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SPECIFICATION FOR KEVLAR 49 FIBER AND
SAMPLING PLAN FOR TENSILE STRENGTH

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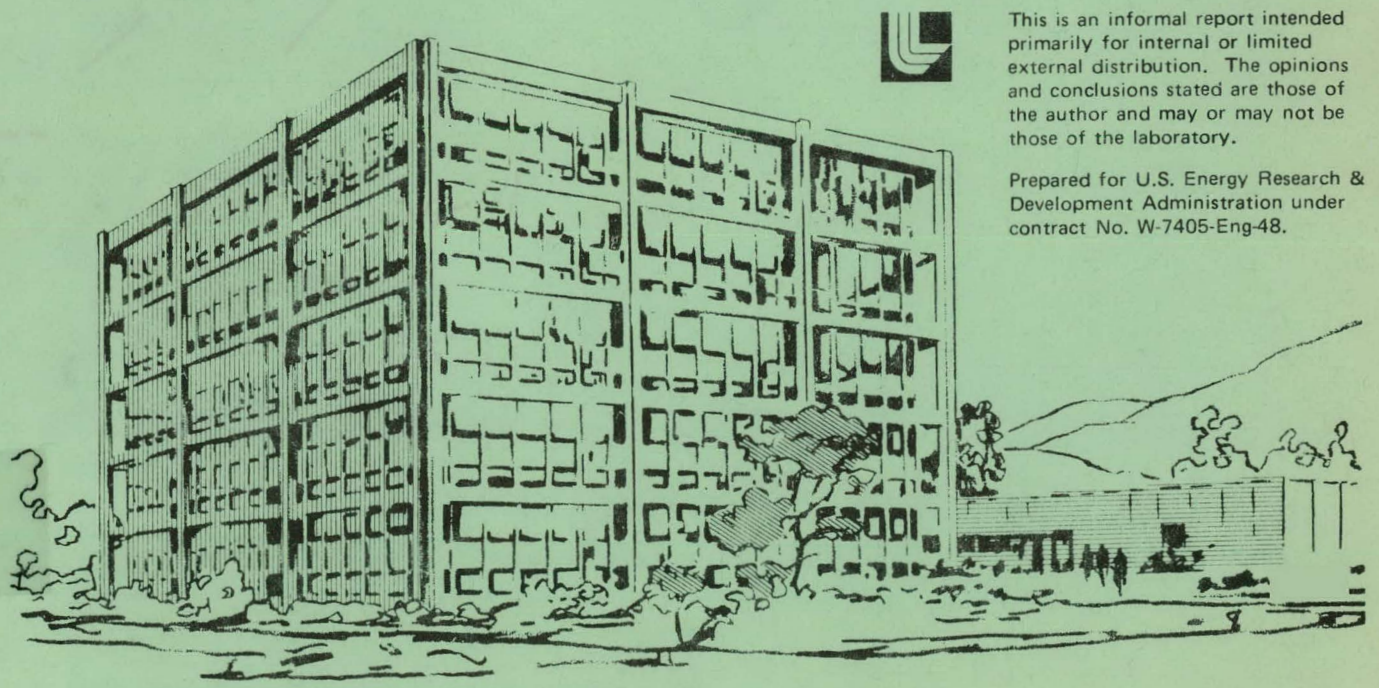

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DISCLAIMER

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PREFACE

The document presented here is in two parts: a specification for Kevlar 49 yarn and a sampling plan for tensile strength. This is not intended to be a report but rather an example of the type of specification and sampling plan that currently serves our needs. These needs are not only to pinpoint the material properties but to determine values representative of the entire lot of material.

The specification may be used for purchase and acceptance or for quality control of Kevlar 49 yarn (380 denier). Applied to yarn used in high-performance filament wound composite applications, it calls for clear definition of the material chemically, physically, and mechanically. The material must conform to preset standards of linear weight (denier), chemical structure, density, water content, and tensile properties. Recommended test procedures are included in the text.

The sampling plan given in Part Two uses tensile strength as a basis, and the tensile property values listed stem from prior experience with the material. It must be pointed out that there are any number of sampling plans that could be used. The one described here is a sequential acceptance type that applies to a property for which a minimum value is set.

PART ONE

SPECIFICATION FOR 380 DENIER KEVLAR 49 YARN

Lynn Penn

I. Scope

This specification covers only Kevlar 49 organic fiber yarn, 380 denier, without finish. The material shall consist of parallel, unidirectional continuous filaments, 267 +0, -3 per strand, yellow in color, with approximate twist of 328 turns per meter. The lot of yarn to be considered shall be designated by a single multi-digit merge number.

II. Properties

Table 1 gives the desired property values to which the fiber shall conform.

III. Method of Testing

A. Chemical Structure — Infrared spectroscopy shall be used to verify the chemical structure of Kevlar. An aliquot of fiber shall be ground to a fine powder for 10 min in a ball mill grinder cooled by liquid nitrogen. The resulting powder is incorporated into a standard KBr pellet for infrared transmission spectroscopy.

Table 1. Property values.

Property	Value
Denier	380 ± 19 denier
Chemical structure	Match attached spectrum (Fig. 1)
Density (of conditioned fiber)	1.450 ± 0.005 g/cm^3
Water content (% by wt at 23°C, 50% RH)	3.5 ± 0.2
Tensile strength	≥3590 MPa (521,000 psi)
Tensile modulus at 0.5% strain	≥124 GPa (18 × 10^6 psi)
Elemental analysis, % by wt	C 68.60 ± 1.70
	H 4.37 ± 1.72
	N 11.27 ± 0.64
Ash content	<1.40%

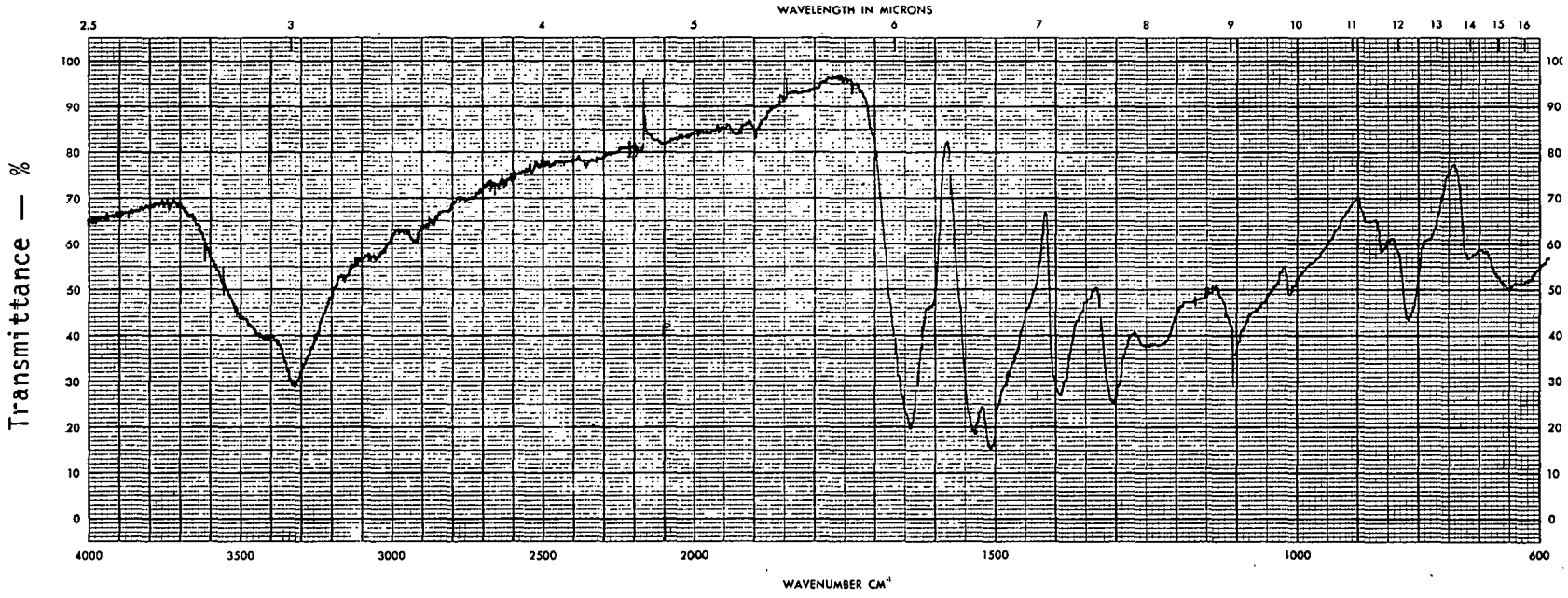

Fig. 1. Infrared spectrum of Kevlar 49 yarn.

B. Tensile Properties — Fiber strands coated with amine-cured epoxy* resin shall be prepared and tested in tension as described by T.T. Chiao and R.L. Moore,† with the following modifications. The cured impregnated fiber strand is cemented into the end clamps, eliminating the use of any rubber strips or foil strips. A sufficiently strong adhesive (Resinbond Adhesive 907 from Resin Formulators Company, Culver City, California) is coated onto the bottom clamp plates, the fiber is aligned on the clamp ends, and the top clamp plates are screwed into place. The adhesive must be allowed to cure overnight at room temperature before testing. The values specifying tensile properties in this document are based on LLL tests of 380 denier Kevlar 49 strands. The coating resin was DER332/T-403 with a room temperature cure.

C. Denier — The denier may be determined on dry fiber on a balance as described in ASTM D1577. The material shall be dried overnight under vacuum in an oven at 50°C to remove absorbed water. Samples of fiber 900 mm in length shall be weighed and the denier calculated:

denier = wt (g) of 900 mm sample × 10,000.

D. Elemental Analysis — An aliquot of approximately 3 mg of dry fiber (vacuum oven at about 13 Pa, 50°C for 16 h) is to be analyzed in a Perkin Elmer Model No. 240 Elemental Analysis apparatus or equivalent. Elements C, H, and N, are expressed as percent of original weight.

E. Ash content — An aliquot of 6 to 10 g of dried fiber (vacuum oven, 50°C, 13 Pa, for 16 h) is to be placed in a porcelain crucible. This is ashed in a muffle furnace under a stream of oxygen sufficiently slow as to not disrupt the sample in the crucible. The following temperature program is satisfactory: starting at room temperature, increase 60°C/h to 650°C; hold at 650°C for 4 h. After cooling and weighing, the ash is reheated to 650°C, allowed to stand, and checked for weight change. When the weight is constant, the ash content is reported as a percent of the original sample weight.

F. Density — Determination of the density of the fiber shall be made in a liquid gradient column according to modified ASTM D1505-68. The gradient column shall be prepared with n-heptane and carbon tetrachloride, both of which are reagent grade and may be used without purification or drying. A

*Bisphenol A epoxy DER332 cured with aliphatic primary amine curing agent, Jeffamine T403.

†T.T. Chiao and R.L. Moore, "A Tensile Test Method for Fibers," J. Composite Materials 4, 118 (1970).

strand of fiber weighing about 0.01 g shall be conditioned for 24 h at 23°C and 50% relative humidity (use gloves for handling). The strand shall be coiled into a small diameter loop (∿ 19.0 mm), moistened with a little gradient fluid to drive off air bubbles, and placed in the top of the column. After 6 h the density is recorded. Density value should be rechecked after 36 h, and any change should be recorded.

G. Water Content — Aliquots of dry fiber (vacuum oven, about 13 Pa, 50°C for 16 h) shall be weighed. Then the aliquots shall be weighed after standing in a chamber at 23°C, 50% relative humidity for a minimum of 24 h. The water content at 23°C, 50% relative humidity is expressed as a percent of moist weight; that is, the difference in weight divided by the moist weight × 100.

IV. Sampling — Any statistical sampling plan may be used that will estimate differences within a unit (spool) as well as differences between spools. It is desirable that at least 7% of the total spools be randomly selected and tested.

PART TWO

SEQUENTIAL SAMPLING FOR ACCEPTANCE (OR REJECTION) OF KEVLAR 49 YARN ON BASIS OF TENSILE STRENGTH

Norriss Hetherington

I. Preliminary Information and Acceptance (Rejection) Conditions

Prior test results for Kevlar 49 fiber were used to set numerical values for the parameters in this sampling plan. The subscript T refers to the statistical parameter of past tensile test experiments.

Ultimate tensile strength, average over several spools, is:

$$\overline{M}_T = 3760 \text{ MPa.}$$

The value from each spool is itself an average of five test samples within that spool. Therefore, $\overline{M}_T$ is an average of averages. The average range of tensile values, an average over several spools, is:

$$\overline{R}_T = 256 \text{ MPa.}$$

The range from each spool is the range over five test samples within that spool and is a single value. Therefore, $\overline{R}_T$ is an average of ranges. The variance between spools is designated by σ_T^2 where

$$\sigma_T = \frac{\overline{R}_T}{2.326} = 110 \text{ MPa.}$$

A necessary condition for the acceptance of a set of spools at 95% confidence level is that the sum of averages, Σ, be equal or greater than (3720 n - 414) MPa, where n is the number of averages. A sufficient condition for rejection is that the sum, Σ, of n averages falls below (3720 n - 414) MPa. In both cases, n (the number of averages, not the number of samples tested) must be greater than 10. Each average is determined from five samples tested within a spool. In addition, the n spools used must be chosen at random from the set presented for acceptance or rejection.

II. Verification of Averages for New Material.

A. Uniformity Throughout Spool — The spool should be sampled in at least 20 locations spread uniformly through the length of the spool, five samples taken at each location.

1. Criterion to be met:

Not more than one of the 20 averages (five samples at each location) should fall outside:

$$\bar{M}_S \pm 0.377\ \bar{R}_S,^*$$

where $\bar{M}_S$ is the mean of 20 averages and $\bar{R}_S$ is the average magnitude of the 20 ranges (each range encompasses five samples). Subscript S refers to new material under test, value within a spool.

2. Criterion to be met:

Not more than one range value out of the 20 range values should fall outside:

$$0.37\ \bar{R}_S \text{ to } 1.81\ \bar{R}_S.$$

B. Uniformity Between Spools and Average Value of Tensile Strength — If both of the criteria in A, above, are met, then >10 randomly chosen spools are to be sampled, using five samples from the initial location of each spool. The average, $\bar{M}_{pop}$, of >10 spool averages, and the average, $\bar{R}_{pop}$, of >10 spool ranges, should meet the following criteria:

$$3380 \text{ MPa} < \bar{M}_{pop} < 3900 \text{ MPa},$$

$$100 \text{ MPa} < \bar{R}_{pop} < 480 \text{ MPa}.$$

III. Decision to Accept or Reject

A. If all criteria in II are met, the acceptance scheme of I is applied.

B. If both criteria in IIA (uniformity throughout a spool) are not met, the set should be rejected.

C. If there is a case where within a spool and between spools there is consistency but levels are different from those outlined here, $\bar{M}_{pop}$ and $\bar{R}_{pop}$ (averages of the >10 spools) may be deemed acceptable by LLL. These values may be substituted for $\bar{M}_T$ and $\bar{R}_T$ with new acceptance (rejection) criteria calculated as follows:

Set: $\alpha = 0.025$ = probability of accepting set when true tensile strength of population is less than m_1,

$\beta = 0.025$ = probability of rejecting set when true tensile strength of population is greater than m_2,

*The coefficient 0.377 comes from the theory of samples of 5.

m_1, m_2 determined by LLL according to engineering requirements $[m_1 < \overline{M}_{pop} < m_2]$.

Calculate:
$$Z_1 = n\left(\frac{m_1 + m_2}{2}\right) - h_1,$$
$$Z_2 = n\left(\frac{m_1 + m_2}{2}\right) + h_1,$$
$$h_1 = \frac{\sigma^2_{pop} \ln\left(\frac{1 - \alpha}{\beta}\right)}{(m_2 - m_1)},$$

where:
$$\sigma_{pop} = \frac{\overline{R}_{pop}}{2.326},$$

n = number of groups of 5 averages used.

Acceptance: Σ of averages of 5 $> Z_2$.

Rejection: Σ of averages of 5 $< Z_1$.

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