

DEPARTMENT OF COMMERCE AND LABOR

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**CIRCULAR**  
OF THE  
**BUREAU OF STANDARDS**

S. W. STRATTON, DIRECTOR

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No. 38

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**THE TESTING OF  
MECHANICAL RUBBER GOODS**

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[1st Edition]  
Issued August 1, 1912



WASHINGTON  
GOVERNMENT PRINTING OFFICE

1912









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## I. INTRODUCTION

The testing of rubber is a matter the importance of which is more generally appreciated now than formerly. The constantly increasing demand for rubber goods by the general public, by railroad companies, and other large consumers points to the necessity for developing standard specifications and tests for rubber as has been done in the case of iron, steel, cement, etc.

The purpose of this circular is to describe the methods of testing used at the Bureau of Standards with the hope that sufficient interest may be aroused among manufacturers and purchasers of rubber goods



to assist in bringing about that concerted action which is necessary for the standardization of tests and to furnish the general public with information which will enable users of rubber goods to determine the quality of materials they secure. It is not intended as a technical discussion of mechanical rubber goods nor as a manual for use in testing laboratories, but as an elementary description of the fundamental principles underlying the manufacture of such materials and the properties they should possess.

### 1. SOURCES AND COLLECTION OF RUBBER

For the information of those who are unfamiliar with the subject a short account will be given of the processes through which rubber passes before reaching the testing laboratory.

The principal sources of crude rubber are South America, Central America, Africa, Asia, and Australasia. The Amazon district of South America is noted for the excellent quality of its rubber. In addition to the large quantity which is collected by natives from trees in the wild state, much rubber is secured from plantations where rubber-bearing trees are cultivated according to scientific principles. This is generally known as "plantation rubber."

Crude rubber is obtained by coagulating and drying the milky latex derived from certain trees and plants.

The quality of the crude rubber which determines its market value depends not merely upon the species of plant from which the latex has been secured, but also upon the locality in which it is grown and in a great measure upon the methods followed in its collection, coagulation, and drying.

Briefly stated, rubber is obtained in the following way: Incisions are made in the bark of the trees, and receptacles are placed under the incisions to collect the gradual flow of latex. The custom usually followed by the natives is to coagulate or dry the latex by means of smoke or merely by exposure to the air. "Plantation latex" is coagulated by the addition of acid, after which the rubber is washed, sheeted, dried, and sometimes smoked. The smoking process has been adopted in an attempt to secure the valuable properties possessed by the wild rubbers, which are coagulated by smoking.

Crude rubber is greatly affected by changes in temperature, becoming stiff when cold and soft and sticky when warm.

Goodyear discovered, in 1839, that if crude rubber to which sulphur had been added was heated to a temperature above the melting point of sulphur it combined with the sulphur, became very much less susceptible to temperature changes, and at the same time gained both in strength and elasticity. This important discovery may be said to mark the practical beginning of the rubber industry, although the crude rubber had been previously used to a limited extent as a waterproofing material. The process is popularly known as "vulcanizing."



## 2. RUBBER SUBSTITUTES

No true rubber substitute—that is, no material possessing all the properties of rubber—has yet been produced on a commercial scale. There are a number of so-called substitutes, however, that may be mixed with rubber to advantage in the production of certain articles. Such materials are produced from vegetable oils, by processes of vulcanization or oxidation.

## 3. RECLAIMED RUBBER

On account of the large amount of waste vulcanized rubber or scrap available, and the high cost of crude rubber, the reclaiming of rubber has assumed such proportions as to constitute an industry in itself. By “reclaimed rubber” is not meant devulcanized rubber, although in some cases the free sulphur is removed. No process has yet been developed by which the process of vulcanization can be reversed and crude rubber reclaimed.

The old method of reclaiming consisted in grinding the scrap and removing the fibers and particles of metal, and other waste material, after which the rubber was mixed with oil, heated in ovens, and sheeted. In a more modern process the fibrous materials are destroyed by treatment with acid, after which the scrap is heated in ovens.

A third method, known as the alkali process, which is carried out on an extensive scale, may be briefly outlined as follows: Old rubber is ground between rollers, particles of iron are removed by magnets, and the ground material is screened. The rubber is then heated in iron vessels containing an alkali solution, by which means free sulphur is removed and the fibrous matter destroyed, after which it is thoroughly washed to remove the alkali and dried by steam coils. It is then mixed between rollers without the addition of oil, and sheeted.

It is said that rubber reclaimed by this process from carefully selected scrap is superior to some of the lower grades of crude rubber.

## II. MANUFACTURE OF MECHANICAL RUBBER GOODS

Crude rubber as received at the factory is in the form of lumps of irregular shape and size, and contains varying amounts of impurities which have to be removed. These lumps are placed in a vat containing water, and boiled in order that they may become sufficiently soft to be handled by the washing rolls.

### 1. BREAKING DOWN AND WASHING

The washing rolls consist of two steel cylinders, about 12 to 18 inches in diameter, which revolve in opposite directions and at different speeds, their axes being parallel and in the same horizontal plane. These rolls are corrugated, and as the crude rubber is fed between them their action is such as to masticate the soft lumps and expose the impurities, which are washed out by a series of water jets and collected in a pan under the rolls. Two sets

of rolls are used in this process. The first set breaks down the lumps while a large part of the impurities is washed out, and the second set, in which the rolls are closer together, completes the process of washing. After a sufficient number of passages through the rolls, the washed rubber has the form of a rough sheet of irregular shape, and contains considerable water, which must be removed before vulcanization.

This washing process is omitted in the case of plantation rubber which has already been washed before shipment.

## 2. DRYING

There are two methods in use for removing the water from washed rubber. The first is to hang the rubber sheets in a warm dry place—usually the attic—steam-heated pipes being used to maintain the proper temperature during cold weather. By this means evaporation takes place slowly, and considerable time is consumed in removing the moisture. This method is usually employed in handling the better grades of rubber, as it produces better results than the second and quicker method, in which vacuum heaters are used. It is claimed by some that a vacuum heater softens and thereby injures the rubber. The rubber having been dried as described above, is “broken down” or worked through smooth steam-heated rolls, by which process it is rendered soft and plastic.

## 3. COMPOUNDING AND MIXING

The rubber is now in condition to be compounded or mixed with sulphur and mineral matter and with reclaimed rubber or rubber substitutes if such are used. It is important that the sulphur and mineral matter be free so far as possible from grit and other particles of foreign matter, and for this reason such materials should be carefully screened.

The ingredients required for a batch having been weighed out in the definite proportions to produce a compound of the desired quality, the mixing is done with smooth rolls operated as in the washing process. Both steam and water connections are provided so that the temperature of the rolls may be regulated to suit the condition of the rubber as it is being worked. The rubber gradually absorbs the sulphur and fillers which are added by an attendant. Such material as passes through without being incorporated with the rubber is collected in a pan and returned to the rolls. The temperature of the rolls is so regulated that as the operation of mixing proceeds the compound sticks to one of them in the form of a sheet. This sheet is cut with a knife, folded upon itself, and passed through the rolls again, the operation being repeated until the material shows a uniform color and is as nearly homogeneous as it is practicable to make it.

## 4. SHEETING

The next step in the process of manufacture depends upon the purpose for which the rubber is intended. If sheet rubber is being made, as for packing or for the tubes and covers of hose, the compound coming from the

mixing rolls is passed through calender rolls. The calender consists of three steam-heated rolls, one above the other, which are so geared together that the middle roll revolves in the opposite direction from that of the other two. The rolls may be adjusted to form sheets of different thickness. The skeleton diagram in Fig. 1 shows the method of operation.

Rubber is fed between the top and middle rolls, and by a proper regulation of temperatures the sheet adheres to the middle one while the top one remains clean. A strip of cloth is taken from the reel 1 and passed between the middle and bottom rolls to the reel 2. The sheeted rubber as it passes between the middle and bottom rolls is received by the cloth and carried to the reel 2, upon which they are wound together, the cloth preventing the layers of rubber from adhering. The sheet may be cut into strips of any desired width by knives which press against the middle roll.

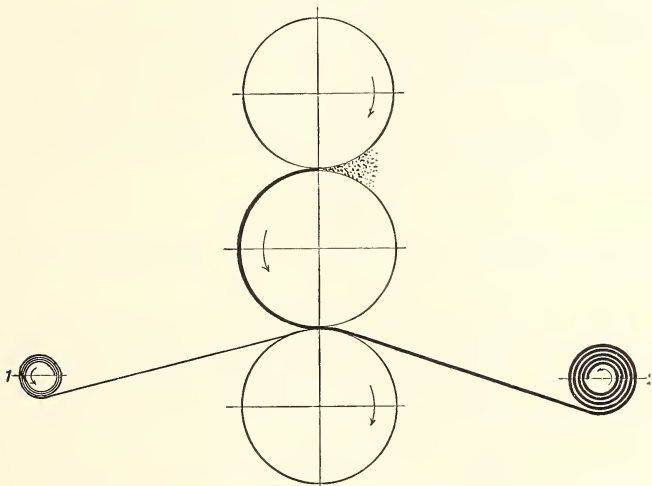


Fig. 1 —Diagram showing operation of calender rolls

Sometimes several calendered sheets are rolled together to form a single sheet. In the case of high-grade rubber hose, such as fire hose, the object is to avoid flaws in the finished sheet. The rubber is now ready to be vulcanized or worked into hose or other fabricated articles.

##### 5. "FRICTION"

What is known as "friction" in the case of rubber hose, rubber belting, and other articles which are made up with superimposed layers of canvas, is the soft rubber compound which is applied to the canvas and by means of which the different layers or plies are held together.

The canvas is first dried by being passed over steam-heated rolls, after which the friction is applied by means of rolls which are operated in the manner just described, and illustrated in Fig. 1.



The friction is fed between the top and middle rolls and forms a coating on the surface of the latter. The canvas to be frictioned is taken from a reel, passed between the middle and bottom rolls and wound onto a second reel on the opposite side of the rolls.

In the case of the friction calender, the bottom roll revolves at about two-thirds the speed of the middle roll, thus causing a wiping action which forces the friction well into the meshes of the canvas.

#### 6. CUTTING THE CANVAS

Canvas for use in making rubber hose is usually cut on the bias from strips 40 to 42 inches wide, into pieces long enough so that when placed end to end and lapped, the resulting strip is just wide enough to produce the necessary number of plies on the hose. There is no waste when cutting on the bias, and the finished hose is more flexible than when the canvas is cut straight. On the other hand, when the canvas is cut straight there is more or less waste on account of the last strip, which is often too narrow to be used. This method of cutting, however, produces the stronger hose, and a hose which will not expand as much, and which will elongate under pressure, avoiding the objectionable feature of longitudinal contraction which is noticed in hose made with bias-cut duck.

#### 7. RUBBER HOSE

The ordinary "plied" hose with rubber tube and cover is manufactured as follows:

##### (a) TUBES AND COVERS

For low-grade water hose of small diameter it is usual to form the tubes by passing the rubber compound through a die which may be adjusted to produce a wall of any desired thickness. The rubber coming from the mixing rolls must be at a sufficiently high temperature to make it plastic, in which condition it is forced through the die by means of a worm. The operation is similar to that of a "soft mud" brick machine, and the tube as it comes from the die is carried away on an endless belt. These tubes are placed on steel mandrels by a rather ingenious process, as follows:

The mandrel, which is about 52 feet long, is placed on an endless belt and held stationary. Powdered talc is blown into the tube to act as a lubricant and to prevent it from sticking to the mandrel during vulcanization. One end of the tube having been placed over the mandrel, air pressure is applied at the other end, sufficient to expand the tube slightly. The belt is now set in motion, and the tube as it is fed onto the belt floats over the mandrel on a cushion of air. In the case of high-grade hose and hose of large diameter, the tube is made from a strip of sheet rubber, cut with a "skive" or tapering cut, which is wrapped over the mandrel by hand, the edges being lapped and pressed flat by means of a small hand roller.

In either case the cover is made from a strip of sheet rubber just wide enough to pass once around the hose and form a narrow lap.

To ensure firm adhesion between the tube and canvas, the former is cleaned with gasoline, preparatory to receiving the frictioned canvas.



## (b) "MAKING UP" THE HOSE

Water hose of small diameter is usually wrapped by machinery consisting of three rolls about 2 inches in diameter and slightly more than 50 feet long. The two bottom rolls lie in the same horizontal plane and the top roll, which is just above and between the other two, may be raised while the mandrel carrying the tube to be wrapped is being placed on the bottom rolls. The top roll is now lowered onto the tube, which is held firmly between the three rolls. A rotary motion imparted to the rolls causes the tube to revolve, and the canvas and rubber cover are wrapped on in a few seconds. This method has the advantage of consuming very little time, but unfortunately it is not applicable to the construction of best-quality hose, which are made up by hand with the assistance of small rollers having a concave face. The rollers are run up and down the hose and serve to press each ply of frictioned canvas onto the next.

Before going to the vulcanizer the hose is wrapped with cloth. First, a long strip is wrapped lengthwise on the hose, and over this a narrow strip is wrapped spirally. This is done very rapidly by causing the hose to revolve in roller bearings while the narrow strip of cloth is held under tension and guided by hand. The operation requires only a few minutes.

## (c) VULCANIZING

The vulcanizer consists of a long cylinder provided with steam and drip connections, and a pressure gauge. The pressure and time necessary for vulcanization depend upon the composition of the rubber compound, the thickness, and the use for which the hose is intended. After vulcanization the wrapping cloth is stripped off, and the hose is removed from the mandrel by means of compressed air, in the same way that the tube was put on. The couplings are now put on and the hose is ready for shipment.

## (d) COTTON RUBBER-LINED HOSE

In the manufacture of woven cotton hose with rubber lining, the tube is made in the usual way and partially vulcanized, in order that it may develop sufficient strength to be drawn through the cover. A long slender rod is passed through the cover, carrying with it a stout cord. This cord is attached to the end of the rubber tube, and the rod is withdrawn. The cord is now drawn through the cover, bringing the tube with it, the tube having been coated with rubber cement. The hose is now filled with steam under pressure, which expands the tube, thus forcing the cement well into the meshes of the woven cover, and at the same time vulcanizes the rubber.

## (e) BRAIDED HOSE WITH RUBBER TUBE AND COVER

A form of braided hose which is claimed to have, and appears to have, decided merit, is made as follows:

The rubber tube passes first through a bath of cement and then to the braiding machine, where the first ply of fabric is braided over the fresh cement. This operation is repeated until the desired number of plies have

been formed, when the rubber cover is put on and the hose is vulcanized in a mold. While being vulcanized the hose is subjected to air pressure from within, which forces the rubber well into the meshes of the loosely braided fabric.

#### 8. RUBBER BELTING

Duck for rubber belting is passed over steam-heated rolls to remove the moisture, and frictioned as described in connection with the manufacture of rubber hose.

The frictioned duck is cut lengthwise into strips, the width of which depends not only upon the size of belt, but also upon the method of manufacture, which is not the same in all factories. These strips are cut by passing the canvas over a drum against which knives are held at points necessary to produce the desired widths.

One method is to make the inner plies of the belt with strips which are equal in width to that of the belt. These strips, stacked one above the other, are placed in the center of a strip of double the width, and in this position they are drawn through an opening with flared edges which folds the bottom strip over the top strips and forms a butt joint on the top face of the belt. The belt then passes between rolls which press the plies firmly together and at the same time lay and press a narrow strip of rubber over the joint. When the belt is to have a rubber cover, as is usually the case, this is calendered onto the outside ply or layer of the canvas before it is put on the belt. Some of the most expensive belts, however, are made without a rubber cover.

Another method is to cut each strip of canvas twice as wide as the belt. The first strip is folded upon itself, as described above, so that its edges form a butt joint. This folded strip is placed with its joint down upon the next strip, which is in turn folded to form a butt joint on the back of the first strip. In this way the belt is built up with the desired number of plies, the last joint being covered with a narrow strip of rubber, which is rolled flush with the surface. The belt is now ready to be vulcanized.

In this process there are two steps. First the closely coiled belt is wrapped so that only its edges are exposed, and in this condition it is put in the vulcanizer. After the edges have been vulcanized the belt is stretched and held under heavy pressure between the steam-heated faces of a long hydraulic press. This drives the friction into the pores of the duck and vulcanizes the belt throughout.

As regards the advantage of using a high-grade rubber cover for belting, the consensus of opinion seems to be that the expense thus incurred, except in the case of conveyor belting, had better be devoted to increasing the quality of friction between the plies of canvas.

#### 9. MECHANICAL RUBBER GOODS

The term "rubber," as commonly employed, does not refer to the commercially pure gum, but to a vulcanized compound as already described, which consists of gum, mineral matter or pigments and sulphur, mixed in

various proportions, according to the purpose for which it is intended. Mineral matter or the so-called fillers serve a very useful purpose, both in cheapening the product and in adding certain desirable properties which could not otherwise be obtained. Their presence, therefore, should not be looked upon as an adulteration.

There is a limited demand for pure gum by the medical profession and a very considerable amount is used in the manufacture of stationery bands, elastic thread, etc., but the amount of rubber thus consumed is insignificant as compared with the enormous quantity used in the manufacture of mechanical rubber goods, such as automobile tires, hose, packing, and footwear. The methods of compounding rubber with various materials available constitute the technique of the manufacturing industry, and this phase of the subject is beyond the scope of this circular. It may be of interest to those unfamiliar with the subject, to know that a properly vulcanized compound of high-grade rubber which is suitable for the best hose and packing, may be stretched to about seven times its original length and has a tensile strength of about 2000 pounds per square inch. On the other hand, there is an enormous demand for less expensive mixtures such as are used for garden hose and the lower grades of packing.

The properties that are desirable in rubber depend in a great measure upon the use for which it is intended. For example, rubber intended for steam hose or steam packing should be of a composition to withstand high temperatures, while rubber for the tread of an automobile tire should offer great resistance to abrasion.

The real value of rubber in any case depends upon the length of time that it will retain those properties which are desirable, and it is a matter of common observation that rubber often deteriorates less rapidly when in use than when lying idle. Deterioration as indicated by loss of strength and elasticity is considered to be the result of oxidation, which action is accelerated by heat and very greatly by sunlight. Other things being equal, the better grades of rubber possess greater strength and elasticity, and may be stretched to a greater extent than the poorer grades, and they also deteriorate less rapidly. The physical properties of rubber, however, are subject to variation within wide limits, depending upon the proportion of gum present, the materials used as fillers, and the extent of vulcanization.

### III. PHYSICAL TESTING OF RUBBER

Rubber testing in the present stage of its development is not susceptible of very great refinement as regards measurement. The nature of the material is such that refinement seems of less importance than uniformity of methods, which is absolutely essential where the work of different laboratories is to be compared. A more general interest in this matter would result in a substantial benefit not only to reputable manufacturers and large consumers, but also to the general public.



## 1. PHYSICAL TESTS MOST COMMONLY EMPLOYED

The different properties that have been found desirable in rubber intended for various purposes have naturally given rise to numerous tests, of which the most widely applicable are the various tension tests. These tests in various forms are used to determine the more important physical properties, such as tensile strength, ultimate elongation, elasticity, and reduction in tension when stretched to a definite elongation.

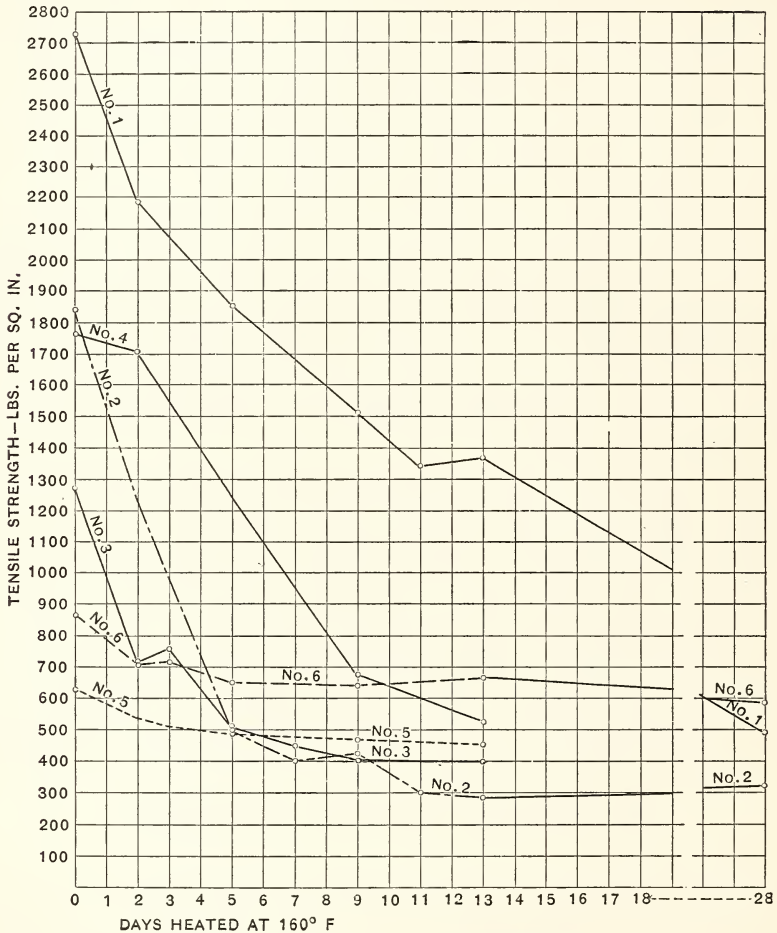


Fig. 2.—Effect of dry heat on the tensile strength of rubber

“Recovery” as applied to rubber is in a way synonymous with elasticity, and is measured by the extent to which the material returns to its original length after having been stretched. The term “set,” as commonly employed, refers to the extension remaining after a specified interval of rest following a specified elongation for a given period of time.



In the case of such materials as rubber hose and rubber belting, which are built up with layers of duck cemented or frictioned together with rubber, it is customary to determine the friction or adhesion between the plies of duck as well as the quality of rubber. It is also usual to subject hose (particularly fire hose and air hose) to a hydraulic pressure test, in order to detect any imperfections in materials or workmanship.

An important test in the case of steam hose consists in passing steam at about 50 pounds pressure through a short length of the hose in order to

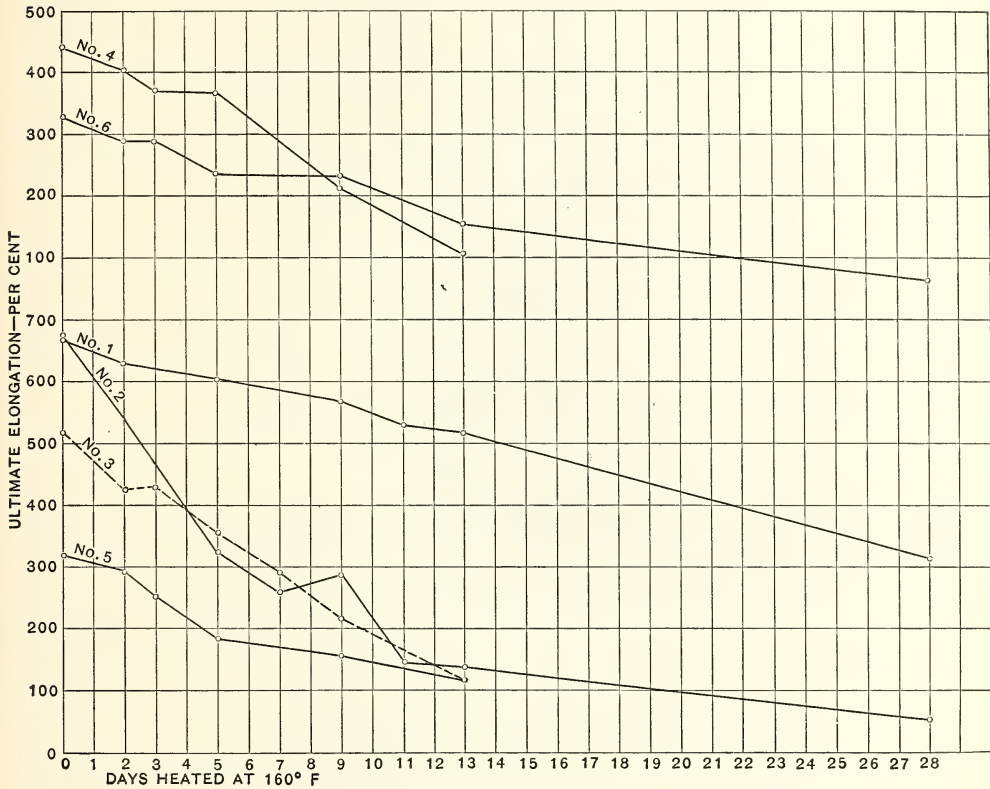


Fig. 3.—Effect of dry heat on the ultimate elongation of rubber

determine if the rubber is of suitable composition to withstand the effects of service conditions. This test usually lasts for about six days, the steam being turned off at night to allow the rubber to cool. A decided hardening or softening of the rubber, or a large decrease in the value of friction, as a result of steaming, is an indication of inferior quality.

No absolutely reliable test (other than an actual service test) has been devised for rubber steam packing, but in many cases valuable information may be obtained by clamping a piece of the packing between metal plates and subjecting it to the action of steam at a pressure equal to or slightly

above that under which it is to be used. A more satisfactory method is to clamp the packing in the form of a gasket between pipe flanges and apply the desired steam pressure from within. The test should last several days, the steam being turned off at night to see if the joint has a tendency to leak as a result of the cooling effect. This, however, practically constitutes a service test.

The testing of tires, or rather the materials used in their construction, is done almost exclusively by manufacturers. Manifestly it would be too expensive for the consumer, or even the dealer, to sacrifice whole tires for the purpose of securing test pieces. The more progressive manufacturers, however, realize that money expended in thorough and careful testing is more than justified by the increase in efficiency of their production.

The tests which have been outlined above will, in the majority of cases, enable one to form a fairly accurate judgment as to the quality of rubber. It sometimes happens, however, that rubber which shows evidence of good quality just after vulcanization is found to deteriorate rapidly with age. This has led to a number of proposed accelerated tests, whose common object is to hasten by artificial means the deteriorating effect of age. A test of this nature which merits further investigation consists in the application of dry heat, and a comprehensive series of experiments embracing different cures of various compounds is now being carried on at the Bureau of Standards. The effect of dry heat at 160° F on the tensile strength and ultimate elongation of six rubbers is shown in Figs. 2 and 3.

#### (a) TENSION TEST

When the material is made up with layers of fabric, as in the case of rubber hose, the first step in preparing specimens for the tension test is to separate the rubber from the fabric. Unless the frictioning is very poor this will necessitate the use of a solvent. If there is more than one layer of fabric, the easiest way is to remove the first layer along with the rubber. The rubber is then separated from the adjoining layer of fabric by means of gasoline blown from a wash bottle. Narrow strips are more easily handled than larger pieces and there is less danger of injuring the rubber. Great pains should be taken during this operation because any flaw or local imperfection will seriously vitiate the results. The rubber should be allowed to rest for several hours in order that it may recover from the stretching it has received and that the gasoline may thoroughly evaporate.

Test pieces are cut with a metal die, which not only saves much time but also insures uniform width which it is impossible to obtain if the specimens are cut by hand. An arbor press is perhaps the most convenient and satisfactory means of forcing the die through the rubber, although many prefer to cut the test pieces by striking the die with a mallet. The operation is facilitated by wetting the cutting edges of the die, and the rubber should rest on a smooth and slightly yielding surface which will not injure the blades. A piece of leather or wood (cut across the grain) is suitable for the purpose. The central portion of the test piece is straight for a distance

of 2 inches, and the ends are enlarged to prevent tearing in the grips of the testing machine. The width of the contracted section is usually made either one-fourth inch or one-half inch. It is impossible to obtain satisfactory specimens one-half inch wide from hose of small diameter.

Parallel lines 2 inches apart are placed on the specimens, and by means of these gauge marks elongation and permanent extension are measured. A stamp consisting of parallel steel blades enables one to mark very fine lines with ink, without cutting the rubber, and in this way much time is saved and all chance of error eliminated. Test pieces in the form of a ring will be referred to later.

There is a special form of micrometer caliper (Fig. 4, A) provided with enlarged contact surfaces and ratchet stop which is well suited for measuring the thickness of rubber and textile materials. The ratchet stop makes it possible to measure each specimen under the same pressure and thus adds to the uniformity of results. A spring micrometer (Fig. 4, B) is exceedingly convenient and is very easily read.

In testing rubber one of the greatest difficulties has been to grip the test piece in such a way as to prevent slipping, without at the same time injuring the rubber. Even a very small scratch on the surface of a rubber test piece is often sufficient to cause failure at that point.

In order to prevent slipping of the test piece as its section is gradually reduced under increasing tension, it has been found advisable to provide means for automatically tightening the grip. This is conveniently accomplished by using a cylindrical roller mounted eccentrically as illustrated in Fig. 7 A, and when the rubber varies in thickness, as is often the case, it is an advantage to use a number of thin cylindrical disks (Fig. 7, B), which act independently, thus producing a uniform pressure over the gripping surface and preventing any uneven slipping. The machines shown in Fig. 5 are used for determining tensile strength and ultimate elongation.

In Fig. 5, H, the dynamometer 1, having a capacity of 200 pounds and graduated to 0.5 pound, is attached to the upper end of column 2, which is slotted to receive the rack 3, carrying the eccentric grip 4 at its upper end. The rack is operated by the handwheel 5, which is geared to a spur (not

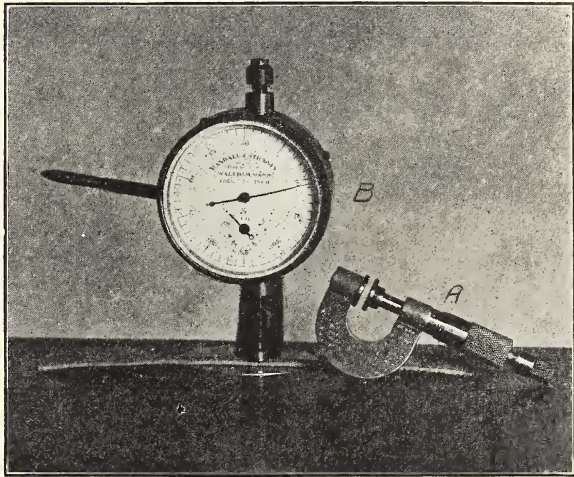


Fig. 4.—Instruments for measuring the thickness of rubber  
A.—Micrometer caliper  
B.—Spring micrometer



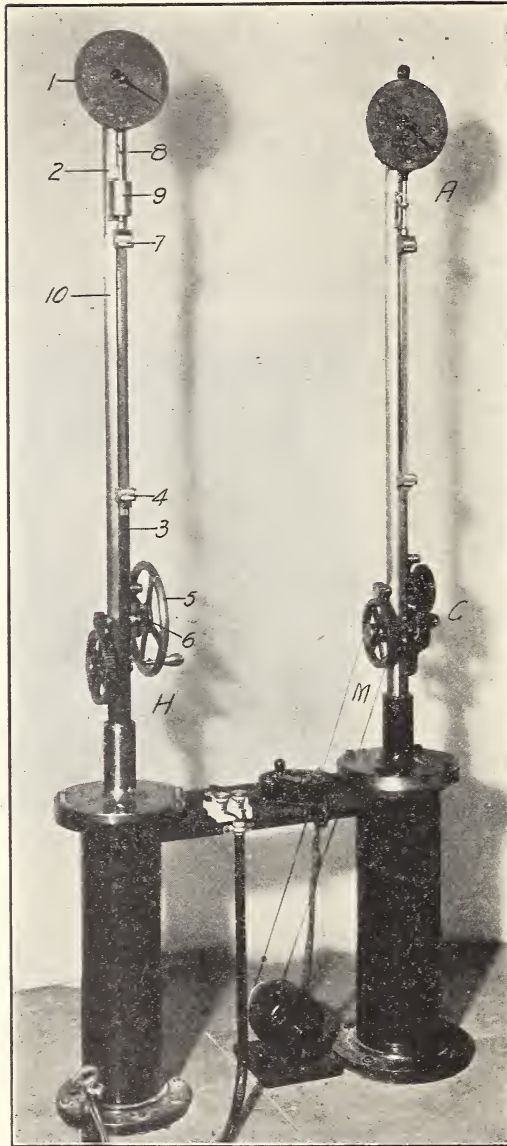


Fig. 5.—Two machines for determining the tensile strength of rubber

H.—Hand driven

M.—Motor driven

For details see Figs. 6 and 7



shown) inside of column 2. The shaft carrying the handwheel is provided with a ratchet wheel 6, by means of which the rack may be held in any position when it is desired to hold the test piece under tension. The eccentric grip 7 is connected to the dynamometer through the universal joint 8, the dash pot 9 serving to cushion the recoil of the springs when a specimen is broken. The dynamometer is so constructed that the pointer remains at the maximum load when a specimen breaks, and is moved back to zero at the beginning of each test. Elongation between gauge marks on the specimen is measured on the scale 10 attached to column 2.

In Fig. 5, M, the dynamometer is of 100 pounds capacity and is graduated to 0.25 pound. The construction of this machine is identical with that of the one just described except that it is motor driven, and that instead of a dash pot to cushion the recoil of the springs, the springs are held under tension when the specimen is broken by means of the plate 11, which acts in the following manner (see Fig. 7, A):

The rod 12, which is rigidly attached to column 2, passes with very little clearance through a hole in the back of plate 11. The front of plate 11 is slotted to receive the rod 13, and is supported by the lugs 14. As tension is applied to the specimen, plate 11 is free to follow the lugs 14, and passes freely down over the rod 12, but when the specimen breaks, the upward pressure of the lugs 14 causes the plate to bind on the rod 12, thus holding the springs under the maximum tension. The load having been recorded, the upper grip is pulled downward to relieve the pressure of lugs 14 against the plate 11, and at the same time the rear end of the plate is raised to release the rod 12. The rod 13 is now free to rise and the pointer may be returned to zero.

The stepped pulleys provide for different speeds to meet the requirements of experimental work. At the end of a test the worm and gear are disengaged by means of the hook 15 (see Fig. 6), and the rack is rapidly raised by hand to its initial position.

Fig. 8 shows a motor-driven machine of 15 pounds capacity which was designed for testing rubber bands. The load is applied through the steel tape 1, at the end of which is the grip 2. The graduated steel tape 3, attached to grip 2, with its zero point coinciding with the center of the spool 4, passes up behind the test specimen and through the column 5 to a reel just behind the spring balance. When the specimen breaks its elongation is determined by the distance between the centers of spools as shown by the tape 3. The plate 6 holds the springs under the maximum tension in a way already explained in connection with Fig. 7, A. Stepped pulleys provide for different speeds. When a specimen has been broken, the worm and gear are disengaged by means of the lever 7, and the bottom spool is raised by hand to its initial position.

This machine is also provided with eccentric grips which are used for testing straight specimens of low tensile strength.

For merely stretching rubber with a view to determining its elasticity or recovery after a definite elongation, without reference to the tension

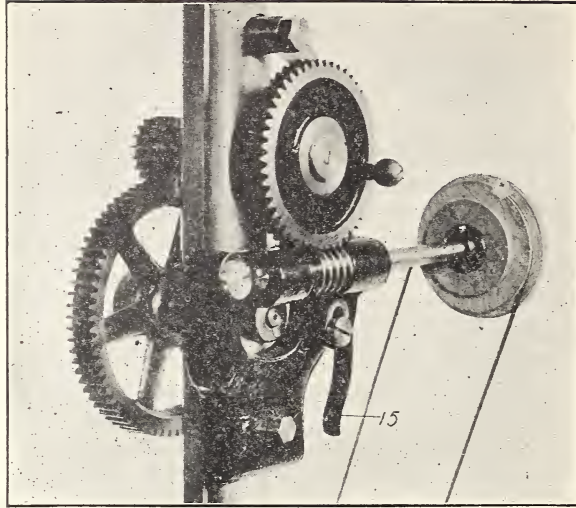


Fig. 6.—Detail C of motor-driven machine M shown in Fig. 5

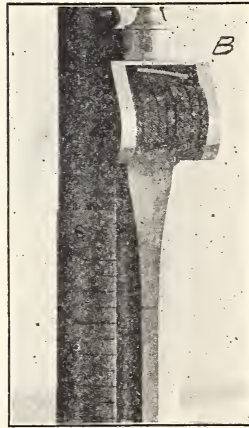
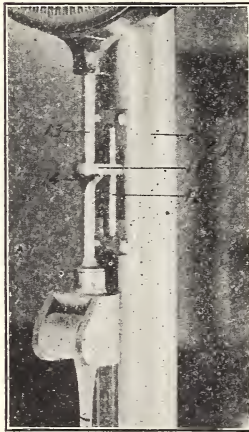


Fig. 7.—Details of motor-driven machine M shown in Fig. 5

applied, the apparatus shown in Fig. 9 is used, in which six specimens may be tested at once. The spools 1 are free to slide on the shaft 2, and are slotted to engage pins 3 (not shown), which act as clutches. The movable grips are attached to three-fourths inch strips of leather belt lacing which pass through clamps 4, and then to the spools 1. The action of these clamps is similar to that of an ordinary letterpress, and with a one-half inch by

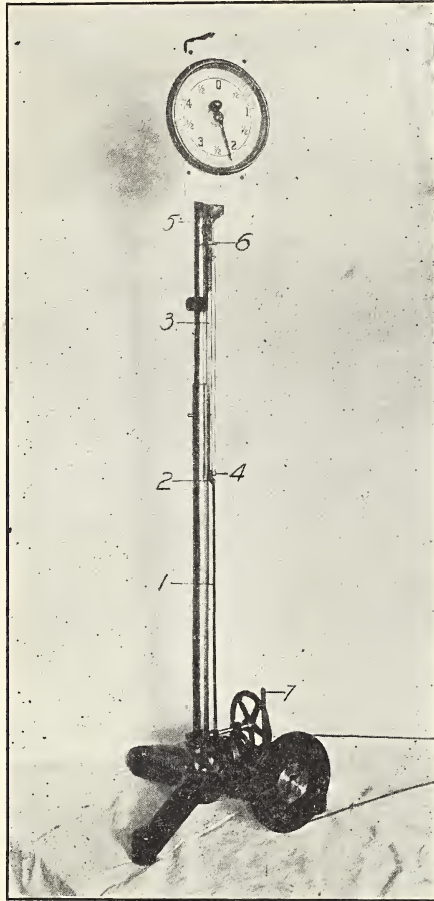


Fig. 8.—Motor-driven machine for testing rubber bands

three-fourths inch bearing plate, a moderate twist of the knurled head is sufficient to prevent any slip of the belt lacing when under tension. The operation of the apparatus is as follows:

Six specimens being in the grips, one of the spools is moved along the shaft until it engages the corresponding pin, and the shaft is revolved until the desired elongation measured between gauge marks on the specimen



is secured. The clamp is tightened to hold the specimen in this position, and the spool is shifted back so as to disengage the pin. The operation is repeated with each of the specimens in turn. Each specimen is released after a specified length of time (usually from 1 to 10 minutes) and after an equal interval of rest the permanent extension or set is measured.

The apparatus shown in Fig. 10, which has a capacity of four specimens, is used in observing the reduction in tension when rubber is held at a definite elongation. The spring balances are provided with live and dead pointers which show the maximum tension as well as the tension at any time during the test. The balances have a capacity of 50 pounds, with 8-inch dials graduated to 0.2 pound. The lower grips are counterbalanced by weights (not shown) which are suspended from cords passing over pulleys just back of the spring balances. In this way each grip is held in an accessible position and is prevented from falling should the specimen break.

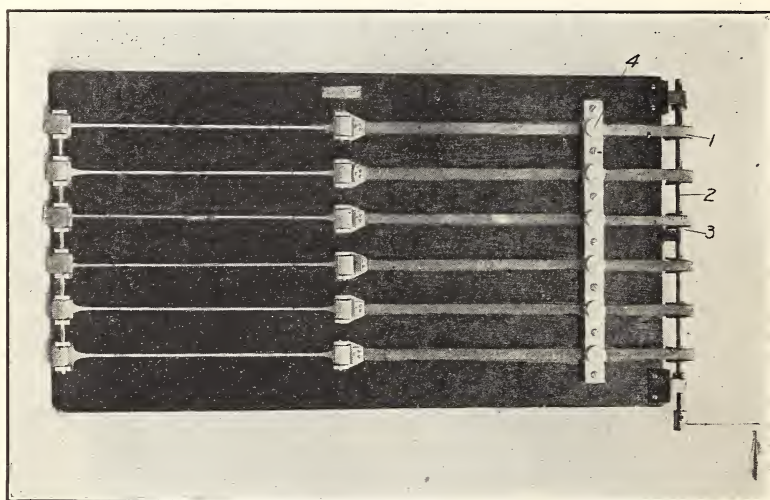


Fig. 9.—Apparatus for stretching rubber to determine its elasticity

(b) CONDITIONS AFFECTING THE RESULTS OF TENSION TESTS

In the absence of uniform methods of testing it is found that results obtained in different laboratories sometimes show marked discrepancies which are due to the varying conditions under which the tests are made.

(1) **Influence of Speed on Tensile Strength and Elongation.**—The speed at which rubber is stretched probably affects the results to a less extent than is often supposed, though doubtless different rubbers are not equally affected.

The results tabulated below were obtained in a comparative test on a limited number of specimens, but a thorough investigation is now being carried on with a view to securing complete data on the subject.



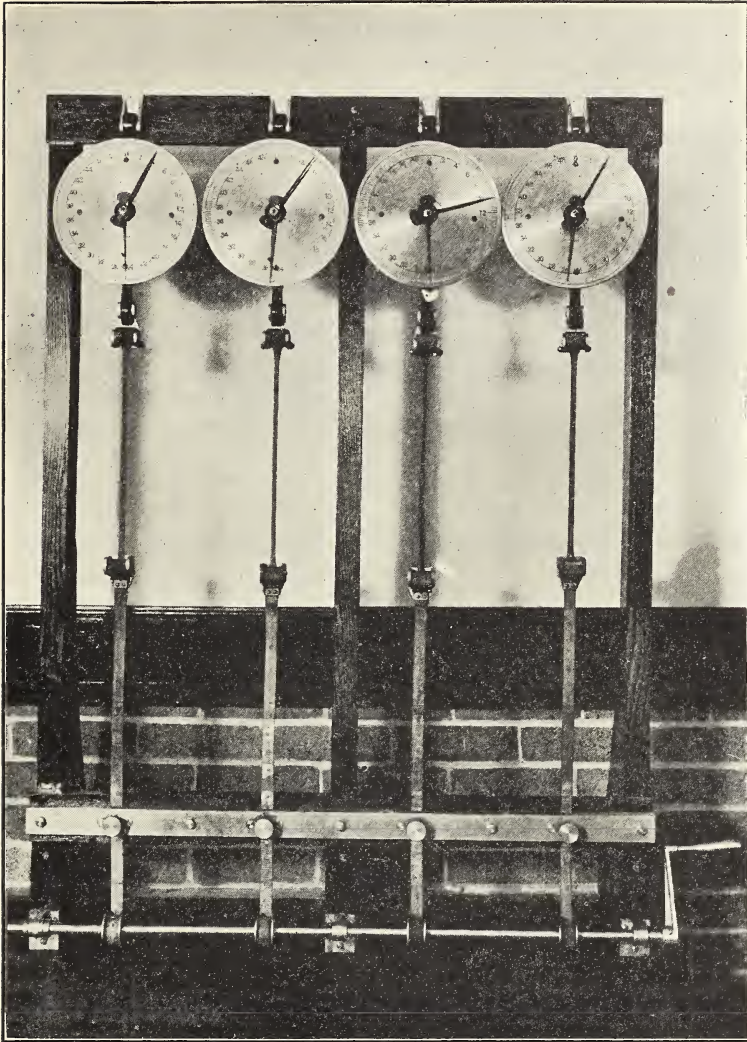


Fig. 10.—Apparatus for determining the reduction in tension when rubber is stretched and held at a definite elongation

TABLE I

Showing Strength and Elongation of Rubber when Stretched at the rate of 30 and 120 inches per minute

[Gauge length=2 inches.]

Rubber No.....	2		3		4		5		6	
Speed in inches per minute.....	30	120	30	120	30	120	30	120	30	120
Tensile strength (pounds per square inch) .....	1740	1690	990	1100	1710	1790	750	920	930	1030
Ultimate elongation (per cent).....	665	670	510	530	460	460	430	430	375	380

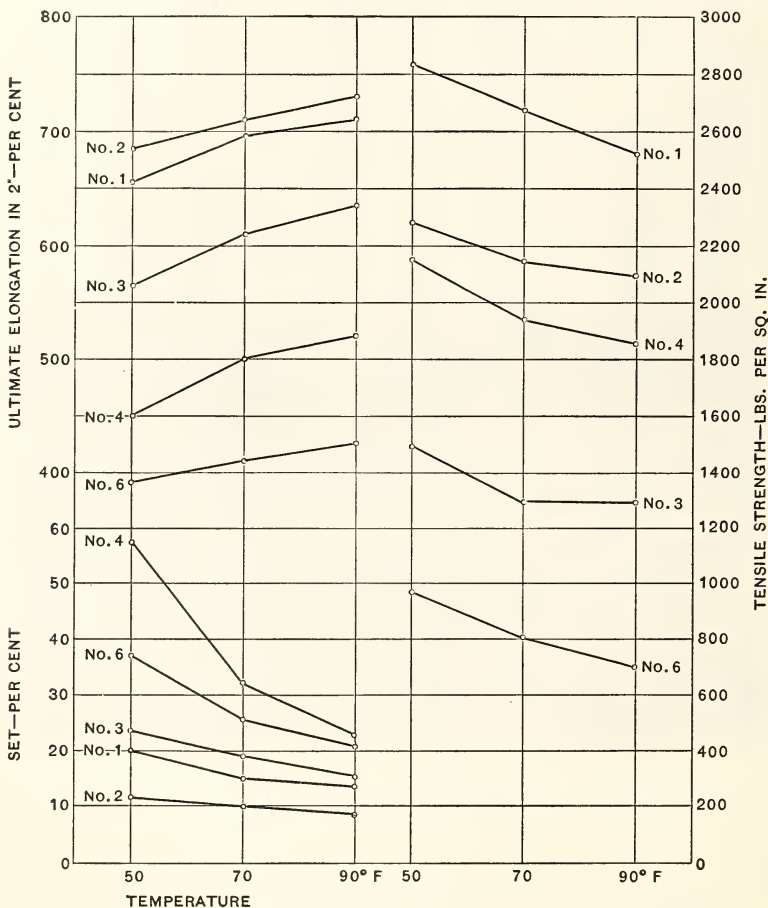


Fig. 11.—Influence of temperature on the tensile strength, elongation, and recovery of rubber

These results would indicate that elongation is not appreciably affected by speed, and that for the lower-grade rubbers greater tensile strength is secured at high speed.

(2) **Influence of Temperature on Strength, Elongation, and Recovery.**—It is generally recognized that the physical properties of rubber are affected by changes in temperature, though, of course, to a less extent after vulcanization than before.

Figure 11 shows the results of tests at 50, 70, and 90 degrees Fahrenheit. In each case the room was maintained at the specified temperature for three hours before the tests were made. It will be noticed that the rubbers are not all affected to the same extent by equal differences in temperature, but there is a marked tendency in each case toward decreased strength, decreased set (increased elasticity), and increased elongation as the temperature is raised. It will be noted further that in nearly every case, greater differences are secured between 50 and 70 degrees than between 70 and 90 degrees.

The set in each case was measured after one minute stretch and one minute rest. Nos. 1 and 2 were stretched 350 per cent, Nos. 3 and 4, 300 per cent, and No. 6, 250 per cent.

(3) **Influence of Cross Section on Tensile Strength and Elongation.**—Tensile strength and ultimate elongation are theoretically independent of sectional area, but as in other materials there is a tendency for small test pieces to develop higher unit values than large ones. Complete data on this subject is not at hand, but it is thought that test pieces one-fourth inch and one-half inch wide will show but little difference in unit strength and elongation, provided the surface is uniform and the wider specimens are sufficiently enlarged at the ends to prevent tearing in the grips.

(4) **Influence of the Direction in which Specimens are Cut on Strength, Elongation, and Recovery.**—The tensile properties of sheet rubber are not the same in all directions, as will be shown later in connection with comparative tests on straight and ring-shaped test pieces. It will be mentioned here that specimens cut longitudinally or in the direction in which the rubber has been rolled through the calender show greater strength and (at least for the better grades of rubber) less elongation than specimens cut transversely or across the sheet. The recovery, however, is greater in the transverse direction. Table I illustrates this.

No. 3 shows greater strength transversely and No. 5 greater elongation longitudinally, while No. 6 shows the same elongation in each direction. The exception noted in No. 3 is attributed to experimental errors or to small defects in the specimens which escaped detection. It may be, however, that low-grade rubbers, such as Nos. 5 and 6, are not more extensible transversely than longitudinally. Further tests are necessary to determine this point.

(5) **Influence of Previous Stretching on Strength, Elongation, and Recovery.**—Previous stretching seems not only to increase the ultimate elongation, as is generally known, but also the tensile strength, at least in the case of high-grade compounds.



TABLE II

Showing the Relative Strength, Elongation, and Recovery of Rubber when tested in the Longitudinal and Transverse Directions

Rubber No.....	1	2	3	4	5	6
Tensile strength <sup>1</sup> (pounds per square inch):						
Longitudinal.....	2730	2070	1200	1850	690	880
Transverse.....	2575	2030	1260	1700	510	690
Ultimate elongation (per cent):						
Longitudinal.....	630	640	480	410	320	315
Transverse.....	640	670	555	460	280	315
Set <sup>1</sup> after 300 per cent elongation for 1 minute with 1 minute rest (per cent):						
Longitudinal.....	11.2	6.0	22.1	34.0	27.5	34.3
Transverse.....	7.3	5.0	16.3	24.0	25.0	25.9

<sup>1</sup> The set and tensile strength were determined with different test pieces.

Table III gives the tensile strength and ultimate elongation obtained in testing six samples of rubber, first, with a single stretch, and, second, by repeated stretching, beginning with 200 per cent and increasing each stretch by 100 per cent until failure.

TABLE III

The Influence of Repeated Stretching on Tensile Strength and Ultimate Elongation

Rubber No.....	1	2	3	4	5	6
Tensile strength (pounds per square inch):						
Single stretch.....	2470	1740	990	1710	750	930
Repeated stretch.....	2610	1960	1180	1790	790	920
Ultimate elongation (per cent):						
Single stretch.....	645	665	510	460	430	375
Repeated stretch.....	765	780	645	555	440	465

The recovery after a definite elongation is usually greater if the rubber has been previously stretched than if determined in the usual way. This is illustrated by the results shown in Table IV, in which the columns marked "Repeated stretch" show the set after repeated stretching, beginning with 100 per cent and increasing 100 per cent for each subsequent stretch. The results in columns marked "Single stretch" were obtained in the usual way, each specimen being stretched but once. In each case the set was measured after one minute stretch and one minute rest, the tabulated results being the average of a number of observations.

TABLE IV

The Influence of Repeated Stretching on the Recovery of Rubber

No.	Method of Testing	Per cent set after being stretched—				
		100 per cent	200 per cent	300 per cent	400 per cent	500 per cent
1	Repeated stretch.....	1.0	4.5	9.5	16.0	25.0
	Single stretch.....			11.7	19.8	29.0
2	Repeated stretch.....	1.8	4.0	7.7	13.7	21.2
	Single stretch.....			8.0	14.7	21.5
3	Repeated stretch.....	3.7	9.0	17.7	27.0	37.0
	Single stretch.....			21.7	34.0	47.0
4	Repeated stretch.....	4.0	12.3	28.7	48.7	.....
	Single stretch.....		14.3	33.0	56.0	.....
5	Repeated stretch.....	8.1	19.4	34.0	.....	.....
	Single stretch.....		19.3	33.0	.....	.....
6	Repeated stretch.....	4.3	16.3	34.0	.....	.....
	Single stretch.....		17.0	35.3	.....	.....

It will be noted that the effect of previous stretching is very marked in the case of Nos. 1, 3, and 4; that it is very slight in the case of Nos. 2 and 6; and that in the case of No. 5 the set is slightly increased by previous stretching.

(6) **Influence of the Form of Test Specimen on the Results of Tension Tests.**—There is a wide difference of opinion in regard to the relative merits of the straight and ring-shaped test specimen. The ring, which is highly recommended by some, undoubtedly possesses certain advantages as regards convenience in testing, and uniform results may be obtained by this method.

Ring specimens, however, do not show the full tensile strength of rubber, on account of the uneven distribution of stress over the cross section. This fact is evident from a simple analysis, and may be verified by comparative tests with straight and ring shaped test pieces, provided the straight test pieces are sufficiently enlarged at the ends to prevent failure in the grips, and provided further that the change in width is not made too abruptly.

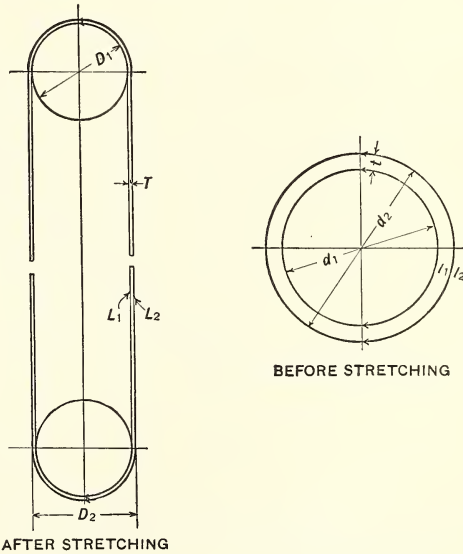


Fig. 12.—Ring test piece before and after being stretched

Assuming for simplicity that the extensibility of rubber is the same in all directions, it will be seen by reference to Fig. 12, which represents a ring test piece before and after extension that:

If  $E_1$  = per cent elongation of the inside surface at breaking point (automatically measured),

$$\text{then } E_1 = \frac{L_1 - l_1}{l_1} \cdot 100 \dots \dots \dots (1)$$

If  $E_2$  = per cent elongation of the outside surface when specimen breaks,

$$\text{then } E_2 = \frac{L_2 - l_2}{l_2} \cdot 100 \dots \dots \dots (2)$$

$$L_2 = L_1 + \pi/2 (D_2 - D_1) = L_1 + \pi T \dots \dots \dots (3)$$

$$T = t \sqrt{\frac{l_1}{L_1}} \text{ approximately } \dots \dots \dots (4)$$

(assuming that the volume of rubber is constant.)

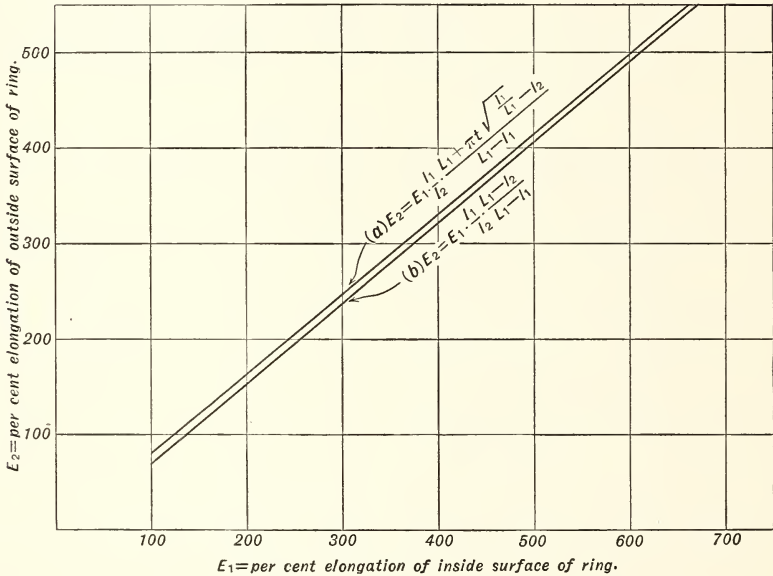


Fig. 13.—Relation between the elongation at the inside and outside of a ring

From equations 1, 2, 3, and 4, we have

$$E_2 = E_1 \cdot \frac{l_1}{l_2} \cdot \frac{L_1 + \pi t \sqrt{\frac{l_1}{L_1}} - l_2}{L_1 - l_1} \dots \dots \dots (5)$$



which is represented graphically in Fig. 13 (a) for the usual size of ring in which  $l_1 = 70$  mm and  $t = 4$  mm.

This relation is practically a linear one and  $E_2 = 0.83E_1$  approximately.

Fig. 13 (b) shows the slight error introduced by neglecting the term  $\pi t \sqrt{\frac{l_1}{L_1}}$

Now, since the percentage of elongation at the outside surface of the ring

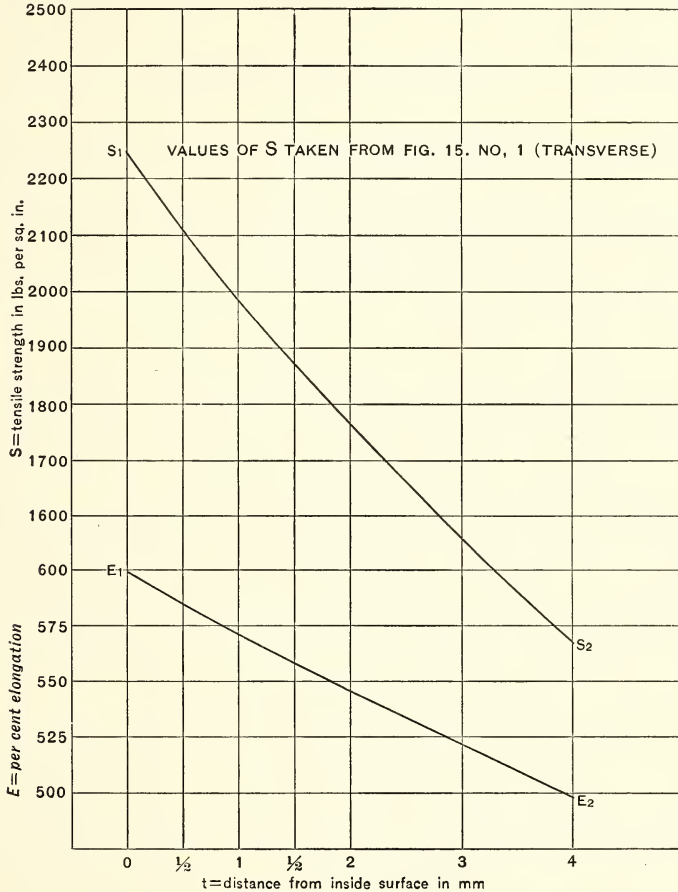


Fig. 14.—Variation in elongation and tensile strength from the inside to the outside of a ring

is less than at the inside surface, the tensile stress must also be less at the outside than at the inside surface. From equation (5) it follows that the decrease in the per cent of elongation is approximately uniform from the inside to the outside of the ring, this relation being shown in Fig. 14 for  $E_1 = 600$  per cent and  $l_1 = 70$  mm. The relation between stress and elongation being practically a linear one for values of elongation near the breaking point, the decrease in tensile stress must also be fairly uniform from the inside to the

outside of the ring at the time of failure. This is illustrated graphically in Fig. 14, in which the values for tensile stress were taken from the stress-strain diagram shown in Fig. 15, No. 1.

If  $S_1$  = the stress at the inside surface of the ring at failure, or true tensile strength of the rubber corresponding to  $E_1$ , and  $S_2$  = the stress at the outside surface, corresponding to  $E_2$ , and  $S$  = the average stress over the cross section of the ring, which is the value for tensile strength obtained by the ring method, we have

$$S = \frac{\text{Breaking load}}{\text{Area of section}} = \frac{S_1 + S_2}{2} = \frac{S_1}{2} \left( 1 + \frac{S_2}{S_1} \right) \text{ approximately} \dots\dots\dots (6)$$

Now, since the ratio  $\frac{S_2}{S_1}$  varies for different rubbers,  $S$  does not bear a constant ratio to, and therefore can not be taken as, a measure of tensile strength. Elongation, however, is measured at the inside surface of the ring and therefore represents the maximum extension of the rubber. The average elongation over the cross section of the ring is,

$$E = 1/2 (E_1 + E_2) \text{ approximately} \dots\dots\dots (7)$$

If the extensibility of rubber were the same in all directions, values of  $S$  and  $E$  obtained from equations (6) and (7) would, theoretically, give a point lying very near the stress-strain curve for the same rubber tested in the form of a straight specimen. This, however, is not the case, as has been already pointed out, and as may be seen from Fig. 15.

The difference between  $S$  and  $S_1$  is greater for high grade rubbers than for compounds of poor quality, as may be seen by reference to Fig. 15, which represents stress-strain curves plotted from the results of tests on straight and ring specimens. Table V shows values for tensile strength and ultimate elongation obtained for the same rubbers by the two methods.

The ring test piece obviously does not give a true stress-strain curve on account of the varying stress over its cross section.

The straight specimens were cut both longitudinally and transversely with reference to the direction in which the rubber had been passed through the calender rolls. They were tested with the machine shown in Fig. 5, M, and the ring specimens were tested with a Schopper machine, Fig. 16. In each case the specimens were stretched at the rate of about 8 inches per minute. A number of test pieces, both straight and ring shaped, particularly in the case of No. 3, showed abnormally low tensile strength and elongation on account of small holes or particles of grit at the point of rupture. Such specimens are not included in the results tabulated above; each of which represents the average of from 5 to 15 tests.

A line was drawn across each of the ring specimens to indicate the longitudinal direction, and the point of failure was noted. There was a tendency for the rings to rupture along this line, thus indicating that the sheets were strongest longitudinally, or in the direction of rolling. This difference in strength is shown by the straight test pieces, except in the case of rubber

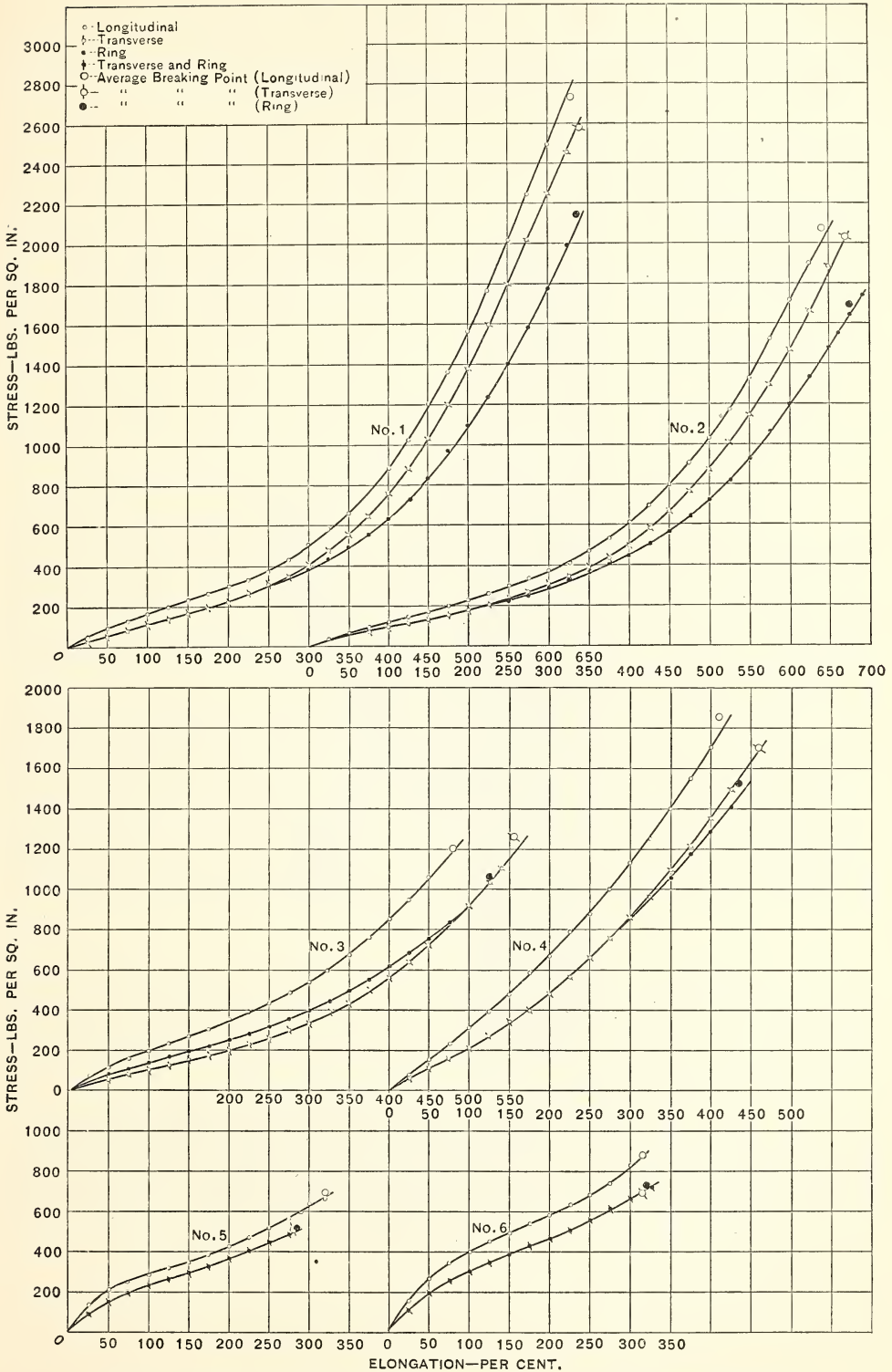


Fig. 15.—Stress-strain curves for rubber tested with straight and ring-shaped specimens



TABLE V

Results Showing the Relative Strength and Elongation of Rubber Tested with Straight and Ring Shaped Specimens

Rubber compound No.....	1	2	3	4	5	6
<b>Tensile strength (pounds per square inch):</b>						
Straight specimens—						
Longitudinal (L) <sup>1</sup> .....	2730	2070	1200	1850	690	880
Transverse (T).....	2575	2030	1260	1700	510	690
Ring specimens (R).....	2140	1690	1060	1520	510	730
R/L.....	0.78	0.82	0.88	0.82	0.74	0.83
R/T.....	0.83	0.83	0.84	0.89	1.00	1.06
<b>Ultimate elongation (per cent):</b>						
Straight specimens—						
Longitudinal.....	630	640	480	410	320	315
Transverse.....	640	670	555	460	280	315
Ring specimens.....	635	675	525	435	285	320

<sup>1</sup> Longitudinal—indicates the direction in which the rubber has been passed through the calender rolls.

No. 3. It is seen from Fig. 15 that the curve for transverse specimens lies below that for longitudinal specimens, thus showing that a given stress will produce a greater elongation if applied transversely than if applied longitudinally. It is to be expected, therefore, that the elongation of a ring will be less than that for a transverse straight specimen. The natural variation in rubber, however, is often sufficient to obscure small differences in elongation due to the methods of testing.

In the case of Nos. 5 and 6, the curves for the ring specimens practically coincide with those for the transverse straight specimens, and the tensile strength of these rubbers when tested by the two methods is seen to agree fairly well. It is to be noted, however, that for the higher grade rubbers the difference in tensile strength by the two methods is very marked. Although the difference is not great, there is a tendency for the transverse specimens to show a greater ultimate elongation than the longitudinal specimens, notwithstanding the greater strength shown in the latter case.

#### (C) FRICTION TEST

The "friction" or adhesion between the plies of canvas on rubber hose and between the canvas and the rubber tube and cover, is of great importance; in fact, the life of hose depends in great measure upon the efficiency of this adhesion. The same is true and to an even greater extent in the case of rubber belting.

The friction of "plied" hose is determined in the following manner (Fig. 17): In preparing test pieces, a short length of hose is pressed tightly over a slightly tapered mandrel, such as the ones shown. The mandrel is put in a lathe, and 1-inch rings are cut with a pointed knife. Beginning at the lap a short length of canvas is separated and the ring is pressed snugly

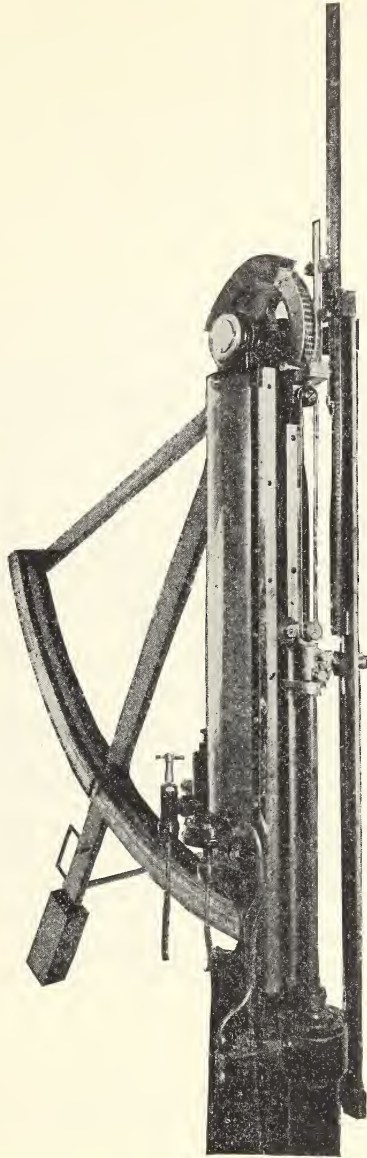


Fig. 16.—“Schopper” machine for testing rubber

This machine is worked by hydraulic power, its operation, briefly stated, being as follows:

The rubber test ring is placed over the spools and the lower spool is geared to the rack in such a way that it is caused to revolve during a test. This motion is transmitted to the top spool by the rubber test ring, the object of rotating the spools being to equalize the tension at all parts of the specimen. As the tension is increased, the weighted lever, to the short arm of which the top spool is attached, is gradually deflected. When the test ring is broken the lever is held at the point of maximum load by means of a set of pawls, the breaking load being read from the curved scale and the elongation being indicated by the vertical scale just opposite the test ring.

over a mandrel which is free to revolve in roller bearings. The rate at which the canvas strips under the action of a specified weight suspended from its detached end is taken as a measure of the friction.

A marked difference is often found in the friction between different plies of the same hose, as well as at different points along the same ply. Uniformity in the friction is desirable.

The results of this test are influenced by the temperature conditions, the rate of stripping caused by a given weight being greater at high than low temperatures. Also, the rate of stripping is greater if the mandrel

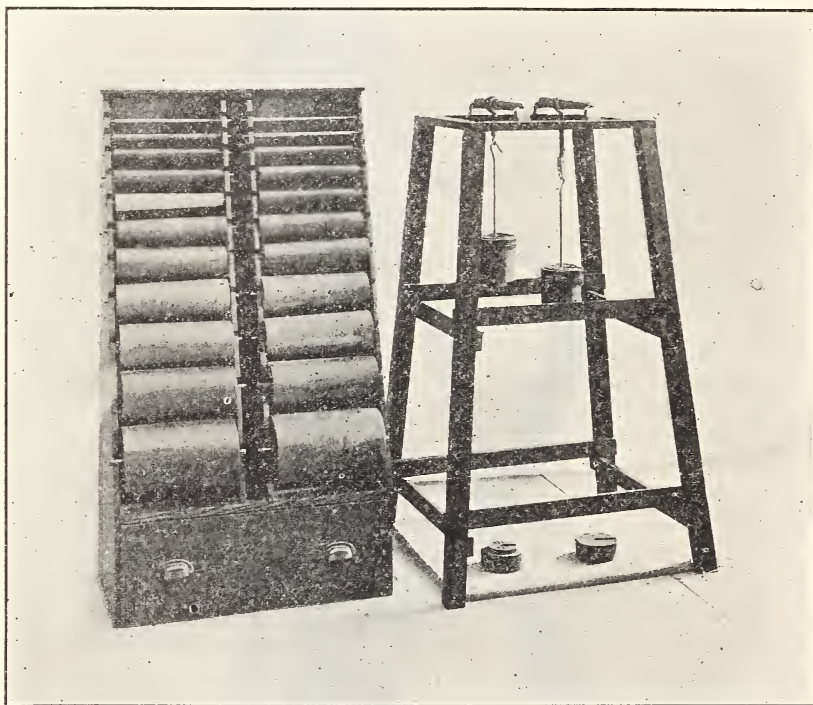


Fig. 17.—Apparatus for testing the "friction" of rubber hose

fits snugly in the ring than if the ring is allowed to sag over a loose mandrel. The variation in friction, however, in the same hose is often such as to obscure these influences unless observations are made under conditions which differ greatly.

In connection with this test, attention may be called to a point which, though generally recognized, is sometimes lost sight of in the interpretation of results.

It has been observed that no stripping is produced by increasing the weight up to a certain point, after which the rate of stripping increases gradually at first, and then more rapidly, with small increments in weight, until finally a very small increase in weight causes a large change in the

rate of stripping. The general behavior is illustrated graphically in Fig. 18, in which each point represents the average of a number of tests on a very uniformly frictioned hose.

As a result of this behavior, an air hose, for example, which is required to show a rate of stripping not exceeding 6 inches in 10 minutes under 25 pounds, might be regarded as of very inferior quality if it stripped, say,

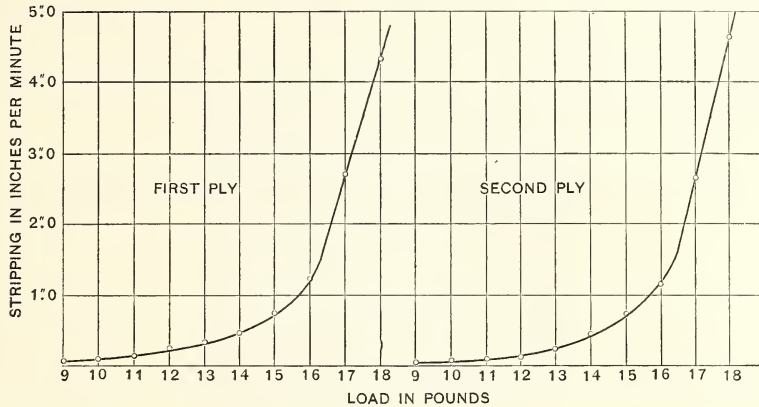


Fig. 18.—“Friction” test of rubber. Curves showing rate of stripping under different loads

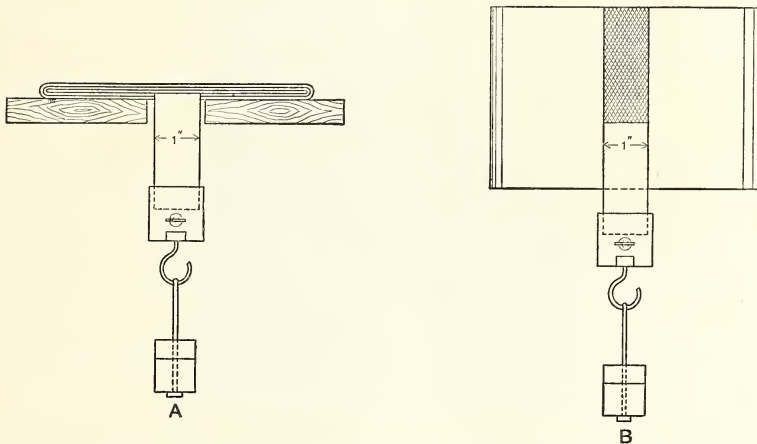


Fig. 19.—Two methods of testing the “friction” of rubber belting

20 inches in 10 minutes, whereas the same hose would probably show little or no stripping under 20 pounds, and come within the required limit under 22 pounds.

The “friction” of rubber-lined fire hose is usually determined as follows: A 1-inch strip is cut and a portion of the tube separated from the jacket. The detached end of the jacket is clamped in a stationary grip and the weight is suspended from the rubber tube.



The "friction" between the plies of duck in rubber belting is sometimes measured in the same way (Fig. 19, B), but some prefer to apply the load in a direction at right angles to the plane of separation, as in the case of "plied" hose. This is done by cutting the belt about halfway through along parallel lines 1 inch apart. The belt rests on horizontal supports just outside of the strip which has been cut, and the weight is suspended from the detached end of the duck (Fig. 19, A). It is found that for a given weight the rate of stripping is decidedly greater by the former method than by the latter.

Table VI gives comparative results obtained by the two methods in the case of a six-ply belt.

TABLE VI  
Showing Comparative Values of "Friction" by Different Methods  
[Inches stripped per minute.]

Weight (pounds).....	12	15	18	21
<b>First ply:</b>				
Tested as in Fig. 19, <del>X</del> B.....	0.08	0.26	1.26	3.56
Tested as in Fig. 19, <del>X</del> A.....				0.11
<b>Second ply:</b>				
Tested as in Fig. 19, <del>X</del> B.....	0.07	0.48	2.18	7.65
Tested as in Fig. 19, <del>X</del> A.....				0.15
<b>Third ply:</b>				
Tested as in Fig. 19, <del>X</del> B.....	0.04	0.32	1.33	7.00
Tested as in Fig. 19, <del>X</del> A.....				0.16

(d) HYDRAULIC PRESSURE TEST

The pressure test as usually made consists simply in subjecting a short length of the hose to water pressure created by a force pump of any convenient type. The coupling at the free end is closed with a plug and the pump connection is made with a reducing coupling. By using two clamps at each end it is possible to make a tight joint even under high pressure. It is necessary to provide a check valve to protect the pressure gauge against shock when a hose bursts. When testing a full length of hose, or even a short length of large diameter, a pet cock should be provided to release the air as the hose is being filled.

Requirements of specifications as regards the pressure test vary according to the kinds of hose, but, as a rule, the test is made, not with a view to developing the ultimate strength of the hose, but rather to detect defects in workmanship, which are usually noticeable at a pressure well below that necessary to rupture the hose.

In the case of fire hose it is usual to specify a certain pressure when the hose is lying straight or when bent to the arc of a circle of given radius; and the hose must stand a specified pressure when doubled upon itself. It must not show excessive expansion, elongation, or twist under pressure, and the twist must be in a direction tending to tighten the couplings.

## (c) STEAMING TEST

Figure. 20 illustrates a method of testing steam hose. The header 1 is provided with six outlets, each of which is controlled by a one-half-inch globe valve. The header 2, which is connected to a steam trap 3, is similarly provided with inlets and controlling valves. The hose to be tested is cut into lengths that will just fit between the connections on the headers, the bottom connections being made with unions. Steam passes through a regulating valve (not shown) into the header 1, and thence through the hose to the header 2, from which the condensation is carried to the steam trap.

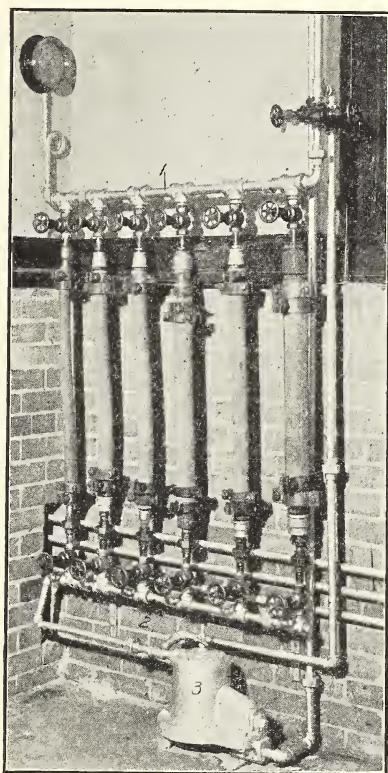


Fig. 20.—Six samples of steam hose connected up for steaming test

#### IV. THE CHEMISTRY OF RUBBER

Although rubber has been extensively used for a number of years, it is only recently that we have known very much about its chemical nature. The synthesis of rubber shows that it belongs with the terpenes, having the formula of  $(C_{10}H_{16})_n$ , but so far all attempts to show the actual size of this molecule have been unsuccessful. The synthesis is accomplished by the polymerization of the simple terpene, isoprene, which has the formula

$C_5H_8$ . Additional proof of the correctness of the above formula is obtained by means of the various addition products which have been formed, such as the tetrabromide, nitrosite, ozonide, etc. These latter show that in the rubber molecule, each group of  $C_{10}H_{16}$  is capable of combining with two atoms of sulphur. It is this adding of sulphur during the process of vulcanization which transforms the crude, sticky gum into a tough, elastic material.

The crude rubbers, however, contain other substances than the pure rubber just mentioned. In addition to scraps of bark, twigs, dirt, etc., that accompany some of the crude rubbers, they contain varying proportions of proteids, resins, hydrocarbons, etc. In some cases the rubber is only a very small fraction of the entire mass, and in such cases, it requires extensive treatment to obtain the pure gum.

The mechanical impurities and water-soluble constituents are removed by the washing process described elsewhere in this circular. The resins remain behind and form one impurity which must be determined by chemical analysis. The amount and character of these resins are of great assistance in determining the nature of the rubber used in compounding. In some cases the percentage of resins is exceptionally high and then the crude rubbers must be subjected to a deresinizing process before they can be used.

The acetone extraction for the purpose of determining the quantity of such resins is made by taking a weighed sample of the finely ground material and extracting it with acetone for a period of from 8 to 15 hours. The acetone is removed by distillation, the residue weighed, and the latter, consisting of the rubber resins, subjected to a very careful examination.

If the extraction is made on a vulcanized compound, the acetone also extracts the free sulphur and any mineral oils or waxes that may have been used. The free sulphur can be readily determined by any of the methods given in the text books, and the amount so determined must be deducted from the total extract. This gives a corrected figure called "organic extract" or, sometimes, simply "corrected acetone extract." For the best grades of Para rubber, this figure should not exceed 5 per cent of the rubber present. A higher percentage of resins would indicate the presence of other rubbers than Para, while the presence of mineral oil indicates the possibility of reclaimed rubber having been used, inasmuch as practically all the reclaimed rubbers are compounded with more or less mineral oil to make them work easier.

The acetone extraction is one of the most promising tests for the examination of rubber goods. A vast amount of work remains to be done before the analyst will always be in a position to say exactly what has been used in compounding. Even to-day, however, a great deal of information can be obtained from it.

The process of vulcanization consists simply in the chemical combination of sulphur and rubber. Varying amounts of sulphur, depending upon the nature of the crude gum as well as upon the properties desired in the finished product, are added to the compound, and after heating, varying



amounts of the sulphur will be found to have combined chemically with the rubber, giving thus a new chemical compound with new and desirable properties that are not possessed by the crude material.

It is often desirable to limit the amount of sulphur in a compound, and this calls for a method of determining the total amount of sulphur present. This part of the analysis of rubber goods has received considerable attention, and several methods are available which may be depended upon to give accurate results, even in the hands of comparatively inexperienced analysts.

In addition to the sulphur combined with the rubber, and the free sulphur already mentioned, sulphur may be present in the mineral fillers. There can be no objection to such sulphur, providing the mineral containing it has no injurious effect on the rubber, and further that the amount of such sulphur can be readily determined. Barytes is one such compound, and it is permitted in practically all compounds where the amount of sulphur is specified. Sublimed lead (largely a basic sulphate of lead, of varying composition) does not yet fulfill the conditions just mentioned, but it is quite probable that we shall soon be able to determine it accurately, and it will then be merely a question of deciding whether it is a desirable filler in high-grade compounds.

The determination of the amount of rubber present in a vulcanized compound is an important, though difficult matter. For a long time this was determined by igniting a weighed sample, and determining the mineral fillers, or ash. The rubber was calculated by the difference between 100 per cent and the sum of the ash, total sulphur, and corrected acetone extract. The results obtained by this method are not very accurate, it is true, but with care will give a close approximation to the amount present.

The problem is to-day being attacked from several points. Some chemists are endeavoring to find a suitable solvent which will remove the rubber, and permit the weighing of the mineral residue. Turpentine, terebene, anisole, phenetol, and other substances have been suggested, and some of these are giving fairly good results. Others are working along the line of the direct determination of the rubber present by means of the various addition products, such as tetrabromide, nitrosite, etc., but so far only a fair amount of success has been attained.

The determinations which have been briefly mentioned above are those most commonly employed. For the detection of adulterations or substitutes, other tests have been devised. After the sample has been extracted with acetone, extraction with chloroform will show whether any tar or bituminous matter has been used. Following the extraction with chloroform, the sample is extracted with alcoholic potash, which reveals the presence of sulphonated oils, or vegetable oils not soluble in acetone or chloroform. Para rubber contains only a very small percentage of material extracted by these two solvents.

The methods of analysis in use at this Bureau are now in course of thorough testing and revision. It is hoped that within a comparatively short time the results of this work will be published as an addition to this circular.



It is not within the scope of this circular to go very much into the details of the subject. For those who are interested and wish further information, the following textbooks are suggested: India Rubber, by Philip Schidrowitz; Chemistry of India Rubber, by Carl Otto Weber; Crude Rubber, by H. C. Pierson; Der Kautschuk and seine Prüfung, by Hinrichsen and Memmler.

In addition to the foregoing, the following scientific journals will be found to contain many articles on this subject: Journal of Industrial and Engineering Chemistry, India Rubber Journal, India Rubber World, Journal of the Society of Chemical Industry, Gummi-Zeitung, Kolloid-Zeitschrift.

## V. REGULATIONS REGARDING TESTS

The Bureau has been for some time testing rubber for the various Government departments and is prepared to perform such service for the general public in special cases. The Bureau will also be glad to cooperate with investigators, manufacturers, and others not only in executing tests, but also, on request, in furnishing any information at its disposal concerning methods of testing rubber.

**Fees.**—The Bureau is not yet in position to adopt a complete schedule of fees, but a reasonable charge will be made in each case, depending upon the nature of the test. A few of the more usual tests will entail a fee, as shown in the following schedule:

### Schedule No. 115

(a) Rubber water hose, suction hose, or fire hose.....	\$3.00 to \$5.00
(b) Rubber air hose.....	4.00 to 6.00
(c) Rubber air-brake hose.....	4.00
(d) Rubber steam hose.....	5.00 to 10.00
(e) Rubber dredging sleeves.....	5.00
(f) Rubber sheet packing.....	2.00 to 4.00
(g) Rubber and asbestos sheet packing.....	2.00 to 4.00

When a pressure test is desired, samples of hose should be about 5 feet long, otherwise a 2-foot length will be sufficient. Samples of sheet packing should be not less than 6 inches square.

**Application for test.**—The request for test should state explicitly the specifications according to which test is to be made.

**Remittances.**—As soon as the fee is assigned to a test, a bill is sent at once, and payment should be in advance, made by money order or check drawn to the order of the "Bureau of Standards." Results of tests are not certified until fees are paid.

**Shipping Directions.**—Materials should be shipped prepaid as all transportation charges are payable by the party desiring the test. Articles should be addressed simply "Bureau of Standards, Washington, D. C."

S. W. STRATTON,  
*Director.*

Approved:  
BENJ. S. CABLE,  
*Acting Secretary.*