered plan shown. Figure 5 also indicates the effects of a porous ($\epsilon = 0.35$) earth of high permeability: the ground reservoir damps the pressure excursion and lowers the flow potential.

Both LASL and LLL have performed experiments and field tests yielding data to correlate with and use in calculations of porous flow.⁸⁻¹⁰

Analytical techniques can account for the effects of condensation in the determination of pressure. A dramatic reduction in penetration is predicted.

BRIDGING

Bridging of stemming material should be avoided. Bridging is caused either by the stemming material's settling-out from under an arched, compacted, fixed section of material or by failure of the material to pass through a constriction. Bridging causes the formation of voids that reduce the emplaced stemming density and establish a potential for stemming slumps. Others have reported on bridging and have developed a set of equations establishing the relative potential for bridging as a function of material properties ($\gamma, c, c_w, \phi, \phi_w$), which are all a function of moisture.¹¹

Fig. 4. Pressure vs time at 40 and 80 m from cavity center at LLL and LASL.

$$D = \frac{c}{\gamma} \frac{\cos \phi}{(1 - \sin \phi)} \quad (\text{for cased holes}),$$

and

$$D = \left\{ \frac{c}{\gamma} B + \left[\left(B - \frac{w}{c} \right)^2 - \left(\frac{c_w \sin \phi}{c} \right)^2 \right]^{1/2} \right\} \quad (\text{for uncased holes}),$$

where B equals $\sin \phi_w \cos \phi_w$ ($[\cos \phi / 1 - \sin \phi] + [c_w \cot \phi_w / c]$), c is the cohesion factor, ϕ the angle of internal friction, γ the density, c_w the cohesion of the wall, ϕ_w the angle of friction at the wall, and D the maximum width of channel across which the static bridge will form if the appropriate values for the given materials are used.

The moisture content of emplaced materials thus is a critical parameter and must be kept at a very low level (3.5 wt% or less at LLL).¹² Figure 6 is an estimate of the plot of the above equations after substitution of material properties at appropriate moisture levels.

The emplacement of stemming material will cause air to be trapped within the stemming column, and this air will initially support some of the weight of the stemming material. Eventually, the air will diffuse up the hole or into the formation. This pressure buildup favors bridging, which can be detrimental when formed unexpectedly (thus blocking passage of additional material) or when stability is marginal and collapse unpredictable both in time and consequences. Thus, overton sand is preferable when experimental configurations result in narrow annuli.

Fig. 5. Two-dimensional calculation for a node located 190 ft from pressure source.

The solution to the problem of settlement or consolidation is complex. It can be expressed mathematically¹³ as a function of 12 independent materials and geometrical parameters and consequently is very difficult to present in any general form. Testing actual systems has yielded some information.

Figure 7 shows the air-pressure rise at depth for a stemming rate of 135 ton/hr for two materials. Pressure, depth, and rate are related by the equation

$$\Delta P \sim \frac{\text{rate} \times \text{depth}}{\text{area} \times \text{velocity}},$$

where the rate in question is the stemming rate, the depth that of the stemming interface, the area that of the emplacement annulus, and the velocity the terminal velocity of the material. For a given depth and velocity,

$$\Delta P \sim \frac{K_1 \times \text{rate}}{\text{area}}$$

And if ΔP_{max} is specified, then

$$\frac{\text{rate}}{\text{area}} \sim \Delta P_{\text{max}} / K_1 \quad (\text{slope of the stemming rate-area curve}).$$

Fig. 6. Estimated bridging width vs moisture.

Fig. 7. Pressure rise at constant stemming of 130 tons/hr.

The curves of Fig. 8 represent LLL rates of stemming to give less than a 2 psi pressure rise at a depth of 1000 ft in cased holes.¹⁴ The pressure rise in uncased holes of the same size and depth will be less, due to the leak off into the formation. The rate of consolidation is more rapid with more porous material.

The majority of slumps are in cased holes, indicating that pressure buildup might be a major factor. Table 2 lists 14 events for which data exist on bridging release (slumps). None of these slumps resulted in equipment or cable failure, although load changes to the emplacement string were as high as 20 klb (in the Carpetbag Event).

CHEMISTRY

The normal chemical behavior of stemming materials describes corrosion, shielding, reaction thermodynamics and heat transfer, and post-shot radiation chemistry. However, some members of the containment community think there exists a membrane (designated M³ for "Magic Mystical Membrane") at the cavity boundary that acts as a gas-sealing layer. This M³ could be a glass lining for the cavity with a zero-porosity compressed zone for some distance above it. If such a layer exists prior to collapse, it acts to seal the bottom of the stemming column and as such should be as gas tight as possible. A stemming material producing this seal would contain little or no reactive ingredients to produce noncondensable gases.

This conclusion leads LLL to use overton sand from a level adjacent to the working point to a level outside the cavity-seal membrane. Overton sand has a potential CO₂ content of 0.5 wt% as opposed to LLL mix at 3.5 and pea gravel as high as 7.

Often it is necessary to place material around the device to ensure the detection of trace elements of radiation chemistry or to more easily determine the post-event chemical balance.

Some sensitive cables and detectors require shielding from radiation. Gradations in shielding can be managed by selecting and emplacing materials such as magnetite, ulexite, and colmanite in locations and quantities as desired.

Fig. 8. Max stemming rates for depths to 1000 ft (for depths over 1000 ft, the max rate = $1000 \div \text{depth} \times \text{chart}$).

TABLE 2. Slumps recorded.

Event, date	Hole size, in.	Stemming			Load change, klb	Remarks
		Material	Rate, cons/hr	Depth, ft		
Starwort, 4-73	48, cased	LLL mix	100	1600	----	Minor slump
Carpetbag, 12-70	48, cased	Overton sand	150	1926	70	Heavy slump
Bracken, 4-71	86, uncased	LLL mix	120	541	----	Slump
Harebell, 6-71	48, uncased	Overton sand	120	1225	4.0	Slump
Commodore, 5-67	48, cased	Pea gravel	650	2450	----	Slump in 11-ft ² annulus
Greely, 12-66	48, cased	Overton sand	120	----	----	Slump
Lanpher, 10-67	48, cased	Overton sand Pea gravel Des. fines		0	----	40-ft slump, due to stemming pipe with steel balls
Baneberry, 12-70	86, uncased	Overton sand	325	----	----	Slump in 20-ft ² annulus
		Overton sand	567	----	----	Slump in 40-ft ² annulus
Faultless, 1-68	26, cased	Overton sand	40	----	----	Slump in 2.71-ft ² annulus
Cannikin, 11-71	10 3/4, emplaced casing	Pea gravel	75	----	----	Bridge broken by drill string from surface
Cheshire, 2-76	48 cased (wet bottom)	LLL mix	25-50	105	----	- 9-ft and 5-ft slumps - Cables parted at 1598 ft - Bottom of hole wet, due to leak
		Overton sand	23-41	2125	2.4	- Near top of overton sand
Muenster, 10-75	48, cased (with guides etc.)	LLL mix	16	3332	1.4	- 3-ft slump
		LLL mix	16	3349	1.0	- 17-ft slump
		LLL mix	18	2852	2.5	----
		LLL mix	60	2173	2.5	- 11-ft slump
		LLL mix	60	2024	5.8	- 15-ft slump
		LLL mix	50	1142	3.7	----
		LLL mix	50	851	3.8	----
		LLL mix	100	746	2.2	----
Colby, 3-76	48, cased	LLL mix	100	647	----	- 24-ft slump
		LLL mix	17	3247	----	Slump
		LLL mix	19	2722	----	18-ft slump
		Pea gravel	110	2430	----	5-ft slump
		LLL mix	17	2302	15	5-ft slump
		LLL mix	24	1660	15	49-ft slump
		LLL mix	30	1657	----	16-ft slump
Fontina, 2-76	48, cased (wet bottom)	LLL mix	30	1657	----	8-ft slump
		Overton sand	40	2060	15	Slump

PLASTIC PLUGS

Plastic plugs are used as stemming platforms and gas blocks in vertical shaft events. LLL's Standards Practices Manual for Nuclear Testing specifies the design and emplacement of these plugs. The standard design information and data are contained in "The Design and Evaluation of Plastic Stemming Plugs" (ENN 76-66, LLL, 1976),¹⁵ which includes prior design information (ENN 72-93, LLL, 1972) and new or additional data. At LLL, the predominant emplacement-hole configuration is shown in Fig. 1. It contains a 34-m-deep surface casing and is uncased below that depth. Present practice uses a combination platform and gas seal in the surface casing and a stemming platform in the uncased portion. Where ground motion is expected to cause deceleration forces from the stemming to damage the gas-seal plug, a rigid foam plug is emplaced between the stemming and plug to act as a shock absorber.¹⁶

The design of these plugs is complicated by many variables affecting strength and bonding characteristics:

- cure time,
- temperature during cure,
- temperature when loaded,
- temperature gradients,
- strain rates, and
- loading modes.

The development of acceptable plugs has included both theoretical^{17,18} and experimental approaches.¹⁹ The need for blocking radioactive gases is obvious. The need for stemming platforms only becomes apparent during analysis of "experiment" failure modes. A low-yield (dud) experiment can result in the stemming column's falling into the cavity. This effect can be minimized by placing platforms under some "minimum allowable" stemming volume.

MECHANICAL SURVIVAL -- EXPERIMENTAL COMPATIBILITY

Mechanical survival throughout the experiment's emplacement phase is assured by the application of proper procedures and practices. These require the calculation of loads on the emplaced and stemmed hardware. Stemming loads are calculated by LCSTEM,²⁰ a code that uses the properties of stemming

material in calculating the stemming loads on the emplaced system. This code also predicts pipe and canister strain (displacement) from loads induced by stemming. These loads can be reported at any particular depth and at the collar as a function of stemming depth. A maximum stress curve can be plotted vs depth. Correlation of measured data with LCSTEM predictions has been made on many events.²¹

Mechanical survival of the emplaced diagnostics past the times of normal prompt diagnostic reporting is required on pinex and other line-of-sight events. Pipe responses to high-energy disposition and ground shock are studied for each event, and equipment is placed at proper elevation and stemmed according to need.^{22,23} UDYN, a program to calculate the dynamic response of emplacement structures, is used to generate displacement, velocity, acceleration, and friction force vs time plots at nodes of interest.¹⁴ Stress and strain time histories and stress vs strain history are also plotted. Other specific load histories and peak-value histories can be plotted.

Plastic plugs may be used, for instance, to couple the pinex pipe to the hole wall (or casing) at the pinex plate station in order to eliminate relative pipe motion. This results in maintaining the integrity of the pipe above this station, making plate recovery possible.

The stemming, casing, and pipe strength determine the location of pipe closure due to ground shock below the pinhole. An example of the potential effects of stemming options is demonstrated by comparing the effect of pea gravel with that of sand for an analysis of the stress in and around the emplacement hole. Figure 9 depicts the peak stress in a hypothetical section of pipe, stemming, casing and grout-earth in response to a 1 Kbar force applied to a plane for 1 sec. Note the effective lowering of the location for peak casing stress when the sand is replaced by pea gravel. This suggests that stemming changes affect some things not directly related to stemming emplacement.

The success of predicting gross location of pipe closure has been demonstrated. Pipe instrumentation records the internal pressure below the pinhole and shows that normally this pipe structure is closed prior to gas leakage.

The success of experimental diagnostics depends upon the survival of the signal. Cables and detectors must be shielded with stemming materials. Good radiation shielding requires the filling of cracks or voids with proper materials. Sometimes overton or Monterey sand is enough. At other times lead

Fig. 9. Predicted peak stress in sand and pea gravel at a fixed depth.

shot, iron shot, magnetite, ulexite, colmanite, and many other materials must be used as radiation filters or device-performance modifiers.

In short, the placement procedures and effectiveness of a specific stemming material also depend on its special properties.

CABLES

Cables are potential modifiers of any stemming plan, and their survival depends on the choice and application of stemming materials. The loading induced on a cable by stemming is partly a function of the stemming material's bulk density and angle of internal friction and of course is different for each material used. It is also a function of the area of cable in contact and the cable position in the hole relative both to the emplacement string and to the casing.

The bulk properties of stemming materials also vary with time from emplacement;¹³ total cable loads therefore also vary with time. As the entrapped gas and differential pressure tend to zero, the lateral stemming force rises because of the increase in bulk density and, thus, adds load to the cable system.

LLL has tested materials and cables to obtain the necessary data with which to calculate loads on emplaced equipment (see Appendix B) and will continue to do so.

Cables individually represent flow paths of low resistance unless they are gas-blocked. A continuously gas-blocked cable is most desirable, but other cables are specifically gas-blocked at least in the two upper plastic plugs.

Cable interstices must be considered channel-flow passages. These passages are filled if fine, dry stemming material is used. To ensure an additional porous flow block, fan-outs or cable-separation lengths are included in each emplacement for a length sufficient to provide the resistance required to prevent cable-channel flow. Fan-outs position the cables radially into the stemming, increasing the load on them. The load is a function of the cable location between the emplacement system and the hole casing and will be a maximum about half way between.²¹ This effect was monitored on the Labis event.²⁴ The load was measured as a function of position and was found to vary by a factor of up to three. These measurements indicated a higher load on cables located further out in the stemming material.

Fan-out length is determined by calculating the minimum distance in the medium that will experience no pressure rise from condensable gas flow during the time of interest (cavity-pressure decay times). This distance is then multiplied by an appropriate factor of safety.

SUMMARY AND CONCLUSIONS

Stemming plans can be divided into either layered or continuous forms. Both plans have been successful since Baneberry.

All commonly used materials reach the necessary overburden density when emplaced without large voids. The bulk density of granular stemming material is a function of particle size, water content, consolidation time, and rate of emplacement. The slower the rate, the shorter the time to reach a given density; larger grain sizes consolidate faster than fines.

Overton sand at the lower end of the stemmed column provides a cavity-sealing material relatively free of CO₂-forming compounds and thus minimizes the generation of noncondensable gases.

If applied rigorously, LLL's standards and specifications will provide an emplaced permeability of 1.5 to 2.0 D, which approximates earth permeability at NTS. This low permeability allows cavity pressure to decay to ambient before a gas front advances any significant distance.

Low-yield conditions may allow stemming to fall into the cavity. CTE plugs are redundantly emplaced at appropriate depths both as support platforms for the stemming and as gas seals.

The stemming material's small grain size, low moisture content, and low rate of emplacement reduce the chance of bridging. The choice of material also affects pipe motion and stemming load. The density, cohesion, internal angle of friction, and consolidation time of the material influence both the time and position of pipe closure due to ground shock, and the pipe response below the pinhole assembly.

Recorded data do not support the conclusion associating slumps with cable failure. Cables do not generally fail as a direct result of slumping unless a slump or consolidation occurs during plug solidification when a plug slips.

Cables are a potential channel of flow through the stemming. They must be gas-blocked both internally and by stemming the cable-bundle interstices. LLL currently uses fan-outs to accomplish the latter.

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21. A. Blake, LLL, Internal Document ENN 75-31 (1975).

22. M. Hartzman and J. Staats, LLL, Internal Document ENN-72-101 (1972).
23. M. Zaslowsky, LLL, Internal Document (to be published, 1976).
24. B. White to D. Simms, "Labis Cable Stemming Load," LLL, private communication (May 6, 1970).

APPENDIX A

- A1. Backfilling Emplacement Holes
- A2. Stemming Procedure
- A3. Standard for Polymer Plugs

February 10, 1976

Prepared: R. J. Horton
Approved: Victor N. Karpenko

BACKFILLING EMPLACEMENT HOLES

This standard establishes maximum stemming rates for commonly used back-fill material, a wait time at the polymer plug stemming material interface prior to emplacing plugs, and a wait time after polymer plug emplacement.

These criteria will apply to all nuclear events conducted by LLL and shall not be modified nor altered in any way, without prior approval of Nuclear Test Engineering Division or included as a part of Mechanical Engineering stemming and containment drawings.

Stemming Rates

The maximum allowable stemming rates were derived empirically from field observation and instrumented configurations. They depend strongly on particle size distribution, particle velocity, and net area of annulus.

The maximum allowable rates for the various materials used by LLL is presented in graph form in Appendix I for hole depths between 100 and 1000 ft deep. For deeper holes, the value from the graph in Appendix I must be adjusted by multiplying the allowable rate by the expression $\frac{1000}{D}$ where D = depth of hole.

For hole depths less than 1000 ft no multiplier shall be used, except that the depth above the top polymer plug, if less than 100 ft, may be stemmed at the maximum capacity of the stemming equipment.

Caution

When selecting the net annulus (A_N) from the graph, attention must be given to geometric obstructions in the emplacement hole, i.e., canister, drag plates, cable bundles, etc. The annulus is to be; that area available for the stemming material to pass through. Therefore, in some zones with such constructions either in them or above them the allowable stemming rate may be less than the minimum attainable with the equipment being used. In these circumstances, the maximum rate per hour must be achieved by intermittent stemming (select reasonable time increments nearly equal, i.e., 15 stem, 15 wait, etc.).

Residence Time Before Column Loading

Experience indicates that stemming settles after it is emplaced, therefore, after stemming up to the level for locating a polymer plug a "wait time" of eight (8) hours shall be allowed prior to pouring the polymer. In addition, a "wait time" of ten (10) hours shall be allowed after the first (structural layer) of plug is poured before additional stemming or polymer loads are imposed. Also, samples representing the plug must be tested and accepted before loading (see NS 76-3-2).

These "wait times" must ensure that no activity takes place in the emplacement hole during the last four hours of the eight hour wait time prior to pouring the polymer plug and for the entire ten hour period. In addition, the ten hour wait time for polymer plug cure must be free from disturbance by other nuclear events.

Reference Documents

1. LLL Mechanical Engineering Specification MEL 75-001282, 10/17/75, Specification for LLL Stemming Mix II.
2. LLL Mechanical Engineering Specification MEL 76-001292, Specification for Overton Sand.
3. LLL Mechanical Engineering Specification MEL 76-001296, Specification for Gravel for Use in Polymer Plugs.
4. LLL Mechanical Engineering Stemming and Containment Drawings for Specific Events.
5. ANS 4-3574, 2/17/70, Stemming Material Velocity, Add-on Experiment, Preliminary Report.
6. ENN 69-24, Damage to Instrumentation and Cabling in an Emplacement Hole During Stemming.
7. ENN 70-23, Terminal Velocity of Stemming Material.

PRESSURE INCREASE IN CASED HOLE APPENDIX I PG. 2

February 19, 1976

Prepared: R. J. Horton

Approved: Victor N. Karpenko

STEMMING PROCEDURE

This procedure will describe a standard stemming operation including specifications and standards which apply to the operation.

I. Stemming Plan

Stemming of emplacement holes begins with a stemming plan designed by the Containment Group at LLL. This plan is included in the Containment Prospectus and reviewed by the CEP for the event under consideration. After this review (sometimes concurrent or in advance), an engineering drawing is released from which LLL's Field Engineering Group (LLL-N E&C) obtains criteria and materials required for implementing the plan. A copy of this drawing is also sent to H&N so they have the information from which the polymer plug dimensions, tolerances and materials can be derived and a polymer plug pouring schedule established. In this regard, H&N is required to coordinate with the responsible E&C Field Engineer for the support in equipment and manpower necessary to batch, transport, mix and emplace the polymer - gravel ingredients of the plug - all done under the surveillance of LLL's Containment Group.

This engineering drawing also provides electrical cable gas block locations and is further used to determine material procurement and stockpile requirements.

II. Emplacement of Material

Emplacement holes are generally backfilled with Ulexite around the canister containing the explosive, Magnetite around the diagnostic canister, Overton sand several hundred feet above the Magnetite and LLL Mix (MEL 75-001282) to the surface except for the zones containing polymer plugs.

Because of the usually narrow annular areas around the canister, Ulexite and magnetite are emplaced at a very slow rate, like NTS Fines, either by hand pouring from bags, or through the conveyor system at intermittent feed times. The Overton sand and LLL mix is usually emplaced directly from a stockpile which has been sampled for moisture content and, in the case of LLL mix, for particle size distribution.

If qualified, the material is brought from the stockpile in front loading scoop rigs. Each scoop load is elevated and dumped into a large, approximately 18 cu. yd., capacity hopper. The hopper has a control gate at the bottom and screen over the top which prevents large items from entering the system.

This hopper is mounted over the low end of a conveyor belt which transports the material at about a constant rate up and into another hopper. This second hopper is part of a material weighing machine called a weightometer. It continuously weighs and transports the material to the hole on a small

conveyor belt. There the native material is blended with Pozzmix to make LLL Mix. Data presented by the machine is the instantaneous rate in tons per hour and cumulative tons placed in the hole. From the information presented, knowing the hole geometry, and periodic tagging of the stemming surface on an on-going basis, in-place density calculations are performed and adjustments made to stemming rates if necessary to stay within acceptable limits. An overall summary of the in-place density is made available for the D-1 briefing.

When the elevation to emplace a polymer plug is approached, stemming is stopped, the surface is tagged and the level adjusted to provide a few feet of gravel for the polymer to contact. After an eight hour "wait" time the hole is ready to receive a polymer plug.

Polymer plugs consist of a mixture of coal tar, epoxy, hardeners, reactants, and gravel. Thus, about 40% of each plug volume is coal tar-epoxy (CTE) discharged into a full volume of gravel.

Two methods of plug emplacement are used. (1) Free fall or pour from the surface for depths <500 feet, and (2) by pumping with Halliburton pumper truck through a tremie pipe for depths >500 feet.

For the plug which is poured from the surface (<500 feet), a portion, about 1/3, of the gravel is added to the liquid CTE in an in-transit mixer truck which is backed to the hole collar and the mixture dumped much like concrete. The balance of the gravel (2/3) is simultaneously discharged into the hole by the conveyor system.

For the plug which is pumped through a tremie pipe (>500 feet), the pipe is positioned about 15 feet above the plug location and the gravel comes off the conveyor in free fall. Detectors are mounted on the emplacement casing to signal the arrival of the CTE; thus, ensuring a simultaneous emplacement of gravel and CTE.

This description of a CTE plug and its emplacement fits both a hard (LAE-59) and a soft (LAE-59 MY) plug. Other designations and formulations are used, however, they all fall into these general descriptions.

An operational limitation which is imposed after pouring a hard plug is a 10 hour "wait" time before additional loads are placed on the plug. This provides time for the plug to cure and obtain its structural characteristics. This is also a quiet time whereby no work is permitted in the hole that could cause vibration to the system. After the plug cures, stemming proceeds in accordance with the stemming plan previously described.

On some events which produce high slapdown forces, a plug consisting of sand and rigid plastic foam cubes is emplaced in the surface conductor to reduce the load on the hard plug located in the same conductor. The composition is built-up out of 15 layers of combined foam cubes and Overton sand. Each layer is emplaced (usually <100 feet) by first dropping the foam cubes then adding the sand. Every effort is made to

distribute both the cubes and the sand evenly over the conductor area. After the last layer of cubes and sand has been emplaced, several feet of gravel is added to provide a foundation for a soft (LAE-59MY) sealing plug.

III. Material Specifications

The following specifications apply to the material used to backfill emplacement holes:

1. MEL 75-001282, Specification for LLL Stemming Mix II
2. MEL 76-001292, Specification for Overton Sand
3. MEL 76-001296, Specification for Gravel
4. NS 76-3-2 , Standard for Coal Tar Epoxy
Formulation Requirements for Stemming Plugs
5. MEL 71-500060C, Specification for Epoxy Resin
6. MEL 76-500068, Specification for Refined Coal Tar
7. MEL 76-500069, Specification for Silane Coupling Agent
8. MEL 76-500070, Specification for Epoxy Curing Agent,
Mercaptan Type
9. MEL 74-500067, Specification for Curing Agent (DETA)
10. MEL 76-500071, Specification for Epoxy Curing Agent, Ketimine Type
11. MEL 76-001300, Specification - Diagnostic Material Requirements -
Magnetite
12. MEL 001299, Specification - Diagnostic Material Requirements - Ulexite

IV. Materials Testing

A. CTE Plugs

CTE hard plug material is tested in accordance with the following specifications:

1. NS-76-5-2 Coal Tar Epoxy Stemming Compound; Gel-time and Exotherm Time Testing at Elevated Temperature
2. NS-76-5-4 Beam Fracture (Flexural) Test for Stemming Compounds
3. NS-76-5-3 Cylindrical Wall Shear Test for CTE Compounds

B. Granular Materials

During the entire stemming operation, the quality of the emplaced stemming materials is controlled by maintaining the following parameters within specified limits:

1. Moisture Content: A statistical control chart, derived from experimentally measured stockpile moisture distribution, is used to detect significant moisture increases in stemming samples taken off the conveyor belt.¹

The sampling frequency is such as to give a moisture measurement for every four diameters of stemmed hole. Thus, the moisture content of the entire stemmed hole is kept at an acceptable level (i.e., <3% nominal) to a high degree of confidence, and any local variations can be detected and corrected before a significant amount of wet stemming is in the hole.

2. Loose and Rodded Stemming Density: A density sample is taken off the conveyor belt every 50 tons of stemming. Both loose and rodded densities are measured and are used to evaluate the emplaced density of the stemming column. Thus, the stemming rate may be slowed down to below the standard value, if it is discovered that the stemming gives a density that is too low (i.e., that has a high slump potential).
3. Sieve Analysis: The same sample as is used for density measurements is used to measure the particle distribution in the stemming being used. Any deviations from LLL-II specification will be reported to the Containment Engineer for on-the-spot judgment (i.e., accept, change fly-ash proportion to return to LLL standards, or reject the lot).

V. Rates of Backfill

Granular materials are emplaced at a rate not exceeding that described in NS 76-7-4. Those materials not shown on the graph (Magnetite and

¹ NS-76-5- Standard for Stockpile Testing

Ulexite) and emplaced at the rate allowable for LLL Stemming Mix. The rates can be adjusted downward depending on hole configuration, anticipated pressure buildup,² or other considerations where a more conservative stemming rate is prudent.

² ANS 3575

February 25, 1976

Prepared: P. Fleming

Approved: *Victor N. Karpenko*
Victor N. KarpenkoSTANDARD FOR POLYMER PLUGS

This standard covers materials, formulations and testing of polymer systems used as an adjunct of a stemming plan for a nuclear test system; namely, polymer plugs.

All materials constituting the polymer plug(s) must meet all requirements of the following specifications when used in the batching of plug formulations (Reference: Table I):

<u>Specification</u>	<u>Material</u>	<u>Function</u>
MEL 71-500060D	Modified Epoxy Resin	Base Resin
MEL 76-500068	Refined Coal Tar	Extender
MEL 76-500069	Silane	Coupling Agent
MEL 76-500070	Mercaptan	Accelerator
MEL 76-500071	Ketamine	Accelerator
MEL 74-500067	Diethylenetriamine	Hardener
MEL 76-500072	Glycerol Solution	Activator
MEL 76-001296	Overton Pea Gravel	Filler

The key to a successful plug design is the formulation of these several materials into a compound which, after exothermic reaction, will produce a plug with desired physical and mechanical properties. The overton pea gravel is used as a convenient filler. It adds strength and acts as a heat sink.

Formulation of materials for polymer plugs must be in strict accordance with this standard unless specifically and expressly modified by the originator of this standard.

Three basic tests shall be conducted on polymer compounds used for "hard" plugs (Table I) after batching and mixing operations are completed. These tests are to ensure the correctness of the formulation and the compounding. The tests shall be conducted as described in the following test specifications:

NS-76-5-2	Gel-time and exotherm time testing at elevated temperature
NS-76-5-3	Cylindrical wall-shear test for stemming plug compounds
NS-76-5-4	Beam fracture (flexural) test for stemming plug compounds

The results of the tests shall be reviewed and initialed by the LLL Containment Engineer, or his alternate, prior to resumption of stemming operations.

TABLE 1

MODIFIED EPOXY MATERIAL AND FORMULATION REQUIREMENTS FOR STEMMING PLUGS

MATERIAL	FORMULATION IDENTIFICATION												
	LAE-59	LAE-59M	LAE-59MT	LAE-59MY	LAE-59P	LAE-59Y	LAE-59Z	LAE-60	LC-204	LC-204H	LC-204P	NB-104	NB-104.5
	FORMULATION (WEIGHT %)												
MEL 71-500060D	29.630	31.915	25.478	25.210	39.344	23.762	26.374	33.802	30.372	32.424	30.341	29.925	30.000
MEL 76-500068	61.728	66.489	74.310	73.529	49.180	69.307	65.934	56.338	63.275	60.794	63.211	62.344	62.500
MEL 76-500069	0.247				0.328	0.198	0.219	0.282	0.253	0.270	0.253	0.249	0.250
MEL 76-500070	6.173	1.170		0.924	8.197	4.950	5.495	7.042					
MEL 76-500071									5.315	5.674	5.310	6.983	7.000
MEL 74-500067	2.222	0.426	0.212	0.336	2.951	1.782	1.978	2.535	0.760	0.811	0.860		
MEL 76-500072									0.025	0.027	0.025	0.499	0.250
DENSITY (Mg/m ³)	1.17	1.18	1.18	1.18	1.16	1.18	1.17	1.17	1.15	1.15	1.15	1.15	1.15
MEL 76-001296	AS REQUIRED												
CURED FORM	Hard	Soft	Soft	Soft	Very Hard	Medium Hard	Hard	Hard	Medium Hard	Hard	Medium Hard	Flexible	Flexible

APPENDIX B
REFERENCE LIST TO DOCUMENTS ON
STEMMING-MATERIAL PROPERTIES

APPENDIX B

The following table lists references pertaining to stemming material properties and effects. Most are internal LLL documents.

<u>Description</u>	<u>References</u>
1. Porosity, Bulk Density & Particle Size Distribution	UCRL 73607 (1972)
2. Poisson's Ratio, All Stemming Materials	NTED Materials Reference Table, M. Zaslowsky
3. Constrained Modulus Tests on All Stemming Materials (M , μ , σ)	NTED Materials Handbook M. Zaskawsky
4. Shear Stress vs. Normal Stress (Dry) (LLL Mix, Sand, LLL Mix with Pozmix)	1976 NTED Edition, UCRL 51934 (71) M. Zaslowsky
5. Plug Data: Confined Compression Tests	NTED Materials Handbook M. Zaslowsky
6. Plug Data: Mechanical Prop. Plugs	ENN 73-74 (1973)
7. Frictional Drag Tests, Pipe and Stemming Materials	NTED Materials Handbook M. Zaslowsky
8. Equation of State for Plug Material	1970 Memo, NTED Handbook
9. Design of Plugs	ENN 72-93 (1972)
10. Cycle Frictional Tests with Stemming Materials (all) (Static and Dynamic)	ENN 72-193
11. Lateral Pressure Criteria for Stemming Materials	ENN 72-138
12. Resistance Factor q_{Δ} Friction Factor f	UCRL 51034
13. Angle of Responce Stemming Materials	NTED Materials Handbook
14. Experimental Data on q_{Δ} , Soil Resistance Factor	ENN 73-149
15. Behavior of Diagnostic Cables as Related to Emplacement Pipe Slip Joint Movement	December 1, 1971 J. Uhlar & M. Zaslowsky
16. Hondo Calculations on Wet Sand - Casing - Cement - Tuff System. Also Dry Sand, Also with Slip Lines	M. Zaslowsky - LLL (unpublished) 1976

<u>Description</u>	<u>References</u>
17. Wave Propagation Studies Using Hondo - NTS Fine Overton Sand	M. Zaslowsky - LLL
18. Cable Pull Tests	1971 (at EG&G)
Cable Type Max. cable failure load at 75° and at temperature stress-strain curve	PEG 71-114
NV-CC-3	
NV-CC-8	
NV-RF-1	
NV-RF-4 (PLJ-5)	
NV-RF-4 (Foamflex)	
NV-RF-2 (NHJ-5)	
NV-RF-8 (THJ-5)	
19. Abrasion Tests - All Cables (1973)	NTED Materials Handbook M. Zaslowsky, et al.
20. Pressure & Temperature Testing NV-RF-1	1973, J. Uhlar
21. Rio Blanco Cable Support System	1972, M. Zaslowsky
22. ASE Cable - Analysis (Failure)	1973 NTED Materials Handbook M. Zaslowsky
23. Creep & Tensile Properties of RF1 and Rochester Cable. Tensile Property of RG81/V	1973, ENN 73-27
24. Tensile Test Data	NTED Materials Handbook M. Zaslowsky
RF-1	
RG81-V	
RG213	
25. Summary of Electrical & Mechanical Tensile Tests on Electrical Cables	NTED 69-2
RG213/V	
NV-RF-1	
26 Cond A & F	
30 Cond NV-CC-3	
NV-RF-4	
NV-RF-8	
26. Temperature Effects on Cable Data	1968 Memos: Data NTED Materials Handbook M. Zaslowsky
27. Cable Abrasion Tests, Review of All Cables	1968 NTED Materials Handbook J. Uhlar
28. Horizontal Creep Tests on Coaxial Cables	1970, M. Zaslowsky

<u>Description</u>	<u>References</u>
29. Properties of Alluvium Rate Effects	NTED Materials Handbook R. Dong, M. Zaslowsky
30. Triaxial Properties of Alluvium	Brandt Data
31. Moisture Content and Particle Size Distribution of Alluvium	1973 NTED Materials Handbook
32. Confined Compression Tests on Alluvium	NTED Materials Handbook R. Dong, M. Zaslowsky
33. Theoretical Relationship Correlation Between Mechanical Properties and Particle Size	S. Cowin M. Zaslowsky's private file 1972
34. Flow of Stemming Materials (Theoretical Study)	S. Cowin, 1976
35. Relationship Between Cased and Uncased Holes, Wet & Dry Holes, Bridging	M. Zaslowsky's private file 1976

APPENDIX C
REFERENCE LIST TO DOCUMENTS
ON STEMMING AND ITS EFFECTS

APPENDIX C

The following list represents many of the LLL documents on stemming and its effects.

1. Pitts, J. H., ENN 71-151, The Increase of Pressure with Time Under the Plastic Sealing Plugs on Pamassia, October 18, 1971.
2. Gleason, H. C., ENN 72-34, Delayed Hardening Stemming Materials, February 16, 1972.
3. Benedix, C. P., ENN 72-36, Dianthus Downhole and Stemming Loads, February 22, 1972.
4. Pitts, J. H., ENN 72-142, Feasibility of Utilizing Airjets to Reduce the Terminal Velocity of Overton Rock (Dave = 7/8"), August 31, 1972
5. Holl, P. M., ENN 73-901, Mechanical Engineering Safety Note: 2-Phase Flow Test Experiment, January 1, 1973.
6. Watling, H. E., ENN 75-60, Camembert LC204 Stemming Platform, June 9, 1975.
7. Dow, J. P., ENN 70-45, Stemming Blow-Out From Cavity Pressure, June 30, 1970.
8. Morrison, F. A., ENN 70-63, Transient Gas Flow in a Porous Column, September 23, 1970.
9. Norris, D. M. and J. R. Staats, ENN 70-85, Dynamic Loads on Grouted Emplacement Systems, UCID-25994, December 1970.
10. Pitts, J. H., ENN 71-06, A Mechanism for Venting of Underground Nuclear Explosions, January 11, 1971.
11. Pitts, J. H. and W. Kleck, ENN 71-101, Permeability and Porosity of Some Stemming Materials, UCID-15876, June 21, 1971.
12. Pitts, J. H., ENN 71-128, The Buckley-Leverett Equation and Its Solution to Predict Quasi-Static Flow Characteristics in Porous Media or to Obtain Relative Permeability, July 20, 1971.
13. Blake, A., ENN 72-59, Strength of Underground Cavities, March 13, 1972.
14. Pitts, J. H., ENN 72-70, Seepage Through the Backfill on Chaenactis, April 12, 1972.
15. Gleason, H. C., ENN 72-81, Final Report - Second Batch Delayed-Setting Cement, June 1, 1972.
16. House, P. A., ENN 72-93, The Design of Plastic Stemming Plugs, September 18, 1972.
17. Pitts, J. H., ENN 72-107, The Status of NTED Effort on the Problem of Fluid Flow Through Porous Materials, June 27, 1972.

18. Pitts, J. H., ENN 72-110, Fluid Pressure Required to Emplace the Lower Plastic Plugs on the Flax Experiment, July 12, 1972, UCID-16085.
19. Blake, A., ENN 72-138, "Review of Lateral Pressure Criteria for Stemming Materials", August 18, 1972.
20. Bowman, B., ENN 72-143, Notes on Fluidization of a Porous Column, September 8, 1972.
21. Blake, A., ENN 72-154, Simplified Model for Analysis of Unbalanced Stemming Pressure, October 2, 1972.
22. Hamilton, W. A., ENN 72-159, Stemming Loads on Emplacement Systems, August 1969.
23. Wakeman, William and A. Blake, ENN 72-160, Application of Lateral Pressure Criteria to Pre-Stemmed Diagnostic Canister, October 11, 1972.
24. Bowman, B. R., ENN 72-187, TWIG, a Computer Code for the Calculation of Two-Dimensional Ideal Gas-Flow in Porous Media, November 30, 1972.
25. Bowman, B. R., ENN 72-188, Prediction of Porous Flow From the Solanum Cavity, December 12, 1972.
26. Blake, A and Zaslowsky, M., ENN 72-193, Frictional Resistance of Stemming Materials, January 15, 1973.
27. Bowman, B. R., ENN 73-06, Heat Transfer Analysis of One-Dimensional Porous Flow Experiment, January 9, 1973.
28. Hartzman, M., ENN 73-80, Elastic-Plastic Stress Strain Relations Used in Code EPSOLA, April 20, 1973.
29. Bowman, B. R., ENN 73-29, Porous Flow From an Underground Reactor Summary, February 14, 1973.
30. Zaslowsky, M., ENN 73-38, Confined Compression Test Results on Stemming Materials and Plugs, February 20, 1973.
31. Bowman, B. R., ENN 73-69, Porous Flow Calculations for the Starwort Event, April 2, 1973.
32. Zaskawsky, M. and L. Trent, ENN 73-74, Mechanical Properties of LAE 59 (Plug Material), April 11, 1973.
33. Norris, D. M., B. R. Bowman, and J. Thomsen, ENN 73-137, Seafoam Potential for Late-Time, July 20, 1973.
34. Bowman, B. R., ENN 73-181, Porous Flow From the Pinedrops Cavities, September 18, 1973.
35. Sutton, S. B. and E. W. McCauley, ENN 74-41, Fliprz Pipe Flow Calculations for Harebell with Some Additional Remarks on Starwort and Stanyan I, January 29, 1974, UCID-16545.

36. Morrison, F. A., ENN 75-05, Transient Gas Flow Through Layered Porous Media, January 16, 1975.
37. Blake, A., ENN 75-31, STEM Code--A Correlation of Theory & Experiment, March 19, 1975.
38. McCauley, E. W. and R. W. Martin, ENN 75-37, An Assessment of Edam Cavity Gas Transport by Porous Media Flow, March 19, 1975 (CFRD).
39. McCauley, E. W., ENN 75-38, Camembert Porous Media Flow--Preliminary. April 3, 1975 (CFRD).
40. McCauley, E. W., ENN 75-41, LCSTEM--A New Version of the STEM Code Using LCM, April 3, 1975.
41. Koger, R. G., ENN 75-51, Camembert - U19q Hardware and Stemming Contribution Load Calculations and Results, July 9, 1975.
42. Watling, H. E., ENN 75-60, Camembert LC204 Stemming Platform, June 9, 1975.
43. Kopytoff, V., ENN 75-65, Standardization of Rigid Foam Plugs, June 18, 1975.
44. Martin, R. W. and E. W. McCauley, ENN 75-70, Porous Flow Calculations from Esrom, September 3, 1975 (CRD)
45. Holl, P. M., ENN 75-73, Muenster - U193 Stemming Platform Design Summary, July 16, 1975.
46. Watling, H. E., ENN 75-98, Esrom Plastic Plug Design, October 1975.
47. Watling, H. E. and H. S. Adasiak, ENN 75-99, Permanent Bridge Plug Specification, October 1975 (Rev. A).
48. McCauley, E. W. and R. W. Martin, ENN 75-100, Porous Flow Assessment for Leyden, October 1975 (CFRD).
49. Karpenko, V. N., ENN 75-110, Backfilling Emplacement Holes Stemming Criteria, October 22, 1975.
50. Martin, R. W., ENN 75-128, Porous Flow Assessment For Cheshire U20n, December 1975, (CFRD).

APPENDIX D

LLL SPECIFICATIONS FOR STEMMING MATERIALS

SPECIFICATION

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

MEL-75001282

PAGE 1 OF 2

MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE SPECIFICATION FOR LLL STEMMING MIX II	WRITTEN BY	DATE
	R. J. Horton	10-17-75
	APPROVED - SPEC. & STDS.	
	D. J. Nutt <i>[Signature]</i>	
APPROVED - DIVISION HEAD		
V. N. Karpenko		

PURPOSE

This specification will establish allowable limits on physical properties of LLL Stemming Mix II when used to backfill emplacement holes at the Nevada Test Site.

SCOPE

This specification supersedes all other specifications, letters, verbal agreements or other forms of communications related to LLL Stemming Mix. It applies to the emplaced material; hence, ground zero quality assurance is required.

REFERENCE DOCUMENTS

LLL Standard II Stemming Plan
AAA 71-111187-00

LLL Mechanical Engineering Stemming and Containment Drawings for Specific Events.

REQUIREMENTS

As it goes into the emplacement hole, LLL Stemming Mix II must conform to the following minimum and maximum requirements:

INGREDIENTS

	<u>Min.</u>	<u>Max.</u>
NTS Area I Material	92.5%	95.0%
Pozzmix (Flyash)	5%	7.5%
Moisture Content		3.5%
		(< 3% NOM)

LL-256-1 (REV. 8/71)

		CLASSIFICATION
REV.	BY	
		D-2

SPECIFICATION

MEL-75001282

PAGE 2 OF 2

SIEVE ANALYSIS OF AREA I MATERIAL

		<u>Min.</u>	<u>Max.</u>
Through	1/2"	100%	
Through	3/8"	95%	100%
Through	#10	60%	75%
Through	100	10%	15%

TEMPERATURE

Ambient
Unless
Otherwise
Specified 50°C

SPECIFICATION

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

MEL 76-001292

PAGE 1 OF 2

MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE OVERTON SAND	WRITTEN BY <i>HEN</i>	DATE
	H. E. Watling	4/14/76
	APPROVED - SPEC. & STDS.	
	D. J. Nutt <i>[Signature]</i>	
APPROVED - DIVISION HEAD		
V. N. Karpenko <i>[Signature]</i>		

1. SCOPE

This specification establishes allowable limits for the physical properties of Overton sand used to backfill emplacement holes for Lawrence Livermore Laboratory (LLL) events conducted at the Nevada Test Site.

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein.

American Society for Testing and Materials (ASTM)

E11 Wire Cloth Sieves for Testing Purposes

Lawrence Livermore Laboratory (LLL)

NS 76-7-5 Stemming Procedure

3. REQUIREMENTS

3.1 Physical Properties When placed in the hole, the sand shall have the following physical properties:

3.1.1 Sieve Analysis The particle size distribution shall be within the following limits when tested using a sieve per ASTM E11.

a. 100% shall pass through a 2.00 mm (#10) sieve.

b. Not more than 5% shall pass through a 75 μ m (#200) sieve.

3.1.2 Moisture Content The moisture content shall not exceed 0.2% by weight.

LL-256-1 (REV. 8/71)	(See paragraph 5.1)		CLASSIFICATION
REV.	BY	DATE	D-4

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MEL 76-001292

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3.1.3 Temperature Unless otherwise specified, the sand shall be at the ambient temperature except that it shall not exceed 50° C.

3.2 Stemming Plans The sand shall be placed in the hole in accordance with LLL Mechanical Engineering's stemming and containment drawings for the specific event.

4. INSPECTION AND TESTING

During backfilling of emplacement holes, tests shall be performed in accordance with LLL procedure NS 76-7-5.

5. NOTES

5.1 Revisions Revisions to this specification shall be reviewed by the originator of this specification and require approval of the division head, Nuclear Test Engineering Division.

SPECIFICATION

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

MEL 76-001296

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
☐ STANDARD

TITLE OVERTON PEA GRAVEL	WRITTEN BY H. E. Watling	DATE 4/14/76
	APPROVED - SPEC. & STDS. D. J. Nutt	
	APPROVED - DIVISION HEAD V. N. Karpenko	

1. SCOPE

This specification establishes allowable limits for the physical properties of Overton pea gravel used to backfill emplacement holes for Lawrence Livermore Laboratory (LLL) events conducted at the Nevada Test Site. The gravel may also be used as a component of the epoxy stemming plugs for the holes.

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein:

American Society for Testing and Materials (ASTM)

E11 Wire Cloth Sieves for Testing Purposes

Lawrence Livermore Laboratory (LLL)

NS 76-7-5 Stemming Procedure

3. REQUIREMENTS

3.1 Physical Properties When used in stemming operations, the gravel shall have the following physical properties:

3.1.1 Sieve Analysis The size distribution shall be within the following limits when tested using a sieve per ASTM E11.

Sieve	% Passing Through
12.5 mm (1/2")	100% min.
9.5 mm (3/8")	95% min.; 100% max.
4.0 mm (#5)	0% min.; 15% max.
2.0 mm (#10)	0% min.; 5% max.

LL-256-1 (REV. 8/71)	(See paragraph 5.1)		CLASSIFICATION
	REV.	BY	
		D-6	

SPECIFICATION

MEL 76-001296

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3.1.2 Moisture Content The moisture content shall not exceed 2.0% of wet weight.

3.1.3 Temperature

- a. When used in backfilling emplacement holes, the gravel shall be at the ambient temperature except it shall not exceed 50°C.
- b. When used as a component of an epoxy stemming plug, the temperature of the gravel shall be 21 to 32°C.

3.2 Stemming Plans The gravel shall be used in stemming operations in accordance with LLL Mechanical Engineering's stemming and containment drawings for the specific event.

4. INSPECTION AND TESTING

During stemming operations, tests shall be performed in accordance with LLL procedure NS 76-7-5.

5. NOTES

5.1 Revisions Revisions to this specification shall be reviewed by the originator of this specification and require approval of the division head, Nuclear Test Engineering Division.

SPECIFICATION

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MEL 76-500072

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE ACTIVATOR FOR EPOXY COMPOUNDS	WRITTEN BY	DATE
	P. G. Fleming	4/14/76
	APPROVED - SPEC. & STDS.	
	D. J. Nutt	
	APPROVED - DIVISION HEAD	
	V. N. Karpenko	

1. SCOPE

This specification establishes the requirements for a glycerol-water solution to be used as an activator in epoxy compounds.

2. REFERENCE DOCUMENTS

The following document forms a part of this specification to the extent specified herein.

United States Pharmacopeial Convention Inc.

U.S. Pharmacopeia (USP)

3. REQUIREMENTS

3.1 Glycerol The glycerol (anhydrous glycerin) used in the solution shall be USP grade except that it shall have a specific gravity of 1.265 minimum.

3.2 Activator Solution The activator solution shall be comprised of the following:

Material	Parts by Weight ($\pm 0.1\%$)
a. Glycerol	1
b. Distilled water	6

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	REV.	BY	DATE

SPECIFICATION

MEL 76-500072

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4. PACKAGING AND MARKING

The activator solution shall be mixed and stored in a polyethylene or other containers providing equivalent corrosion resistance. Each container shall be marked to indicate the following:

- a. Specification number (MEL 76-500072)
- b. Product designation
- c. Name of producer or supplier of glycerol
- d. Date solution was compounded

5. NOTES

5.1 Storage Material should be stored at temperatures from 5°C to 35°C. In a well sealed container, the shelf life is considered unlimited.

5.2 Revisions Revisions to this specification shall be reviewed by the originator of this specification and require approval of the division head, Nuclear Test Engineering Division.

SPECIFICATION

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

MEL 71-500060 C

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE EPOXY RESIN	WRITTEN BY P. G. Fleming <i>PGF</i>	DATE 8/25/72
	APPROVED - SPEC. & STDS. D. J. Nutt <i>DN</i>	
	APPROVED - DIVISION HEAD	
	E. R. Bissell <i>ERB</i>	

1. SCOPE

This specification establishes the requirements for a low-molecular-weight diepoxide based on the diglycidyl ether of bisphenol A. It shall be a low-viscosity material suitable for applications where good impregnation and bubble-free castings are required. It shall be a product that has been tested and qualified by the Lawrence Livermore Laboratory (LLL) in accordance with paragraph 6.1.

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. The issue used shall be the one in effect on the date of request for quotation.

American Society for Testing and Materials (ASTM)

D92 Flash and Fire Points by Cleveland Open Cup

D1652 Epoxy Content of Epoxy Resins

D2393 Viscosity of Epoxy Compounds and Related Components

3. REQUIREMENTS

Material furnished under this specification shall have the following properties:

Property	Required Value	Test Method*
a) Viscosity at 25°C, centipoises	1600 to 2200	ASTM D2393
b) Epoxy equivalent weight, g/g mole	215 to 235	ASTM D1652
c) Specific gravity at 25°C	1.140 to 1.147	
d) Flash point (open cup)	400°F min.	ASTM D92

*or an LLL-approved equivalent test

LL-256-1 (REV. 8/71)

		CLASSIFICATION
C	Fleming/Nutt <i>DN</i>	8/9/74
B	Fleming/Nutt <i>DN</i>	9/10/73
A	Fleming/Nutt <i>DN</i>	9/18/72
REV.	BY	DATE D-10

4. INSPECTION AND TEST

Unless called for in the purchase order, the seller is not required to test individual shipments of material (beyond seller's normal quality control) to demonstrate conformance with the requirements of paragraph 3. LLL reserves the right to perform inspections and tests as necessary to determine compliance with the prescribed requirements.

5. PACKAGING AND SHIPMENT

The standard container for shipment of the epoxy resin shall be a 55-gallon, non-returnable, closed head, black iron drum (ICC Type 17E) containing 510 pounds net. When specified in the purchase order, large volume shipments shall be made by tank car or by tank truck. Each container shall be marked, or otherwise identified, as follows:

- a) Manufacturer's name
- b) Epoxy Resin per MEL 71-500060
- c) Batch number
- d) Pounds of material contained

6. NOTES

6.1 Qualified Products The products listed below have been tested for conformance with this specification by LLL and qualify for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence of nonconformance of a product to any requirement of this specification may cause loss of qualification approval.

- a) Celanese Corp., EPI-REZ 50817
- (A) b) CIBA-GEIGY Corp., Araldite 9430
- (B) c) Dow Chemical Co., SD-696
- d) Hysol Division, The Dexter Corp., EA-9350
- e) Shell Chemical Co., CS-236

(C) 6.2 Storage In a well sealed container, the epoxy resin will keep indefinitely (18 months). Contamination by either acidic or basic substances may cause exothermic reaction and should be avoided.

6.3 Safety Precautions Skin contact with the epoxy resin should be avoided. Prolonged or repeated contact may cause sensitization.

SPECIFICATION

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LAWRENCE LIVERMORE LABORATORY

MEL 76-500068

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
☐ STANDARD

TITLE REFINED COAL TAR	WRITTEN BY P.G. Fleming	DATE 1/19/76
	APPROVED - SPEC. & STDS. D.J. Nutt <i>[Signature]</i>	
	APPROVED - DIVISION HEAD R.A. Berg <i>[Signature]</i>	

1. SCOPE

This specification established the requirements for refined coal tar that can be used as an extender in formulations with epoxy resins. The material shall have the properties specified herein and shall be procured from a source that has been qualified per paragraph 6.1 by the Lawrence Livermore Laboratory (LLL).

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. The issues used shall be those in effect on the date of request for quotation.

American Society for Testing and Materials (ASTM)

D20 Distillation of Tars and Tar Products
D70 Specific Gravity of Semi-Solid Bituminous Materials
D95 Water in Bituminous Materials by Distillation
D92 Flash and Fire Points by Cleveland Open Cup
D1665 Engler Specific Viscosity of Tar Products

3. REQUIREMENTS

The refined coal tar shall have the following properties:

Property	Value	Test Method
Specific Gravity, 25/25°C	1.13 to 1.24	ASTM D70
Moisture, % by weight	0.5 max	ASTM D95
Solids Content, % by weight	75 min	ASTM D20
Viscosity at 50°C	10 to 26	ASTM D1665
Flash Point, °C	77 min	ASTM D92

CLASSIFICATION

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REV.	BY	DATE
		D-12

4. INSPECTION AND TEST

Unless called for in the purchase order, the seller is not required to test individual shipments of material (beyond seller's normal quality control) to demonstrate conformance with the requirements of paragraph 3. LLL reserves the right to perform inspections and tests as necessary to determine compliance with prescribed requirements.

5. PACKAGING AND SHIPMENT

The standard container for shipment of the coal tar shall be a 55-gallon, non-returnable, closed head, black iron drum (ICC Type 17E). When specified in the purchase order, large volume shipments shall be made by tank car or tank truck. Each container shall be marked or otherwise identified as follows:

- a. Specification number: MEL 76-500068
- b. Manufacturer's name and product designation
- c. Batch number or manufacturer's code number
- d. Weight of material contained

6. NOTES

6.1 Qualified Products The products listed below have been tested for conformance with this specification by LLL and qualify for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence that a product does not conform to this specification may cause loss of qualification approval.

<u>Manufacturer</u>	<u>Product</u>
a. Allied Chemical Corp	Refined Coal Tar CP524
b. Reilly Tar and Chemical Corp	Refined Coal Tar 7704 Base

SPECIFICATION

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LAWRENCE LIVERMORE LABORATORY

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE SILANE COUPLING AGENT	WRITTEN BY	DATE
	P.G. Fleming	1/19/76
	APPROVED - SPEC. & STDS.	
	D.J. Nutt <i>DJ Nutt</i>	
APPROVED - DIVISION HEAD		
R.A. Berg <i>R.A. Berg</i>		

1. SCOPE

This specification establishes the requirements for silane that can be used as a coupling agent in epoxy compounds. The material shall have the properties specified herein and shall be procured from a source that has been qualified per paragraph 6.1 by the Lawrence Livermore Laboratory (LLL).

2. REFERENCE DOCUMENTS

The following document forms a part of this specification to the extent specified herein. The issue used shall be that in effect on the date of request for quotation.

American Society for Testing and Materials (ASTM)

D92 Flash and Fire Points by Cleveland Open Cup

3. REQUIREMENTS

The silane shall have the following physical properties:

Property	Value
a. Specific Gravity, 25/25°C	0.94
b. Flash Point, °C per ASTM D92	93 min
c. Refractive Index, $n_D^{25^\circ\text{C}}$	1.42
d. Boiling Point, °C	
at 30 mm. Hg	123
at 760 mm. Hg	217

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		CLASSIFICATION
REV.	BY	

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4. INSPECTION AND TEST

Unless called for in the purchase order, the seller is not required to test individual shipments of material (beyond seller's normal quality control) to demonstrate conformance with the requirements of paragraph 3. LLL reserves the right to perform inspections and tests as necessary to determine compliance with prescribed requirements.

5. PACKAGING AND SHIPMENT

When applicable, the method of shipment and limits on the container size will be specified in the purchase order. Each container shall be marked or otherwise identified as follows:

- a. Specification number: MEL 76-500069
- b. Manufacturer's name and product designation
- c. Batch number or manufacturer's code number
- d. Weight of material contained

6. NOTES

6.1 Qualified Products The products listed below have been tested for conformance with this specification by LLL and qualify for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence that a product does not conform to this specification may cause loss of qualification approval.

<u>Manufacturer</u>	<u>Product</u>
a. Union Carbide Corp	A-1100 Silane
b. Union Carbide Corp	A-1102 Silane

SPECIFICATION

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LAWRENCE LIVERMORE LABORATORY

MEL 76-500070

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE EPOXY CURING AGENT, MERCAPTAN TYPE	WRITTEN BY P.G. Fleming	DATE 1/19/76
	APPROVED - SPEC. & STDS. D.J. Nutt <i>DJN</i>	
	APPROVED - DIVISION HEAD R.A. Berg <i>RAB</i>	

1. SCOPE

This specification establishes the requirements for a mercaptan curing agent for epoxy resins. The material shall have the properties specified herein and shall be procured from a source that has been qualified per paragraph 6.1 by the Lawrence Livermore Laboratory (LLL).

2. REFERENCE DOCUMENT

The following document forms a part of this specification to the extent specified herein. The issue used shall be that in effect on the date of request for quotation.

American Society for Testing and Materials (ASTM)

D92 Flash and Fire Points by Cleveland Open Cup

3. REQUIREMENTS

The curing agent shall have the following properties:

<u>Property</u>	<u>Value</u>
a. Viscosity at 25°C	2200 to 2700 cps
b. Density at 25°C	1.14 to 1.17 g/cm ³
c. Flash Point per ASTM D92	110°C min

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		CLASSIFICATION
REV.	BY	

4. INSPECTION AND TEST

Unless called for in the purchase order, the seller is not required to test individual shipments of material (beyond seller's normal quality control) to demonstrate conformance with the requirements of paragraph 3. LLL reserves the right to perform inspections and tests as necessary to determine compliance with prescribed requirements.

5. PACKAGING AND SHIPMENT

When applicable, the method of shipment and limits on the container size will be specified in the purchase order. Each container shall be marked or otherwise identified as follows:

- a. Specification number: MEL 76-500070
- b. Manufacturer's name and product designation
- c. Batch number or manufacturer's code number
- d. Weight of material contained

6. NOTES

6.1 Qualified Products The products listed below have been tested for conformance with this specification by LLL and qualify for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence that a product does not conform to this specification may cause loss of qualification approval.

<u>Manufacturer</u>	<u>Product</u>
Celanese Coatings Co	Epi-Cure 861-M
Diamond Shamrock Chemical Co Nopco Chemical Div	Dion 3-900

SPECIFICATION

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LAWRENCE LIVERMORE LABORATORY

MEL 74-500067

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE CURING AGENT, DIETHYLENETRIAMINE (DETA)	WRITTEN BY P. G. Fleming	DATE 8/9/74
	APPROVED - SPEC. & STDS. D. J. Nutt	
	APPROVED - DIVISION HEAD J. A. Jensen	

1. SCOPE

This specification establishes the requirements for diethylenetriamine (DETA) curing agent. The material shall have the properties specified herein and shall be procured from a source that has been qualified per paragraph 6.1 by the Lawrence Livermore Laboratory.

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. The issues used shall be those in effect on the date of request for quotation.

American Society for Testing and Materials (ASTM)

D891	Test for Specific Gravity of Industrial Aromatic Hydrocarbons and Related Materials
D1078	Test for Distillation Range of Volatile Organic Liquids
D2849	Testing of Urethane Foam Polyol Raw Materials

3. REQUIREMENTS

The diethylenetriamine shall have the following properties:

Property	Value	Test Method
Specific gravity, 20/20°C	0.953 - 0.958	ASTM D891
Distillation, °C		ASTM D1078
Initial boiling point	185 min.	
5% by volume	195 min.	
95% by volume	210 max.	
Dry point	215 max.	
Water, %/wt.	0.5% max.	Para 4.2.1

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	REV.	BY	

4. QUALITY ASSURANCE PROVISIONS

4.1 Lot Definition A lot shall consist of all material produced as a single batch or during one continuous process and offered for acceptance at one time. The seller shall assign a unique identifying number to each lot.

4.2 Testing by Seller The seller shall be responsible for performing inspections and tests to assure conformance with the requirements of paragraph 3. Each lot of material shall be tested for water content as described below.

4.2.1 Water Content The water content shall be determined by the Karl Fischer titration procedure of ASTM D2849, Sections 60 - 69, except that Section 67.1 shall be modified as described below:

- a. The modified procedure requires an additional reagent, glacial acetic acid, reagent grade.
- b. In place of Section 67.1 of ASTM D2849, substitute the following:
67.1 Adjust the nitrogen valve to pass dry nitrogen into the titration vessel at a slow rate (20 to 50 ml per min.). Introduce approximately 100 ml titration solvent and 10 ml glacial acetic acid into the titration vessel making sure that the electrodes are covered with solvent. Adjust the stirrer to give adequate mixing without splashing. Titrate the mixture with Karl Fischer reagent to the end point (see Section 62).

4.3 Tests by LLL The Lawrence Livermore Laboratory (LLL) reserves the right to perform inspections and tests as necessary to determine compliance with the prescribed requirements.

5. PACKAGING

All material in each individual container shall be from a single lot (see para 4.1). Individual containers shall be marked with the following information:

- a. Specification number: MEL 74-500067
- b. Manufacturer's name and product designation
- c. Manufacturer's lot number (see para 4.1)

6. NOTES

6.1 Qualified Products Diethylenetriamine produced by the following sources has been tested for conformance with this specification by LLL and qualified for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence of nonconformance of a product to any requirement of this specification may cause loss of qualification approval.

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- a. Dow Chemical Company
Midland, Michigan 48640
- b. Union Carbide Chemical Company
270 Park Avenue, New York, N. Y. 10017
- c. Jefferson Chemical Co., Inc.
750 Welch Road, Palo Alto, Ca 94304
- d. Ames Labs Inc. (The)
200 Rock Lane, Milford, Conn 06460
- e. Pacific Anchor Chemical Company
1145 So. 10th St., Richmond, Ca 94804

6.2 Storage and Use Limitations Material should be stored at 50 to 85°F. The shelf life of this material is unlimited, provided it is stored in tightly sealed containers.

6.3 Safety Precautions **WARNING:** This material may produce contact dermatitis and sensitization or allergy. Skin contact is to be avoided in handling. If accidental skin contact occurs, the contaminated skin should be washed with soap and water. All precautions associated with handling aliphatic polyamine compounds shall be observed.

SPECIFICATION

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LAWRENCE LIVERMORE LABORATORY

MEL 76-500071

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MECHANICAL ENGINEERING DEPARTMENT, LIVERMORE

☐ LABORATORY
STANDARD

TITLE EPOXY CURING AGENT, KETIMINE TYPE	WRITTEN BY P. G. Fleming	DATE 1/20/76
	APPROVED - SPEC. & STDS. D. J. Nutt	
	APPROVED - DIVISION HEAD R. A. Berg	

1. SCOPE

This specification establishes the requirements for a ketimine curing agent for epoxy resins. The material shall have the properties specified herein and shall be procured from a source that has been qualified per paragraph 6.1 by the Lawrence Livermore Laboratory (LLL).

2. REFERENCE DOCUMENTS (Not applicable)

3. REQUIREMENTS

The curing agent shall have the following properties:

<u>Property</u>	<u>Value</u>
a. Viscosity at 25°C	7 to 10 cps
b. Amine nitrogen content, % by weight	15.5 to 17.5
c. Epoxy equivalent weight	52

4. INSPECTION AND TEST

Unless called for in the purchase order, the seller is not required to test individual shipments of material (beyond seller's normal quality control) to demonstrate conformance with the requirements of paragraph 3. LLL reserves the right to perform inspections and tests as necessary to determine compliance with prescribed requirements.

5. PACKAGING AND SHIPMENT

When applicable, the method of shipment and limits on the container size will be specified in the purchase order. Each container shall be marked or otherwise identified as follows:

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- a. Specification number: MEL 76-500071
- b. Manufacturer's name and product designation
- c. Batch number or manufacturer's code number
- d. Weight of material contained

6. NOTES

6.1 Qualified Products The products listed below have been tested for conformance with this specification by LLL and qualify for procurement under this specification. Qualification evaluation will be performed prior to the first use of a product. Subsequent evidence that a product does not conform to this specification may cause loss of qualification approval.

<u>Manufacturer</u>	<u>Product</u>
a. Shell Chemical Co	Epon H-1

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☐ LABORATORY
STANDARD

		CLASSIFICATION
(See paragraph 6.1)		
REV. BY	DATE D-23	

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SPECIFICATION

MEL 76-001300

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4. TESTING

4.1 Chemical and Sieve Analysis The seller shall select a representative sample of the material to be shipped and have it tested at an independent test laboratory that has been approved by LLL. The sample shall be tested to determine conformance with the sieve analysis (paragraph 3.2) and the iron content (paragraph 3.1). The chemical analysis shall determine the percentage of the following:

- a. Iron
- b. Titanium
- c. Titanium as illmenite
- d. Iron as magnetite

4.2 Angle of Repose If called for in the purchase order, the following test shall be performed and the results reported for information only (i.e., not used for acceptance or rejection purposes). Rod a sample of the magnetite at maximum moisture content in a standard 305 mm (12 inch) concrete slump cone per ASTM C143 and determine the angle of repose. It will normally be approximately 29°.

4.3 Test Report A report covering the above tests shall accompany or precede delivery of the magnetite. The identifying numbers on the bags of magnetite (see paragraph 5) shall also be marked on the test report to permit correlation with the specific material represented.

5. PACKAGING AND MARKING

The magnetite shall be shipped in bags containing 45 kilograms (100 pounds) or less. Each bag shall be marked with a release number and shipment number or other identifying information to permit correlation with the test data of paragraph 4.3.

6. NOTES

6.1 Revisions Revisions to this specification shall be reviewed by the originator of this specification and require the approval of the division head, Nuclear Test Engineering.

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LAWRENCE LIVERMORE LABORATORY

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☐ LABORATORY STANDARD

1. SCOPE

2. REFERENCE DOCUMENTS

E11 Wire Cloth Sieves for Testing Purposes

3.3 Moisture Content The moisture content of the ulexite shall not exceed 1.25% by weight. The temperature at which the material is dried shall not exceed 120°C (250°F).

The seller shall select a representative sample of the material to be shipped and test it to determine conformance with the requirement for boron content per paragraph 3.1. A report covering the test shall accompany or precede delivery of the ulexite. The identifying numbers on the bags of ulexite (see paragraph 5) shall also be marked on the test report to permit correlation with the specific material represented.

LL-256-1 (REV. 8/71)	(See paragraph 6.1)		CLASSIFICATION
	REV.	BY	DATE

SPECIFICATION

MEL 76-001299

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5. PACKAGING AND MARKING

The ulexite shall be shipped in bags containing 45 kilograms (100 pounds) or less. Each bag shall be marked with a release number and shipment number or other identifying information to permit correlation with the test data of paragraph 4.

6. NOTES

6.1 Revisions Revisions to this specification shall be reviewed by the originator of this specification and require the approval of the division head, Nuclear Test Engineering.

ENGINEERING NOTEFILE NO.
ENN 71-149PAGE
1

SUBJECT

PROPOSED DESIGN AND DATA REDUCTION FORMULAS FOR
STEMMING PLUGS

NAME

Alex Blake *AB*

DATE

September 20, 1971

INTRODUCTION

This report presents a brief review of the design variables for stemming plugs with special regard to a coal tar-epoxy mix designated as NBE-59 and developed for the containment purposes in underground nuclear testing. The basic function of the stemming plug is to prevent the stemming material from falling into the cavity formed by the detonation, thereby providing a barrier restricting gas leak. To date experience with the plugs indicates that their performance is strongly affected by the installation procedures and mechanical strength characteristics of the plug material. The mixture designated as NBE-59 has proven, so far, to be the strongest and has been used exclusively in LLL nuclear test programs.

The object of this study was to develop relatively simple design formulas and techniques for predicting the mechanical strength of stemming plugs in terms of the gross physical properties of the material within the specified range of geometrical constraints. The theoretical models involve the principles of soil mechanics and the theory of elasticity. The developed formulas are largely empirical because of the extreme complexity of materials behavior and the number of independent variables involved. The effort has been made to present the results in a simplified manner suitable for a direct use by the design or field engineers.

The available test results obtained on small scale plugs were used in the derivation of the various empirical relations. The extrapolation of such results to full scale plugs must therefore be subject to the usual limitations of theory and the actual design of a given plug still requires a good measure of engineering judgment and supporting field experience. However the formulas appear to be sufficiently conservative and represent a certain step in the rational process which may be helpful in developing plug design criteria and analyzing future full scale results.

ENGINEERING NOTE

SUBJECT

PROPOSED DESIGN AND DATA REDUCTION FORMULAS FOR
STEMMING PLUGS

NAME

Alex Blake

DATE

September 20, 1971

One of the major difficulties experienced concerns the concepts of the basic, physical properties such as the modulus of elasticity and the Poisson's ratio. The tar-epoxy mixture with the gravel used as a filler leaves no doubt that, at best, the gross physical properties are pseudo-elastic, depending on curing characteristics and working environment of the plugs as well as the size and geometry of the plugs in addition to any field constraints encountered during the process of pouring. No single mathematical relation seems feasible to cover all such variations and the analyst is forced to utilize the empirical indications and the trends which can help to explain various modes of plug failures in the laboratory. Hopefully, such observed trends can then be utilized in the design calculations. For this reason a given set of formulas pertains to specific material characteristics and geometry.

For the purpose of this report it is necessary to define at least three distinct cases of mechanical behavior encountered in the analysis of plug materials and the theoretical models involved. It should be emphasized that any additional modes of behavior and failure are possible, and the three cases quoted by no means exhaust the field of interest. It is felt however that such modes might be best relegated to further research since supplementary systems analysis and materials data are still required for further work. This is especially true in the case of plugs poured in an uncased borehole or in using the failure modes of plug samples in the laboratory to analyze and explain ground behavior and containment problems such as for instance those experienced on BANE BERRY.

The three basic cases considered in this report are as follows:

a) Shear-adhesion model considers the axial load on the plug, distributed over a concentric circular area which in the extreme case is equal to the total cross sectional area of the plug. The load is basically compressive and creates an additional transverse load component on the well casing, containing the plug, due to the Poisson's ratio effect. The failure at the maximum axial load appears to be that of a shear-adhesion type along

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the wall of the casing or a shear plane of failure defined by some form of Mohr-Coulomb criteria. In addition, the plug material tends to expand laterally creating more resistance to loading in the axial sense, particularly in the case of deeper plugs in which shear stresses are predominant.

b) Frictional-adhesion model describes the case of a cylindrical pipe or a cable which is being pulled through the plug. The effect of Poisson's ratio in this instance may be considered to be of the second order and the failure can be described as that of a frictional-adhesion type, provided no prior shear failure takes place in the bulk of the plug material itself. According to the test results available the numerical values of the calculated frictional-adhesion are usually lower than those corresponding to the shear-adhesion type. This general observation appears to be consistent with some of the findings on granular materials (Ref. 1) which indicated that the frictional drag increases with the contact pressure. The frictional-adhesion model, intended originally for the circular geometry, may be extended to other closed sections. The overall mechanical behavior of a plug, within a cable pull-out load configuration, may be modelled with the aid of the theory of a shear spring.

c) Leak-proof model is concerned with some of the theoretical considerations of the material shrinkage and its effect on the stresses leading to the separation of plug material and the casing. The basic properties involved in this situation are tensile adhesion and shrinkage which can be evaluated by tests independently of the actual plug configuration. Since only limited information on this aspect of the plug design can be offered at this time, further experimental and theoretical work would be of value.

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NOTATION

D	Outer diameter of plug, in.
d	Diameter of cable or pipe, in.
E	Apparent modulus of elasticity of plug material, psi
H	Depth of plug, in.
$k = d/D = R_1/R_0$	Plug diameter ratio
$M = R_1/H$	Plug depth ratio
$N = u/R_0$	Shrinkage ratio
P	Radial pressure in thick cylinder theory, psi
q	Containment pressure, psi
R_1	Radius of pipe or load area, in.
R_0	Outer radius of plug, in.
S	General symbol for adhesion stress, psi
S_a	Experimental tensile adhesion, psi
S_h	Horizontal component of normal stress, psi
S_o	Experimental frictional adhesion, psi
S_t	Resultant tension at plug boundary, psi
S_l	Experimental shear adhesion, psi
U	Radial shrinkage, in.
W	Total adhesion load on plug, lbs
W_c	Cable pull-out load, lbs
y	Local indentation, in.
β	Curing time factor
β_t	Temperature correction factor
Δ	Shear spring deflection, in.
γ	Shear spring parameter, in ⁻¹
λ	Shear adhesion design factor
ν	Poisson's ratio for plug material
ϕ_k	Design parameter for leak-proof condition
ψ_k	Empirical factor for leak-proof condition

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GENERAL FORMULA FOR SHEAR-ADHESION

The basic notation for this model is shown in Fig. 3. It is assumed here that the total load W is applied to the plug through a rigid die of radius R_1 , producing a local indentation y . The deflection y corresponds to the load W at which the plug is said to fail. The mode of failure depends on the ratio H/R_0 but in general may be assumed to be of a shear-adhesion type along the wall of the casing, particularly when R_1/R_0 tends to unity. The relation between the dimensional quantities and the elastic constants for a rigid die resting on a soil-like foundation is best expressed by the theory of Boussinesq (Ref. 2) which is concerned with a concentrated load on the plane boundary of a semi-infinite elastic body:

$$W = \frac{2 R_1 y E}{1 - \nu^2} \quad \dots(1)$$

The experiments conducted on 8 inch diameter plug samples with a rigid circular plunger having R_1/R_0 values ranging from 0.25 to 0.75 can be utilized here for the purpose of establishing a straight-line relationship between the apparent modulus of elasticity E and R_1/R_0 ratio. Using these values and Eq.(1), with the assumed value of Poisson's ratio equal to 0.45, the following expression is found:

$$E = 34700 \frac{R_1}{R_0} + 3600 \quad \dots(2)$$

The apparent value of the elastic constant E , given by the above relation, is strictly an empirical quantity derived on the basis of Eq.(1). The upper value of E from Eq.(2) is probably reasonably close to the typical elastic constant which one might obtain in a standard test. The lower bound for $R_1/R_0 = 0$ is likely to be of academic interest and represents certain local rigidity of the plug under a highly concentrated load. Both extreme values of E must be influenced by the inherent viscoelastic properties of epoxy used in the mix.

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The amount of penetration y , observed in the experiments can also be expressed as a function of R_1/R_0 ratio and the depth of the plug H , giving the following relation:

$$y = H \left[0.064 - 0.044 \left(\frac{R_1}{R_0} \right) \right] \quad \dots(3)$$

Introducing nondimensional parameters

$$k = R_1/R_0$$

$$M = R_1/H$$

and combining Eqs.(1) through (3), the following algebraic equation is obtained:

$$W = M H^2 (576 + 5160 k - 3820 k^2) \quad \dots(4)$$

It should be pointed out that the approximate relation given by Eq.(4) is good only for the plug material NBE-59 cured for eight days and invokes the assumption of Poisson's ratio equal to 0.45. The choice of Poisson's ratio in this particular case is largely arbitrary as no reliable experimental value of the Poisson's ratio was available. However, considering certain plastic and viscoelastic tendencies of the mix the value of 0.45 is not unreasonable. A review of great many materials, having similar structural capability indicates that the maximum gross value of Poisson's ratio in practice very seldom exceeds 0.45, on the assumption that the material is reasonably homogeneous. Values greater than 0.5 obtained in various tests can not be defined as Poisson's ratio due to the major local changes of test sample geometry.

A brief examination of Eq.(4) shows that the function of k has a maximum which can not be corroborated by the experimental work conducted to-date. It seems quite obvious that this particular characteristics of the formula is not realistic for empirical reasons. A rather simple algebraic correction however, can be executed to give the following expression:

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$$W = \frac{M H^2 (576 + 7640 k - 3800 k^2)}{1 + k} \quad \dots(5)$$

Although Eq.(5) still has a theoretical maximum value, the numerical difference between the maximum calculated load and the load at $k = 1$ is relatively small and can be neglected. It is reasonable to assume that other variables being constant the total load on the plug at failure should increase with k .

In order to arrive at a more general expression for the total plug load, reference is made to the experimental strength/time and strength/temperature characteristics. This yields

$$W = \lambda \beta \beta_t M H^2 \quad \dots(6)$$

The relevant values of λ , β and β_t are given in Figs. 4, 5 and 6 respectively. The apparent dimensions of Eq.(6) are square inches since the modulus of elasticity has been eliminated via experiment and substitutions. The correction factors given in Figs. 4 through 6 apply to plug material NBE-59. Temperature correction factor β_t was derived from the chart relating the ultimate flexural strength of the NBE-59 material to the temperature of the plug sample (Fig. 2). In view of the limited test data on other thermal effects, the assumption is made here that the general shape and slope of the flexural strength/temperature curve is sufficiently representative of the shear adhesion and frictional adhesion characteristics as a function of temperature. With this stipulation in mind Eq.(6) should give satisfactory results for most practical purposes.

GENERAL FORMULA FOR CABLE PULL-OUT LOAD

The design analysis of cable pull-out load presented in this report involves several engineering assumptions including the well known theory of shear spring behavior. The basic notation for our pull-out model is shown in Fig. 7. The first assumption advanced in this study concerns the plug adhesion strength on outer and inner walls defined by D and d diameters

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respectively. Assuming that the unit adhesion strength is basically the same on both walls it is obvious that the cable pull-out load will precipitate the failure at the smaller diameter. Since both the adhesion bond and the basic shear bond must be involved simultaneously the resultant effect may be termed frictional adhesion. In terms of the elementary stress analysis the resultant effect appears to depend on the combined state of stress consisting of the tension normal to the plug walls and the transverse shear parallel to the wall surfaces.

The distribution of the frictional adhesion forces acting on the cable or a pipe section, under pull-out conditions, can not be expected to be uniform along the length of the plug. As indicated in Fig. 7 the frictional adhesion is probably the lowest at the bottom of the plug and should perhaps reach a maximum value somewhere close to the top. It should be noted here that this assumption is allowed to depart from the standard theory of strength of materials which states that the distribution of the shearing stresses follows a parabolic law with zero values at the bottom and top of the cross section. This departure from the standard theory is predicated here on the following theoretical considerations:

- a) Considerable tensile stresses set up in the cable in the axial direction must produce the decrease in the dimension d . This dimensional change due to the Poisson's ratio effect is likely to create tensile forces at the outer surface of the cable due to the basic adhesion strength of the plug material.
- b) When the plug is only moderately thick the opening around the cable at the bottom of the plug will have the tendency to increase with bending due to the downward load. At the same time the opening at the top will tend to decrease.

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For mathematical convenience the distribution of the frictional adhesion can be represented by the following relation:

$$S = \frac{3 S_0 x^3}{2 H^3} \quad \dots(7)$$

Here S_0 = average frictional adhesion obtained from tests, psi (Fig. 1)

By analogy to the shear distribution in a standard beam of a rectangular cross section, the maximum stress at the neutral axis is $1.5 S_0$. Utilizing this numerical value, but allowing upward shift of the point of the maximum stress Eq.(7) gives $S = 1.5 S_0$ when $x = H$. Since now the elementary load on the cable can be defined as $dW = \pi d S dx$, integrating over the total depth of the plug, gives

$$W = \frac{3\pi d S_0 H}{8} \quad \dots(8)$$

The choice of the shear distribution function given by Eq.(7) is largely arbitrary at this stage of the development since no experimental data on the actual distribution of frictional adhesion is available. It is likely however that such a distribution can be represented by the functional relation of a higher exponent than 2.

The analysis of the cable pull-out load must now differentiate between the two extreme cases of geometry. In the first instance when the plug ratio D/H is relatively high, say more than 1, Eq.(8) may be directly applicable. For instance taking $S_0 = 160$ psi from the plug model experiments at 8 day curing time and room temperature we get:

$$W = 188 H d \quad \dots(9)$$

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On the other hand when D/H becomes rather small, as is the case with more recent field applications, uniform frictional adhesion might yield the total load which can be estimated from the following expression:

$$W = 500 H d \quad \dots(10)$$

In order to further investigate some of the theoretical aspects of the effect of the diameter ratio D/d on the pull-out load, consider a well known shear spring model (Ref. 3). The deflection according to this model is:

$$\Delta = \frac{(1 + \nu)}{\pi H} \left(\frac{W}{E} \right) \log_e \left(\frac{D}{d} \right) \quad \dots(11)$$

Assuming the value of the Poisson's ratio ν to be about 0.45, a useful shear spring parameter may be obtained from Eq.(11)

$$\gamma = \frac{0.46 \log_e (D/d)}{H} \quad \dots(12)$$

Utilizing Eqs.(9),(10) and (12), a linear relation can be set up which gives the expression for the cable pull-out-load W_c in terms of the dimensional parameters usually specified by the design:

$$W_c = 168 d \beta \beta_t [H + 8 \log_e (D/d)] \quad \dots(13)$$

The relevant values of β and β_t can be obtained from Figs. 5 and 6 respectively. It will be assumed here that the curing time and temperature factors postulated originally for the shear-adhesion model, Eq.(6), are also applicable to the load condition of frictional adhesion described by Eq.(13). In the development of Eq.(13) a linear functional relationship has been assumed for the sake of expediency and simplicity of the data reduction procedures. Additional tests for the type of geometry depicted in Fig. 7 may be used to refine the cable pull-out load criteria described by Eq.(13).

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STRUCTURAL ASPECTS OF LEAK-PROOF DESIGN

The design criteria set-up for shear or frictional adhesion characteristics reflected in Eqs.(6) and (13), do not automatically satisfy the requirements of leak-proof performance. It appears that a significant amount of research in this area still remains to be done for the containment purposes and this section presents only some of the preliminary structural considerations related to the problem. One of the basic physical properties, not yet discussed in this report, is the concept of shrinkage as a function of curing temperature, curing time and the nature of epoxy-gravel interactions.

The general stress field conditions at the junction of plug and the wall of the casing is illustrated schematically in Fig. 8. The amount of potential shrinkage, defined as u , is normally assumed to be taken up by the unit stress P . In other words, the plug material exerts a horizontal force on the casing by pulling the epoxy away from the boundary between the epoxy and metal. Theoretically the same force tends to decrease the diameter of the casing. However, because of a very substantial difference between the elastic moduli of casing and epoxy, the corresponding radial deformation of casing may be neglected.

If q is the internal pressure to be contained by the plug, its effect on the plug material will vary with the overall plug depth H . The nature of this variation is not known because of the complexity of the system involved. It is likely, however, that in the immediate vicinity of the pressurized surface of the plug, the familiar plane strain correlation can be invoked. The plane strain conversion of the longitudinal pressure into the horizontal component provides additional variable in the system. With respect to the boundary between the plug material and the casing wall the two horizontal components, namely P and S_h must therefore be acting in the opposite sense to each other as shown in Fig. 8. Assuming uniaxial stress only, the difference between such two components must be smaller than the allowable tensile adhesion strength of the epoxy in order for the

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gas to be contained. This containment condition, however, applies only to flow of gas through a structurally created crack and is assumed to be independent of any other mechanism of leakage.

The allowable tensile adhesion of the mix can be determined experimentally. Denoting the allowable value by S_a the following general statement of the problem may be postulated for the limiting case:

$$P - S_h = S_a \quad \text{....(14)}$$

From the theory of elasticity applicable to thick cylinders the first term of Eq.(14) leads to:

$$P = \frac{UE}{R_o} \phi_k \quad \text{....(15)}$$

Here ϕ_k is a theoretical parameter depending on the ratio R_1/R_o and the Poisson's ratio of the plug material. For the purpose of this discussion this parameter is given in Fig. 9, for a value of Poisson's ratio equal to 0.45. The plane strain correction gives $S_h = 0.82 q$ for the same value of $\nu = 0.45$. Putting $U/R_o = N$, and utilizing previously derived formula, Eq.(2), yields

$$S_t = N \psi_k - 0.82 q \quad \text{....(16)}$$

The formula given by Eq.(16) is intended for the plug material designated as NBE-59. The empirical parameter ψ_k is illustrated in Fig. 10. The value of shrinkage displacement U and the corresponding parameter N must be determined experimentally. The term q in Eq.(16) denotes the internal pressure for which the plug must be designed, and so determined value of stress S_t must be less than the allowable tensile adhesion S_a .

It is clear from the above brief discussion that a lot more experimental effort is needed to formulate leak-proof design criteria. The formula given by Eq.(16) is offered here in the interim period merely

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as a data reduction tool and by no means represents the complete solution. However, it is hoped that this model can be employed as the first step in the right direction.

CONCLUSIONS AND RECOMMENDATIONS

The results of this study are concerned with the three distinct modes of mechanical behavior of stemming plugs under the usual conditions of loading anticipated in the field. The shear adhesion, frictional adhesion and leak-proof models have been reduced to simplified formulas and charts suitable for the direct application to design and testing of NBE-59 plugs. It should be emphasized, however, that certain pseudo-elastic constants have been assumed in the derivation of working formulas and only a limited number of the test results were directly applicable to the analysis described.

In order to gain confidence in the models presented and to develop improved techniques of predicting the strength characteristics of stemming plugs made in other materials, further research is recommended with special regard to the following aspects of the problem:

- a) Determination of pseudo-elastic constants of plug materials for varying ratios of gravel to epoxy.
- b) Development of additional tests to verify the assumptions made in deriving the formulas with special regard to scaling relationships.
- c) Correlation of the theoretical assessment of leak-proof criteria with field experience on full size stemming plugs.
- d) Extension of leak-proof theory based on structural mechanics to other mechanisms and criteria based on gas containment in terms of flow through porous media due to the local cracking.
- e) Experimental evaluation of shrinkage of plug materials in support of the structural mechanics model of leak-proof behavior.
- f) Refinement of the proposed curing and temperature correction factors for large size stemming plugs.

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FIG. 3 NOTATION FOR SHEAR ADHESION MODEL

SQUARE 10 X 10 TO THE INCH AS-0807-5T

FIG. 1. DESIGN FACTOR FOR ESTIMATING SHEAR
ADHESION LOAD ON STEMMING PLUG

SQUARE 10 X TO THE INCH AS 9907 GT

FIG. 5. CURING TIME FACTOR FOR ESTIMATING SHEAR ADHESION LOAD ON STEMMING PLUG

FIG. 6. TEMPERATURE CORRECTION FACTOR FOR SHEAR AND FRICTIONAL ADHESION

FIG. 7. NOTATION FOR CABLE PULL-OUT MODEL

FIG. 8. NOTATION FOR STRESS FIELD AT LEAK-PROOF CONDITIONS

SQUARE 10 X 10 TO THE INCH AS-0807-6T

FIG. 9. DESIGN PARAMETER FOR LEAK-PROOF ANALYSIS

FIG.10. Empirical Parameter for Leak-Proof Analysis of NBE-59 Plug Material

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