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Effect of Mounting Technique on the Laboratory Weathering of Polypropylene Monofilaments

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Synopsis

A comparative study was made of two methods of mounting yarns in the Weatherometer. In the first, in which test materials are wound continuously around separated halves of wooden hubs, the yarns tend to bunch. In the second, the sample is constrained from bunching but is free to relax or to shrink. Use of the second method of mounting significantly shortens exposure time and greatly improves the repeatability of tensile and elongation tests performed on exposed yarns.

INTRODUCTION

Laboratory aging is a commonly used method for evaluating ultraviolet and oxidative stability of materials under conditions that are generally considered to be accelerated compared to outdoor exposure in sunlight. However, such methods are generally regarded as screening tests and not as substitutes for outdoor aging.

Nevertheless, frequent attempts have been made to correlate Weatherometer data with those obtained under sunlight.[1-8] Generally, such attempts have not been too successful because of (*1*) poor reproducibility obtained in outdoor weathering, (*2*) the lack of precision in the laboratory test, and (*3*) the difficulty of reproducing the spectral quality of sunlight in the laboratory. For example, in the carbon-arc Weatherometer, there is a very intense emission peak at 3900 A. that is not present in sunlight. If either the material itself or a modifier added to the material happens to be sensitive to this wavelength, which is often the case, the carbon arc will unduly accelerate decomposition. As was pointed out[9] fully 12 years ago and is still true today, satisfactory correlation between sunlight and the Weatherometer is still an unsolved problem.[10,11]

To get around some of the problems enumerated above, other types of laboratory weathering have been proposed. Among these is the xenon arc method. To date, however, this method overcomes only one of the obstacles to correlation with outdoor aging data. Although the xenon arc spectrum is more like that of sunlight than is the carbon arc, complete reliability and desired accuracy have not yet been attained with this instrument.

Despite the problems listed above, the carbon-arc Weatherometer is very useful as a screening tool for obtaining qualitative information as to how a fiber, yarn, rope, or plastic would perform under actual service conditions out-of-doors.

During the past several years, consideration has been given to possible changes in the method of mounting fiber samples for Weatherometer exposure in our laboratories. The purpose of the study described in the paper was to determine the quantitative effects of mounting method on the level of aging and on the reproducibility of results.

PROCEDURES

Weatherometer Type and Conditions

The Weatherometer employed in these tests was an Atlas Model XWR, regular size, sunshine carbon arc with automatic humidity control. Complete specifications for the Weatherometer are shown in Table I. Test conditions were chosen to conform to ASTM E-42-64, except that a 23-hr. cycle of light and spray was used in the present work.

TABLE I
Specifications and Operating Conditions for Weatherometer

Model	Atlas XWR—Regular size sunshine carbon-arc Weatherometer with automatic humidity control
Line voltage	220 V.
Arc voltage	52–54 V.
Carbon electrodes	
Upper	No. 22, $^7/_8 \times 12$ in. copper-coated sunshine
Lower	No. 13, $^1/_2 \times 12$ in. copper-coated sunshine
No. of electrodes:	3 pair (1 pair burns at a time)
Filters	Eight Corex D optical, heat-resistant glass, with cutoff at 2550 A., increasing to 91% transmission at 3600 A.
Drum diameter	$37^3/_4$ in.
Speed of rotation	1 rpm
Specimen spray	Four F-80 nozzles, 0.5–0.6 pt./min. at 12–18 psi
Rack cooling spray	Two F-80 nozzles, 0.2–0.3 pt./min.
Arc feed	Automatic, motor-operated
Cycle	102 min. dry, 18 min. spray
Black panel	145 ± 2°F.
Dry bulb	108°F.; wet bulb, 82°F.; 33% R.H. (during dry part of cycle)
	Arcs on 23 hr. per day; off, 1 hr. per day (to allow for changing electrodes)

Sample Selection

For the purpose of this program, four fibers (monofilaments) were prepared from two Escan polypropylene base resins to give a representative spread in stability. These fibers were prepared in both natural and pigmented resins (blue and white). Properties of the base resins and the fibers are shown in Table II.

TABLE II
Processing Conditions and Properties of Fibers

	Polypropylene base resin, A	Polypropylene base resin, A	Polypropylene base resin, B	Polypropylene base resin, B
Resin type	G.P.[a]	G.P.[a]	LTHA[b]	LTHA[b]
Color	Natural	Cabot blue	Natural	Cabot white
Date made	5/10/63	5/15/63	1/24/63	1/24/63
Date first tested	5/23/63	5/23/63	2/3/63	2/3/63
Processing conditions				
Stock, °F.	500	500	552	553
Water, °f.	86	85	88	97
Screw speed, rpm.	22	22	16	23
Pressure, psi	2050/1000	2150/1000	725/535	950/720
Godet #1, fpm.	70	70	70	70
Oven #1, °F.	500	500	1250	1250
Godet #2, fpm.	490	490	490	490
Pump, g./min.	47	47	—	—
Godet #3, fpm.	490	490	—	—
Draw ratio	7:1	7:1	7:1	7:1
Fiber properties				
Denier	231.5	172.5	181.1	224.4
Tenacity, g./den.	6.5	6.7	5.5	5.5
Elongation, %	28.0	20.2	18.0	17.8
Resin Properties				
Melt flow	3.0	—	—	—
Heptane insol., %	95.6	—	—	—

[a] (G.P.) general purpose.
[b] (LTHA) long-term heat aging.

Rack Design

To investigate the effect of mounting techniques or rack design on aging characteristics, racks of two basically different types were studied. In the first two techniques, monofilament samples were wound around two separated halves of a wooden hub (A and B, Fig. 1). Two levels of winding were used on the wooden hub racks. In rack A, the fibers were wound with 80 turns, whereas only 16 turns were used on rack B. It was presumed at the outset that bunching of fibers would result in irregular ultraviolet aging characteristics. Decreasing the number of turns to 16 (rack B) might tend to minimize the shading effect if present.

Rack C (right side Fig. 1, full view Fig. 2), which was wound with 21 turns, represented a new design that was originated at our laboratories. This rack, which is fabricated of stainless steel except for the threaded aluminum hubs, has two novel features: (*1*) each strand of monofilament

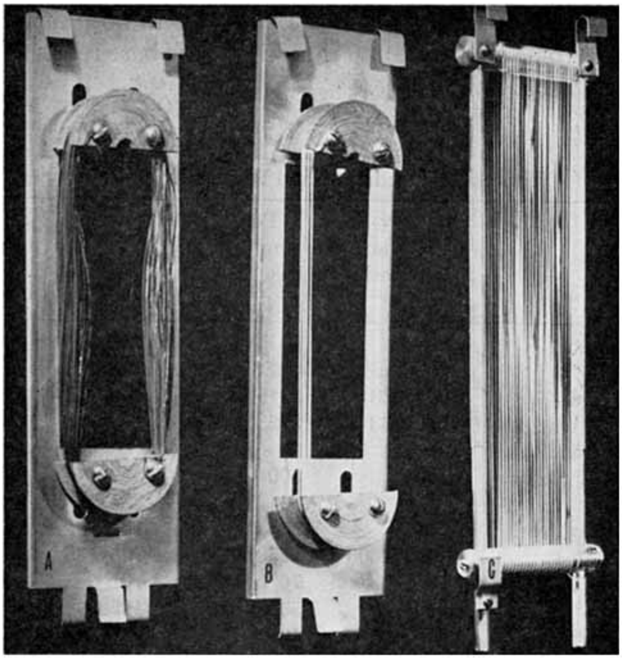

Figure 1.

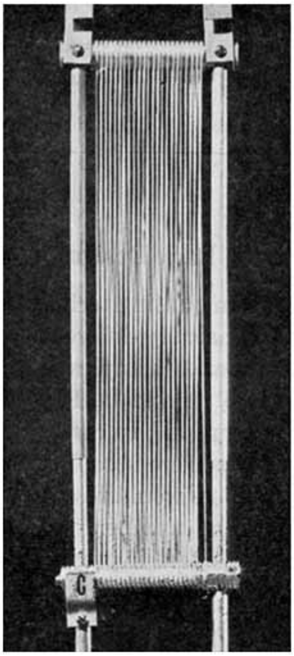

Figure 2.

receives its full complement of radiation; and (2) the bottom hub is fixed during winding of sample but is allowed to ride free during exposure. This allows the fibers to contract without danger of breakage.

One possible criticism of the new rack is that the method would be "too severe" compared to that of the other rack (A or B). However, this question cannot really be decided by intercomparing racks on a Weatherometer but would more properly be done by correlating Weatherometer with outdoor aging results.

Mounting and Exposure

To mount the specimens, fibers were unwound from the spools and wound by hand on racks A, B, and C with the specified number of turns with the lower hubs fixed. A total of twelve racks was wound for each specimen. The lower hubs were loosened and the racks were mounted in the Weatherometer in random order (except that there were an equal number of racks for each specimen on the top and bottom rack holders of the Weatherometer). During exposure, these racks were rotated periodically between top to bottom rack holders to equalize the amount of radiation received by each sample.

The cycle used was a 108/12 min. dry/spray cam, with black panel temperature at 145°F. Detailed specifications and operating conditions are given in Table I. To take advantage of short-term shrinkage or relaxation, samples were withdrawn immediately after one cycle (2 hr. exposure) for tensile and denier measurements. Further tests were carried out at 100, 200, 300, and 354-hr. time steps.

Exposure was conducted "round the clock" rather than with the 6-hr. dark period specified in the recently revised ASTM E-42. However, a dark period of 12 hr. was maintained after each time step. This period was required for tensile tests to determine whether additional exposure would be warranted.

Tensile Tests

For each sample at each time step, three fibers from each of the four sample racks were tested on an Instron tensile tester. Thus, each sample was represented by an average of twelve "pulls" at each time step. The twelve tensile and elongation data were averaged and precision limits were calculated for each. The tensile and elongation tests were carried out in accordance with ASTM D-1380-62T.

RESULTS

The following discussion is based on tests on four representative fibers prepared from resin A (natural and blue) and resin B (natural and white). Detailed properties of the fibers are listed in Table II.

Effect of Mounting Technique on Rate of Weathering

Break Tensile. In Figures 3–6 are plotted the pounds pull, or force, required to break twelve specimens of each sample at each time step. Data for the initial, or zero times are actually data obtained on the samples exposed to one cycle (2 hr.) in the Weatherometer. The shapes of the curves indicate that little or no detectable degradation took place during this short time interval. The data points for the 354-hr. time step were,

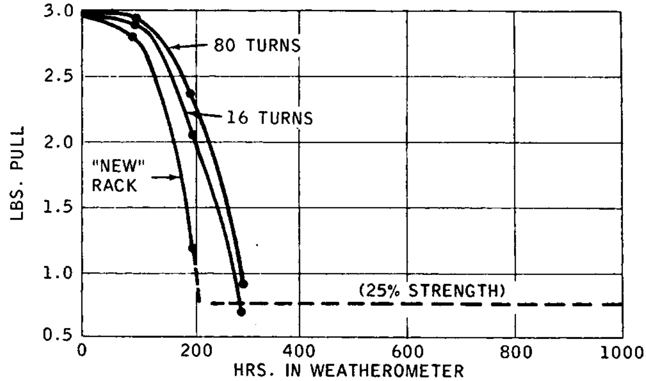

Fig. 3. Comparison of Weatherometer monofilament racks: resin A (natural).

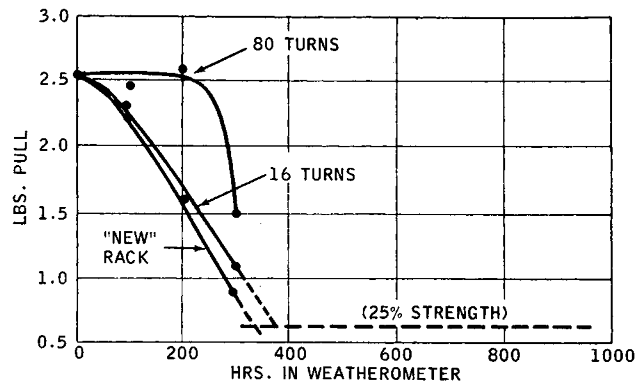

Fig. 4. Comparison of Weatherometer monofilament racks: resin A (blue).

in a number of cases, significantly higher than those of the 300-hr. time step. However, a repeat run failed to show this effect; accordingly, these data are omitted from the plots. A very careful recheck of all the samples and data, plus a considerable amount of testing, has thus far yielded no explanation for this anomaly.

Regarding individual performance of the fibers, the initial rate of degradation was more rapid for resin A than for resin B in both natural and pigmented fibers. This result was expected, since resin B has a higher ultraviolet inhibitor content and a better LTHA (long term heat aging) stabilizer system than resin A. However, as shown in Table III, the time (in the Weatherometer) to 25% of original strength is not markedly better for resin B fibers than for those of resin A. For example, the exposure times to failure on rack A of the natural fibers of resin B were only 15% longer than those of resin A. However, on rack C the differential was increased to 55%. Thus, exposure on the new rack (rack C) apparently permits these two grades to be distinguished more readily than exposure on either rack A or B.

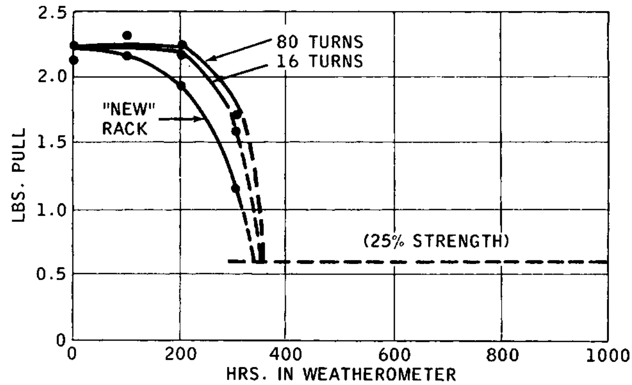

Fig. 5. Comparison of Weatherometer monofilament racks: resin B (natural).

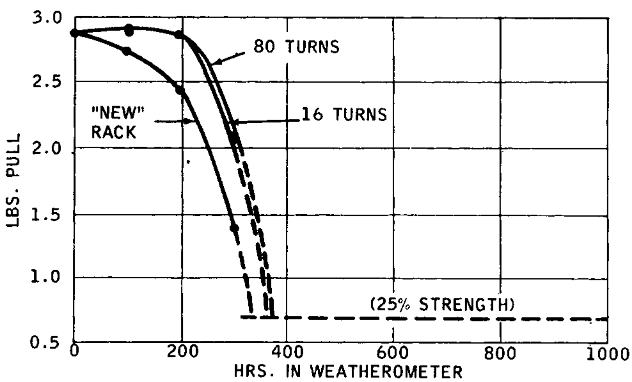

Fig. 6. Comparison of Weatherometer monofilament racks: resin B (white).

Figures 3–6 and Table III indicate that the addition of blue pigment improved resin A's fiber stability to the same stability as resin B. However, incorporation of white pigment in resin B caused no detectable change in the aging characteristics as measured by tensile strength.

A visual comparison of the curves in Figures 3–6 shows that in three cases, racks A and B gave similar aging results, whereas the aging with rack C was noticeably more severe. In three samples, the "shading factor" is as much a problem with only 16 turns as it is with 80 turns. In the fourth

TABLE III
Weatherometer Exposure Time at 25% Strength[a]

Monofilament	Exposure time, hr. Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
Resin A natural	300	290	220
Resin A blue	400[b]	380	340
Resin B natural	360	360	340
Resin B white	380	380	320

[a] Extrapolated from Figs. 3–6.
[b] Extrapolation disregarded 200-hr. tensile data on this material (see Fig. 4).

sample (resin A blue), however, the aging characteristics of the 16-turn rack were close to those of the new rack (Rack C).

To summarize, out of four samples, rack C consistently gave the fastest aging whereas rack A invariably gave the slowest. The performance on rack B was not consistent, however, since in one run results with rack B simulated those with C, whereas in three runs they resembled results on rack A.

Elongation. Figure 7–10 plot the averaged results of per cent elongation at break for twelve specimens of each sample at each time step. As noted in the preceding section, the initial, or zero, exposure values are actually data obtained on fibers exposed to one cycle (2 hr.) in the Weatherometer.

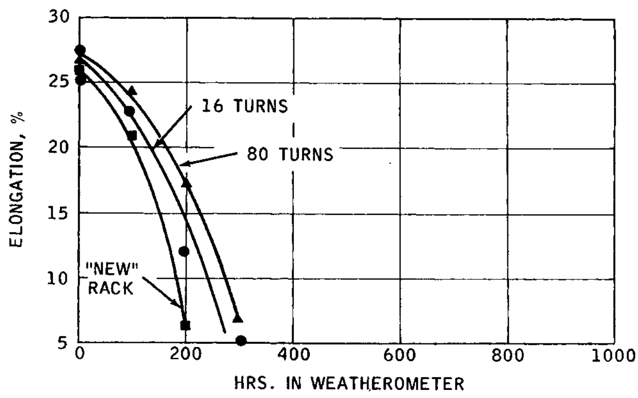

Fig. 7. Comparison of Weatherometer monofilament racks: resin A (natural).

Although elongation of the samples changed smoothly over the whole exposure period, the change between zero and 2 hr. exposure was small and is about the same order as the precision of the test. Unlike the tensile data, the elongation value of samples continued to decrease between the 300 and 354-hr. time intervals.

As was noted previously for tensile strength, the rate of change of per cent elongation with exposure time was more rapid with resin A natural than

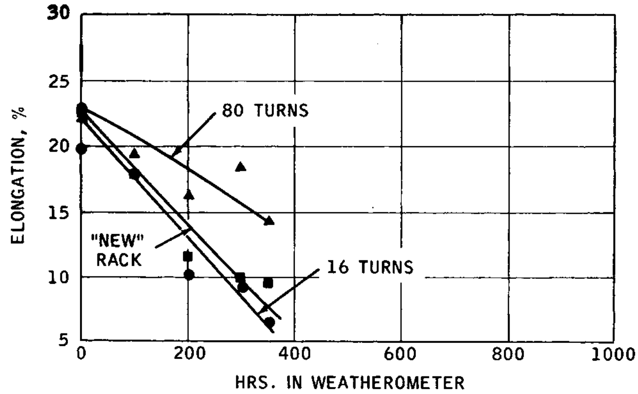

Fig. 8. Comparison of Weatherometer monofilament racks: resin A (blue).

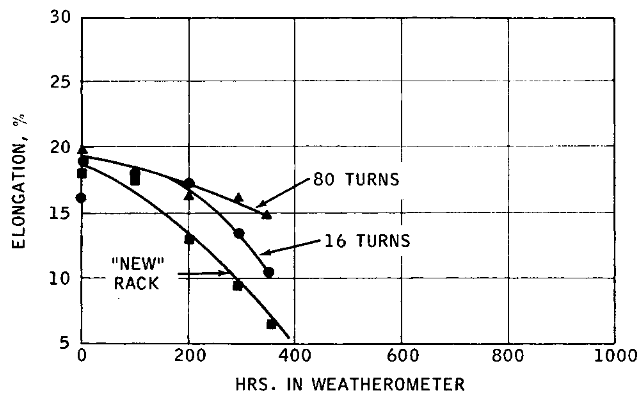

Fig. 9. Comparison of Weatherometer monofilament racks: resin B (natural).

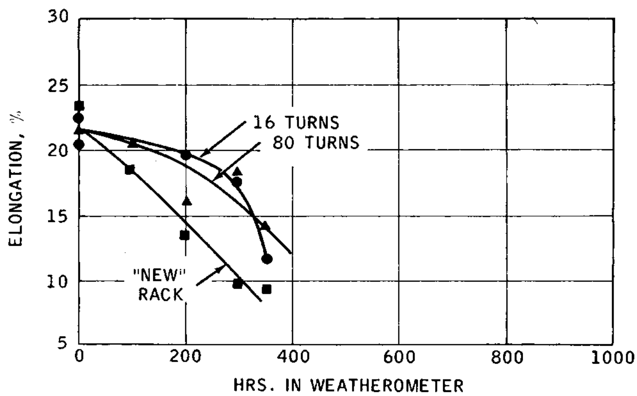

Fig. 10. Comparison of Weatherometer monofilament racks: resin B (white).

TABLE IV
Weatherometer Exposure Time to 10% Elongation[a]

Monofilament	Exposure time, hr. Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
Resin A natural	280	240	180
Resin A blue	460	300	270
Resin B natural	480	360	300
Resin B white	440	360	300

[a] Extrapolated from Figs. 7–10.

resin B fibers. This result would also be predicted from the superior stabilizer system in the resin B. Another comparison is made in Table IV, which lists the time to reach 10% break elongation. This level of elongation was chosen somewhat arbitrarily but does indicate a point at which the fiber has appreciably degraded. Table IV also shows that the addition of a blue pigment to resin A has significantly improved the weathering characteristics of the resin as measured by break elongation. (This same conclusion was reached previously in the tests in which the tensile strength was used as a criterion.) By contrast, the addition of white pigment to resin B produces no discernible changes in elongation characteristics or in ensile characteristics.

From a visual comparison of the curves in Figures 7–10, one concludes that rack A gives the lowest rate of degradation, whereas rack C gives the highest. Rack B generally gives intermediate values, sometimes agreeing more with those of rack A and sometimes with those of rack B. This behavior is probably related to the shading factor inherent in racks A and B. In rack B, which only had 16 turns, the shading factor is of variable importance, depending on the particular fiber.

The curves of the two pigmented fibers show much more scatter of elongation data than those of their natural counterparts. There does not appear to be a clear explanation for this effect, other than the possibilities of greater variation in orientation, inhomogeneity of the pigment in the fibers, or shading. Scattering of elongation data for pigmented fibers was particularly serious in rack A (80 turns), possibly because of the outer turns of pigmented material shaded or screened the inner turns. It will be recalled that the shading factor appeared to be most serious with the blue fiber in scattering the tensile strength data.

In summary, out of four samples, rack C consistently gave the fastest aging whereas rack A was consistently the slowest as measured by elongation changes. Performance on rack B was inconsistent, sometimes resembling that of rack A and sometimes that of rack C.

Effect of Mounting Technique on Test Precision

Break Tensile. Preceding sections have dealt with the effect of mounting technique on the absolute levels of break tensile and elongation after

TABLE V
Precision of Break Tensile Test for Weatherometer Racks[a]

Fiber sample exposure time, hr.	2σ limits, lb. Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
A natural, Original	0.21	0.21	0.21
A blue	0.05	0.05	0.05
B natural	0.12	0.12	0.12
B white	0.08	0.08	0.08
Mean	0.17	0.17	0.17
A natural, 2 hr.	0.42	0.25	0.16
A blue	0.28	0.06	0.05
B natural	0.32	0.41	0.24
B white	0.17	0.05	0.08
Mean	0.31	0.24	0.15
A natural, 100 hr.	0.47	0.42	0.28
A blue	0.27	0.31	0.20
B natural	0.24	0.13	0.12
B white	0.09	0.09	0.31
Mean	0.30	0.27	0.24
Overall mean, 2–100 hr.	0.30	0.26	0.21
A natural, 200 hr.	1.16	1.13	0.29
A blue	0.51	1.04	0.59
B natural	0.22	0.22	0.17
B white	0.27	0.22	0.23
Mean	0.62	0.79	0.36
A natural, 300 hr.	0.72	0.52	[b]
A blue	1.18	0.83	0.28
B natural	0.21	0.31	0.17
B white	0.42	0.46	0.17
Mean	0.73	0.56	0.22
A natural, 354 hr.	[b]	[b]	[b]
A blue	1.21	0.54	0.27
B natural	0.44	0.68	0.57
B white	0.63	0.90	0.22
Mean	0.83	0.72	0.39
Overall mean, 200–354 hr.	0.74	0.69	0.33

[a] 2σ limits of 12 individual determinations.
[b] Too brittle to test.

Weatherometer exposure. A statistical analysis of the data shows that rack design or mounting technique, together with exposure time, affects test precision also.

To carry out the study, 2σ limits were calculated on tensiles for each set of twelve determinations run on each of the four fibers at each time step. These values were then pooled by averaging sigmas squared (Table V).

TABLE VI
Precision of Break Tensile for Weatherometer Racks[a]

Exposure time, hr.	2σ limits, lbs. Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
Original	0.17	0.17	0.17
2–100	0.30	0.26	0.21
200–354	0.74	0.69	0.33

[a] 2σ Range of individual determinations.

The pooled data indicate that the precision of the test becomes progressively poorer for all three mounting techniques as exposure time is increased. However, the rate of decrease in precision is much lower for the new rack than for the 80- or 16-turn, wooden hub racks. Table VI, which compares pooled data on the original fibers and fibers exposed for 2–100 and 200–354 hr., shows the effect in summary form.

As shown by Table VI, the pooled, mean precision (2σ limits) of the four original fibers is 0.17 lb. After 2 hr. exposure, the precision becomes somewhat poorer, but remains at the same level during the first 100 hr. During the next three time steps (200–354 hr.), precision decreases still more.

Although the precision of tensile data of fibers aged on the new rack has decreased to one half of that of the original fibers, the precision is still twice as good as that obtained on either racks A or B. In effect, decreasing the number of turns on the wooden hub rack from 80 to 16 has shown negligible improvement in tensile precision.

In summary, Weatherometer exposure introduces scatter in tensile results. Data from the new rack (C), however, has twice the precision of that obtained from the wooden hub racks (A and B).

Break Elongation. As a parallel to the tensile study discussed in the preceding section, a similar study was made of per cent elongation at break. This study shows that mounting technique and exposure time affect the precision of the elongation values, just as they affect tensile precision.

For purposes of the study, the 2σ values were calculated on elongation to break for each set of twelve determinations run on each of four fibers at each time step. These data were then pooled by averaging the sigmas squared (Table VII). The pooled data indicate that the precision of the test on exposed samples is poorer than those run on the originals, that is, Weatherometer exposure decreases precision of elongation values. This condition becomes progressively worse after 200 hr. for the wooden hub racks but not for the new racks.

As shown by Table VIII, the mean precision (2σ) limits of the four original fibers is 2.9% elongation. After 2 hr. exposure, the precision decreases to about 4%, but does not change appreciably during the first 100 hr. for any of the racks. However, during the next several hundred hours, the precision of the fiber elongation from racks A and B becomes progressively

TABLE VII
Precision of Break Elongation for Weatherometer Racks[a]

Fiber sample exposure time, hr.	2σ limits of % elongation Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
A natural, Original	4.6	4.6	4.6
A blue	1.8	1.8	1.8
B natural	1.8	1.8	1.8
B white	2.3	2.3	2.3
Mean	2.9	2.9	2.9
A natural, 2 hr.	3.4	5.4	4.0
A blue	2.8	1.7	1.3
B natural	4.2	5.9	4.3
B white	3.3	1.9	2.1
Mean	3.5	4.5	3.2
A natural, 100 hr,	3.1	6.2	5.5
A blue	4.8	5.3	4.2
A natural	2.6	1.8	3.1
B white	2.3	2.4	7.3
Mean	3.9	4.4	5.3
Overall mean, 2–100 hr.	3.7	4.4	4.4
A natural, 200 hr.	15.0	9.4	2.8
A blue	5.8	9.0	7.9
B natural	4.2	2.2	3.9
B white	3.0	6.6	2.0
Mean	9.0	7.4	4.7
A natural, 300 hr.	5.2	3.9	[b]
A blue	13.7	7.7	4.4
B natural	6.4	6.4	2.4
B white	7.7	8.4	1.8
Mean	8.9	6.8	3.0
A natural, 354 hr.	[b]	[b]	[b]
A blue	10.4	4.5	2.6
B natural	6.5	5.7	5.5
B white	5.7	8.4	2.2
Mean	7.7	6.4	3.7
Overall mean, 200–354 hr.	8.4	6.9	3.8

[a] 2σ limits of 12 individual determinations.
[b] Too brittle to test.

poorer, whereas that of fibers exposed on rack C remains at the same level. Decreasing the number of turns on the wooden hub rack from 80 to 16 gave only a modest improvement in precision. As discussed in the preceding section, a decrease in the number of turns caused no detectable change in tensile strength repeatability.

Thus, Weatherometer exposure tends to increase scatter in elongation results. The precision of samples exposed on wooden hub racks is about

TABLE VIII
Precision of Break Elongation for Weatherometer Racks[a]

Exposure time, hr.	2σ limits of % elongation		
	Rack A (80 turns)	Rack B (16 turns)	Rack C (new)
Original	2.9	2.9	2.9
2–100	3.7	4.4	4.4
200–354	8.4	6.9	3.8

[a] 2σ Range of individual determinations.

one third of that of the original fibers. However, the rack C gives nearly twice the precision of racks A or B at longer exposures.

CONCLUSIONS

The use of the wooden hub racks described in this study definitely prolongs tests of fibers in the Weatherometer, as evidenced by both tensile strength and elongation measurements. With this type of rack, changes in the number of turns affect the rate of degradation, which evidently is associated with shading of some fibers by others. As would be expected, some pigments have a greater shading effect than others.

Mounting technique affects the precision tensile and elongation results to a marked degree. Increasing Weatherometer exposure time on the wooden hub rack results in poorer and poorer precision in tensile and elongation determinations. By contrast, exposure on the newer metal rack increases scatter initially but precision soon settles down to a constant level.

Most important, from the standpoint of intralaboratory repeatability and efficiency, the precision of tensile and elongation tests with the metal holder is twice that obtained on the wooden hub rack. Thus, for any desired level of precision, the number of replicates required is reduced to one-half.

Further studies designed to improve the laboratory weathering test are in progress.

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Résumé

Une étude comparative a été effectuée concernant deux méthodes de montage des fils dans un appareil de vicillissement. Dans la première méthode, dans laquelle les matériaux études sont enroulés d'une façon continue autour des moitiés séparées des support en bois, les fils sont tendus en touffe. Dans la seconde méthode l'échantillon est serré à partir de la touffe mais est libre de se détendre ou de se contracter. L'emploi de la seconde méthode de montage écourte fortement la durée d'exposition et améliore fortement la reproductibilité des essais de tension et d'élongation effectués sur les fils exposés.

Zusammenfassung

Ein Vergleich zwischen zwei Montierungsmethoden für Garne in der Bewitterungsapparatur wurde durchgeführt. Bei der ersten, bei welcher die zu prüfenden Materialien kontinuierlich um die getrennten Hälften hölzerner Naben gewunden werden, neigen die Garne zur Büschelbildung. Bei der zweiten wird die Probe an der Büschelbildung verhindert, sie kann jedoch relaxieren oder schrumpfen. Die zweite Montierungsmethode setzt die Bewitterungsdauer merklich herab und verbessert die Reproduzierbarkeit der an den Garnen ausgeführten Zug- und Dehnungstests sehr.

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