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DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS
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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR RUBBER GOODS (METHODS OF PHYSICAL TESTS AND CHEMICAL ANALYSES)

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 59a

[Revised May 20, 1925]

This specification was officially promulgated by the Federal Specifications Board on June 15, 1923, for the use of the Departments and Independent Establishments of the Government in the purchase of rubber goods.

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Part 1.—METHODS OF PHYSICAL TESTS

These specifications cover rubber goods used by the various Government departments. The following are details of such specifications and tests as are common to the articles. For specific information applying directly to a particular article, see detail specification, which shall take precedence whenever there is any conflict.

These specifications contain the approved methods of physical and chemical tests.

I. CONSTRUCTION

1. GENERAL REQUIREMENTS

All goods shall be free from defects in material and workmanship, and be of the dimensions as given in the detail specification or proposal submitted to manufacturer.

2. HOSE

Hose shall consist of a rubber tube, cotton reinforcements, and a rubber cover, and shall be of wrapped construction unless otherwise specified in detail specification.

The tube and cover shall be smooth, free from pitting, and of uniform thickness. All canvas layers shall be applied on the bias, with edges lapped at least one-half inch (not sewed), and be well impregnated with a rubber compound. The plies of braided hose shall be evenly braided and embedded in a rubber compound.

Each length of hose shall be fitted with couplings and clamps without the use of tape or rubber tissue. The female coupling shall be fitted with a rubber washer, cut from a rubber tube of the same composition as the hose tube.

3. BELTING

Rubber and balata belting shall be made of impregnated cotton duck.

4. PACKING

Sheet packing shall have smooth surfaces, free from pitting, and be of uniform thickness.

II. MATERIALS

1. COTTON

The cotton fabric layers in hose, belting, etc., shall be well, evenly, and firmly woven, and as free from unsightly defects, dirt, knots, lumps, and irregularities of twist as is consistent with the best manufacturing practice.

2. RUBBER

The rubber compounds shall in all cases be properly vulcanized.

III. BRANDING

1. GENERAL REQUIREMENTS

Branding and stenciling shall contain the manufacturer's name, trade-mark or brand.

2. HOSE

Hose 25 feet or over in length shall have brands of a distinctive color inlaid in the rubber cover at two places on each length, approximately 4 feet from the ends, letters to be at least one-fourth inch high. Hose less than 25 feet in length shall have one inlaid brand approximately in the center.

3. BELTING

On the seam side of rubber belting brands shall be inlaid in red rubber at least every 30 feet. Balata belting shall be branded with stencil only.

4. PACKING

Packing furnished in rolls shall be stenciled at least every 15 feet, and when furnished in sheets at least once on each sheet.

IV. SAMPLING AND INSPECTION

1. GENERAL REQUIREMENTS

The manufacturer shall notify the purchaser sufficiently in advance of the completion of material to permit of arrangements for inspection.

Inspection and tests shall be made at place of manufacture unless otherwise specified. The manufacturer shall provide a place for conducting tests, the necessary help, equipment, etc.

In case it is not practicable to obtain suitable test specimens, the manufacturer shall furnish pieces ⅛ by 1½ by 8 inches, which he guarantees to be of the same material and equivalent cure as that used in article furnished.

Whenever the weight of the fabric is specified, the manufacturer shall submit 1 square foot of unfrictioned fabric with each order.

The inspector shall, after tests, mark the remainder of samples with manufacturer's name, order, requisition, and item numbers and forward them to the proper organization for any further tests. Any lot represented by a sample which fails in one or more tests may be retested at the expense of the contractor. For this purpose two additional samples shall be selected. Failure of either in any respect shall be cause for rejection.

2. HOSE

One length shall be taken from each 2,000 feet or less. A 3-foot section shall be cut from each length so selected for hydrostatic test, after which further tests shall be made on the same sample. Manufacturer shall refit couplings to said lengths, which shall be accepted as full lengths provided they otherwise conform to specifications. When hose is furnished in lengths under 10 feet, extra lengths shall be furnished at contractor's expense for test purposes.

3. BELTING

One sample, the full width of the belt, shall be taken from each lot of 500 feet or less. The sample shall be in accordance with the table below.

4. SHEET PACKING

One sample shall be taken from each lot of 250 pounds as per table.

5. ROD PACKING

One sample shall be taken from each 1,000 pounds or less as per table.

6. GASKETS

One sample gasket shall be taken from each 5,000 or less handhole gaskets, 1,000 or less mud drum gaskets, and 500 or less manhole gaskets.

7. MOLDED AND LATHE-CUT GOODS

One piece shall be taken from each lot of 200 or less.

8. AMOUNTS OF MATERIAL REQUIRED PER SAMPLE FOR TEST

Hose of 3-inch diameter and less	3 feet.
Hose over 3-inch diameter	1 foot.
Hose for pressure or steam test	3 feet.
Sheet packing	1 square foot.
Rod packing:	
½ inch and under	2 feet.
Over ½ inch	1 foot.
Insulated wire	5 feet.
Belting:	
4 inches and under in width	1 foot.
Over 4 inches in width	4 inches.
Bicycle and motorcycle tires	Whole tire.

Automobile tires:	
Solid	Section 18 inches long.
Pneumatic	Whole tire.
Inner tubes	Whole tube.
Miscellaneous rubber goods	Sufficient material to provide at least 15 pieces 1 by 6 inches.

V. PHYSICAL TESTS

1. GENERAL REQUIREMENTS

All tests of rubber shall be made at a room temperature between 65 and 90° F. The samples should remain at this temperature for at least one hour before being tested.

2. TENSILE STRENGTH, ELONGATION, AND SET

(*a*) Definitions.—Tensile strength is expressed in pounds per square inch and is determined by dividing the breaking load in pounds by the cross section of the test piece in square inches. Ultimate elongation is expressed in per cent. For example, if a test piece of cross section 0.25 by 0.1 inch failed at a tension of 50 pounds, when the distance between the gauge marks was 14 inches, the tensile strength would be $50 \div (0.25 \times 0.1) = 2{,}000$ lbs./in.^{2} and the ultimate elongation would be $14-2=12$ inches, or 600 per cent.

The term "set" refers to the extension remaining, after a specified elongation for a given period of time, followed by a specified interval of rest. For example, a test piece is stretched from 2 to 10 inches (400 per cent) for 10 minutes and then released. Its length measured after 10 minutes' rest is 2.4 inches, so that the set under these conditions is 0.4 inch, or 20 per cent.

(*b*) Separating Rubber from Fabric.—The gasoline used in separating rubber from fabric shall be 72 to 76° Baumé and upon evaporation shall not leave an appreciable amount of oily residue. To avoid stretching the rubber unnecessarily it is desirable to cut the material into strips slightly wider than a test piece. The separation should be made gradually and a little at a time, while the rubber is gripped near the point of separation. The rubber should then be so placed as to permit free evaporation from all parts of its surface.

(*c*) Buffing.—Any unevenness of surface, such as impressions caused by contact with fabric parts, which would interfere with an accurate measurement of thickness, is removed by careful grinding with an abrasive wheel of about No. 30 grit. A wheel for this purpose should be provided with a slow feed in order that very little rubber be removed at one cut, otherwise the rubber may be injured by overheating. The speed should be from 2,500 to 3,000 r. p. m.

for a wheel of 5 inches diameter. If a backing is used, as in the case of cotton rubber-lined fire hose, it should be entirely removed in the same way. The face of the wheel should be kept sharp.

In buffing the cover of steam hose after the steam test, care should be taken to remove the least possible amount of material.

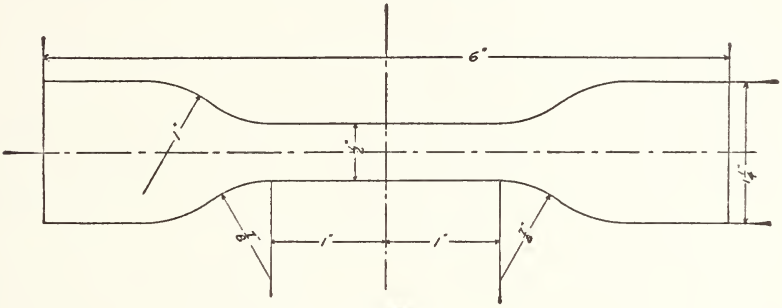

Fig A

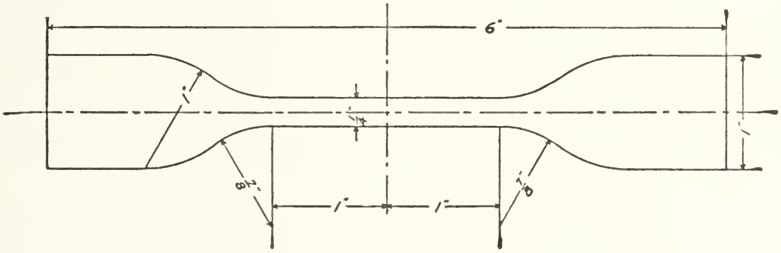

Fig. B

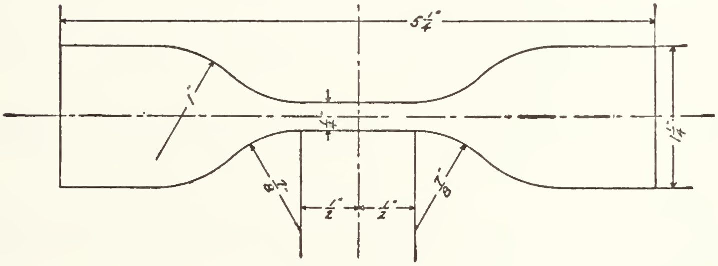

Fig. C

Fig. 1.—*One-fourth inch and one-half inch test pieces with enlarged ends*

(*d*) Cutting Samples.—(1) *Shape and size.*—The central portion of test pieces shall be one-fourth inch wide over a gauge length of 2 inches, the ends being gradually enlarged to a width of 1 inch to provide a satisfactory gripping surface (see fig. 1, B). If one-half-inch test pieces are specified, the end portions should have a width of 1¼ inches (see fig. 1, A).

In the case of pure gum or compounds containing a large percentage of rubber which have a very great elongation, shorter test pieces one-fourth inch wide over a gauge length of 1 inch and 1¼ inches wide at the ends are used (see fig. 1, C).

Gauge marks shall be made with ink by using a stamp consisting of parallel steel blades which produce very fine lines, care being taken in all cases not to injure the rubber.

In the case of insulated wire, rubber bands and jar rings, test pieces shall be used as described under Section VI.

(2) *Equipment.*—Test pieces shall be cut with a metal die (fig. 1) which should be kept sharp to avoid leaving ragged edges on the rubber. The distance between cutting edges of the blade over that portion of the die corresponding to the gauge length of the test pieces shall not vary more than 0.002 inch.

An arbor press is recommended for forcing the die through the rubber in preference to the practice of striking the die with a mallet. The operation is facilitated by wetting the cutting edges of the die. The rubber should rest on a smooth and slightly yielding surface which will not injure the blade. A piece of rubber belting, or preferably leather belting, is suitable for the purpose. When the material on which the test pieces are cut has become slightly rough from use, a sheet of paper placed under the rubber will be of advantage.

(3) *Measuring.*—The width of the test piece shall be measured with a gauge or caliper graduated to 0.001 inch, the instrument being used in such a way that the compression of the rubber between the contact surfaces is negligible. The thickness of the test piece shall be measured with a gauge graduated to 0.001 inch, under a pressure of approximately 3 ounces, exerted by a contact foot of approximately 0.3-inch diameter.

The cross section shall be considered as the product of the minimum width by the minimum thickness of the test piece, between the gauge marks.

(*e*) METHOD OF MAKING TESTS AND COMPUTING RESULTS.—Eight test pieces shall be used, four for the tensile strength and elongation tests, and four for the set test.

(1) *Tensile strength and elongation.*—The machine used for these tests shall fulfill the following requirements: (1) The dial or scale for indicating the applied tension shall be accurate within 0.1 pound for samples having a breaking strength less than 15 pounds. For all loads above 15 pounds the maximum error shall not exceed 0.5 pound. The indicator shall remain at the point of maximum load after rupture of the test pieces. (2) The machine shall be power driven, and the rate of separation of the grips shall be 20 inches per minute. (3) The grips for holding straight test pieces (fig. 1) shall

be of such a design that a uniform pressure will be exerted across the gripping surface to avoid uneven slipping, and to insure failure of the test piece within its constricted portion. For circular test pieces, such as rubber bands, etc., each grip consists of two rollers mounted on ball bearings. The action of these rollers is such that as tension is applied, they rotate in opposite directions, thus tending to equalize the stress around the circumference of the test piece.

Care should be exercised to adjust straight test pieces symmetrically in the grips in order that the tension shall be distributed uniformly over the cross section. If the tension is greater on one side of the test piece than on the other, the gauge marks will not remain parallel and the maximum strength of the rubber will not be developed. The ultimate elongation may be measured by holding a scale (graduated to 0.1 inch) in a slightly inclined position at the back of the test piece with its lower end pressed lightly against the rubber just back of the lower gauge mark. As the rubber is stretched the eye follows the scale just back of the upper gauge mark. With a little practice the elongation at break may be measured with an error not exceeding 0.05 inch.

(2) *Set.*—The test piece is stretched at an approximately uniform rate of speed such as to require about 15 seconds to reach the specified elongation and is held in this position for 10 minutes, including the time required for stretching, after which it is immediately released (without being allowed to snap back) and laid out on the test table. After a rest of 10 minutes the distance between the gauge marks is measured to the nearest 0.01 inch and the set recorded in percentage of the original gauge length. In stretching a test piece it is convenient to use a measuring rod of a length equal to the exact distance required between the gauge marks. By holding the rod beside the test piece while it is being stretched the operation is simplified and the chance of stretching the test piece more or less than the desired amount is very much reduced. In taking the time of the various operations in this test, a stop watch or a watch having a second hand should be used.

(3) *Interpretation of results.*—The average of the results of four test pieces is used for a determination of tensile strength, ultimate elongation, and set. If in any case the result of a single test piece is found to be very much lower than that of the others, indicating a flaw in the material, the low result is discarded and the average of the results of the other three tests is recorded. If the result thus obtained fails by less than 5 per cent to meet the requirements of the specification, a check test is made with four additional test pieces, the results of which, computed as above, shall be considered final.

3. FRICTION

(*a*) Definition.—The term "friction" is used to indicate the adhesion between the two parts considered and is expressed numerically by (1) the average tension, in pounds, required to cause separation at a definite rate (1 inch per minute) under stated conditions, or (2) the average rate of separation in inches per minute caused by a specified tension exerted between the parts under the stated conditions. In either case, the test piece is 1 inch wide.

(*b*) Equipment.—Friction should preferably be determined with a machine which automatically records on a chart the value of the friction at all points of the test piece. The machine should be adapted to maintain a uniform rate of separation of the parts as specified.

If so specified, or if the nature, form, or construction of the material is such as to preclude the use of a testing machine, the dead-weight method is used. This consists in suspending the specified weight from the detached end of the part to be separated. The length stripped is measured after the weight has been removed, between marks made on the other part at the beginning and end of the test. The duration of the test is timed with a stop watch or a watch having a second hand.

(*c*) Method of Making Tests and Computing Results.—The value of the friction is recorded as the average tension required to cause separation at the rate specified, this being determined over that portion of the chart corresponding to an actual separation of the parts being tested. If during a test one of the parts begins to tear, instead of separating from the other part, the material being torn is cut with a knife up to the surface of contact between the two parts and the test started again. If one of the parts repeatedly tears, instead of separating from the other part, the average load at which tearing takes place is taken as the value of friction.

If the dead-weight method is used, the value of friction is recorded as the average rate of separation under the action of a specified weight.

The value of friction is based on the results of a single test. If the average value as determined with an autographic machine fails by less than one-half pound to meet the specification, or if the result of dead-weight test is greater than 1 inch per minute, but less than 1.1 inches per minute, a check test is made, the result of which shall be considered final.

4. HARDNESS

(*a*) Definition.—The term "hardness" is used to express the depth of indentation produced by a spherical-ended plunger of definite size, under a stated normal pressure exerted for one minute.

(*b*) EQUIPMENT.—The instrument used shall consist of a plunger with spherical end of 3.2 or 6.4 mm diameter on which a pressure of 1 kg is exerted by means of a dead weight, and a gauge graduated to one one-hundredth millimeter for measuring the depth of indentation. The smaller plunger is used except in the case of very soft rubbers.

(*c*) METHOD OF MAKING TESTS AND COMPUTING RESULTS.—The sample to be tested is supported in a horizontal position and the instrument is adjusted so that the plunger is vertical. In testing sheet rubber the sample should rest on a smooth unyielding surface. The needle of the gauge is set at zero with the plunger resting on the sample, after which the 1 kg weight is lowered upon the plunger and allowed to remain for one minute. The reading on the gauge dial then shows the depth of indentation in millimeters.

The average of four readings is recorded as the hardness of the sample.

5. STEAM TEST

(*a*) METHOD FOR HOSE.—A header is provided with outlets, each of which is controlled by a valve. Another header which is connected to a steam trap 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 into the first header and thence through the hose to the other header, from which the condensation is carried to a steam strap.

The hose is subjected in this way to the action of steam at a pressure of 75 lbs./in.^{2} for two days of seven hours each, the steam being turned off and the hose allowed to cool during the intervals. The hose then remains under laboratory atmospheric conditions for from 12 to 48 hours before being tested.

(*b*) METHOD FOR MISCELLANEOUS GOODS.—Material such as sheet packing is bolted between iron plates and subjected to the action of steam in a suitable autoclave. The test samples, several of which may be steamed at once by placing thin sheets of iron between them, are supported on a wire stand which holds them well above the water level.

Rubber pump valves are steamed in the same manner except that they are not clamped between plates.

6. ACCELERATED AGING TEST

Test pieces are prepared, measured, and marked as described in Section V, 2. The samples are then suspended in a constant temperature oven and subjected to the action of dry air at a temperature of 158° ± 2° F. for the specified length of time. A constant temperature shall be maintained throughout the test zone by means of a free circulation of air within the oven. Ventilation is secured through holes in the oven walls, but it is not considered necessary to provide for a positive flow of fresh air into the oven.

The samples when removed from the oven shall remain at room temperature for 24 hours before being tested. The results of tests are based on measurements made before heating.

7. HYDROSTATIC PRESSURE TEST OF HOSE

A 3-foot length of hose is laid out straight on a smooth surface. One coupling is connected to the source of water supply, the other coupling being closed with a cap or plug provided with a pet cock for the escape of air while the hose is being filled with water. To insure the complete removal of air from the hose, the surface on which the hose rests should be slightly inclined toward the source of water supply.

With the pet cock open, admit water into the hose gradually until the air has been expelled and the hose is completely filled with water. The pet cock is then closed and water is gradually admitted into the hose in such quantity as will increase the pressure per square inch at an approximate rate of 300 pounds per minute, and while the pressure is being increased the hose is carefully examined for leakage or other defects. When the test gauge indicates the pressure specified for the kind of hose being tested, the water supply is shut off and the hose allowed to remain under this pressure for 10 minutes.

VI. SPECIFIC DETAILS OF TESTING

1. PACKING, BELTING, ETC.

(*a*) Tensile Strength, Elongation, and Set Tests.—Test pieces from material of this sort may be cut in any direction. Strips are first cut of a length and width slightly greater than the corresponding maximum dimensions of the test pieces. If the material contains fabric, the rubber is carefully separated as described in Section V, 2, and after the gasoline has evaporated the surface is buffed over the central portion of the strip for a distance somewhat greater than the gauge length of the test piece. The test piece is then cut with a die as described in Section V, 2.

(*b*) Friction Tests.—(1) *Belting.*—A 1-inch section about 8 inches long is cut from the belt in the longitudinal direction. This section is subdivided into strips of two plies each, one end of which is separated for a distance of 2 inches. One of the loose ends of a test strip is clamped in the upper jaw and the other end in the lower jaw of the testing machine. In this case the rate of separation of the jaws is twice the rate of stripping.

(2) *Packing.*—Test strips are prepared from sheet packing, and from square packing in the same way as from belting, except that if square packing is of a size that does not permit of a 1-inch strip a narrower test piece shall be used. Test pieces are prepared from round packing in the same manner as from plied hose.

In case of packing constructed with light-weight sheeting so that one ply is not sufficiently firm for test, four plies shall be taken and the test made by stripping two from two.

2. HOSE (GENERAL)

(*a*) TENSILE STRENGTH, ELONGATION, AND SET TESTS.—In the case of plied hose, sections of sufficient length for test pieces are cut. The sections are cut into longitudinal strips slightly wider than the test pieces, after which the cover and the tube are separated from the fabric as described in Section V. When the gasoline has evaporated, the fabric impressions are removed by buffing over a portion of the strip slightly greater than the gauge length. The test pieces are then cut with a die as described in Section V. When the diameter of the hose is one-half inch or less, test pieces should be first cut with a die and then buffed.

(*b*) FRICTION TESTS.—(1) *Wrapped hose.*—The most satisfactory way of preparing test pieces is as follows: A short length of hose is pressed snugly over a smooth, slightly tapered mandrel. The mandrel is put in a lathe, and 1-inch sections or rings are cut with a short pointed knife, which is forced gradually through the cover and plies of fabric, but not entirely through the rubber tube. Upon removal from the mandrel the rings are easily cut apart with a knife. The pointed knife used in cutting the rings should be kept sharp and should be wetted before each cut. A cut is made through the rubber cover at the point where the outside ply of fabric ends. The fabric is then unwound until the outside ply carrying the rubber cover is entirely separated from the rest of the sample.

The test piece is pressed snugly over a short wooden mandrel which is free to revolve in roller bearings attached to the upper jaw of the testing machine. The detached end of the fabric is held in the lower jaw. The downward movement of the lower jaw produces a radial pull which separates the fabric, and at the same time makes a graphical record which shows the length of fabric separated and the tension required at each point. In this case the rate of separation of the jaws is the same as the rate of stripping.

(2) *Braided hose.*—One-inch rings are cut as for wrapped hose. A cut is made through the rubber cover and first ply of braid. The braid is stripped just enough to admit of a clamp being attached.

(3) *Hose reinforced with metal.*—When metal reinforcement is present as in suction hose, which is reinforced by means of metal embedded in the rubber tube, between the plies of duck, or embedded in a layer of rubber in the central portion of the hose, test pieces are prepared as follows: A transverse section 2½ inches long is cut from the hose and opened so as to form a flat strip. Two parallel lines 1 inch apart are drawn on the rubber tube, the reinforcement being symmetrically located between them. The sample is then cut through on these lines making a strip 1 inch wide and equal in length to the circumference of the hose. The method of test is the same as that for belting.

3. DREDGING SLEEVES

Samples are prepared and tested in the same manner as belting.

4. FIRE HOSE (COTTON RUBBER LINED)

(*a*) Tensile Strength, Elongation, and Set Tests.—With cotton rubber-lined hose, a section of sufficient length to produce the desired number of test pieces is cut. This section is cut at the lap and subdivided into transverse strips from which the cotton jacket is removed by the use of gasoline. When the gasoline has evaporated, the rubber backing is buffed off and the test pieces are cut as described above, except that the central portion of the test piece shall be one-half inch wide over a gauge length of 2 inches (see fig. 1, A). The fold shall be within the gauge length.

To determine the strength of the lap, another section of sufficient length to produce the desired number of test pieces is cut. This section is cut so that the lap will be in the center of the constricted portion of the test piece. The cotton jacket is removed by the use of gasoline. When the gasoline has evaporated test pieces are cut as described above, without being buffed.

(*b*) Friction Tests.—With a soft pencil draw two parallel lines 2½ inches apart, following the filler strands around the circumference of the hose. Cut the hose at the lap and also along these lines so as to form a strip of a length equal to the circumference of the hose. This strip is laid out flat, and the rubber lining cut through to the jacket along parallel lines 1½ inches apart or as may be specified. The end of the central portion of the lining between these two cuts is separated from the jacket for a short distance, and is clamped in a stationary grip. From the detached end of the rubber is suspended a weight of 12 pounds, and the rate of separation is determined from the length stripped during a measured interval of time. Measurements are made to the nearest 0.05 inch, between marks made on the fabric at the beginning and end of the test. The time shall be measured with a stop watch or with a watch having a second hand,

and the duration of test shall be five minutes or such part thereof (measured to the nearest second) as may be required to strip a length of 5 inches.

5. SOLID RUBBER TIRES

(*a*) TENSILE STRENGTH, ELONGATION, AND SET TESTS.—First cut from the tire a section of rubber 6 inches long, measured in the direction of its circumference. From the center of this section cut a longitudinal strip 1½ inches wide normal to the axis of the tire. This strip is subdivided in a slicing machine or by other suitable means into strips 1½ inches wide and one-eighth inch thick. These strips are buffed, if necessary, before being cut into test pieces.

6. PNEUMATIC TIRES

(*a*) TENSILE STRENGTH, ELONGATION, AND SET TESTS.—Strips 6 inches long and 1¼ inches wide are cut longitudinally from the center of the tread and from the side wall, and the rubber is separated from the fabric with the aid of gasoline. The nonskid portion of the tread rubber is sliced off with a knife, after which the central portion is buffed on both sides over a length of 2½ inches until free from all friction compound, fabric impressions, or any irregularities of surface. The side wall is buffed on one or both sides, as may be necessary.

The test pieces shall be of the shape and size illustrated in Figure 1, B.

(*b*) FRICTION TESTS.—(1) *Preparation of sample.*—Friction is determined with a 1-inch section of the tire. The test piece may be conveniently prepared as follows: Cut a section approximately 2 inches long and remove the beads. Wrap the section tightly around a mandrel, using friction tape. The mandrel is put in a lathe and a 1-inch section cut with a pointed knife.

(2) *Between tread and breaker.*—Remove the tread at one end up to the breaker strip and separate the two for a distance of about one-fourth inch. The detached end of the tread is clamped in the lower jaw and the corresponding end of the tire section in the upper jaw.

(3) *Between side wall and carcass.*—When the friction test between tread and breaker strip has been made, remove the test piece from the machine and separate the tread by hand until the point has been reached where the side wall joins the carcass. Cut off a portion of the tread, leaving only sufficient material to be clamped in the lower jaw; clamp the corresponding end of the tire section in the upper jaw.

(4) *Between breaker and cushion.*—Remove the tread (with breaker strip attached) for a sufficient distance to admit of its being clamped in the lower jaw; clamp the corresponding end of the tire section in the upper jaw.

(5) *Between cushion and carcass.*—Remove the cushion (with breaker strip attached) for a sufficient distance to admit of its being clamped in the lower jaw; clamp the corresponding end of the tire section in the upper jaw.

(6) *Between plies.*—The tread, breaker strip, and cushion having been removed, the outside ply of fabric is separated for a sufficient distance to admit of its being clamped in the lower jaw. The corresponding end of the tire section is clamped in the upper jaw. The other plies of fabric are separated in succession and tests are made in the same way. Cord tires require special treatment because it is necessary to clamp at least three plies of fabric in each jaw.

7. INNER TUBES

(*a*) TENSILE STRENGTH, ELONGATION, AND SET TESTS.—Strips 5 inches long and 1½ inches wide are cut longitudinally from the tube. These strips shall not be buffed unless the impressions left by the wrapping fabric are so pronounced as to vitiate the results of tests. The die used for cutting test pieces from this grade of rubber should be kept very sharp and each strip of rubber should be thoroughly wet before the die is used. A piece of paper should be inserted under the strip before the test piece is cut. The test pieces shall be of the shape and size illustrated in Figure 1, C.

8. RUBBER BANDS

(*a*) UNDER ONE-FOURTH INCH IN WIDTH.—Double-spool grips are used, and the tests are made on a machine similar to that described in Section V, 2. The tensile strength is measured in pounds, and the elongation is expressed in percentage of the original length.

Ultimate elongation

$$\text{per cent} = \frac{(L - L_1)100}{L_1}$$

where

L = one-half the distance around spools at time of failure;
L_1 = initial inside length of band, measured flat (one-half total length).

(*b*) ONE-FOURTH INCH IN WIDTH OR OVER.—Double-spool grips are used and tests are made on a machine as described in Section V, 2. In bands of this size the tensile strength is calculated in pounds per square inch, as follows:

$$\text{Tensile strength in pounds per square inch} = \frac{B}{2wt}$$

where

B = breaking load in pounds,
w = width of band in inches,
t = thickness of band in inches.

Elongation is measured and expressed in the same way as described above for bands under one-fourth inch in width.

9. JAR RINGS

Jar rings are tested in the same way as rubber bands. Owing to the shape of the material, the inside length L_1 becomes one-half of the internal circumference, thus:

$$L_1=\frac{\pi D}{2}$$

where

D = internal diameter in inches.

10. INSULATED WIRE

When the diameter of wire is large enough and the insulation thick enough, test pieces are prepared as follows: The insulation is cut through to the wire in the longitudinal direction and the rubber is removed in one piece. The rubber is then laid out flat and cut with a die. Test pieces are buffed on both sides until they are smooth all the way across the constricted portion.

In certain cases test pieces may be cut to better advantage if the rubber is held down with pins. The cross section of test piece is then approximately a sector of a ring.

When the diameter of the wire is too small to permit of specimens being cut with a die, the insulation is tested as a whole. The general method for removing the insulation as a whole from a single wire is as follows: The insulation is removed for a distance of one-half inch at each end of a 7-inch length of wire and a slight nick is made in the wire at one end of the insulation. The uncovered ends are gripped in the jaws of the testing machine and the wire pulled until it breaks at the nick. The stretching of the wire reduces its cross section, and thus facilitates the removal of the insulation. The end of the wire should be rounded before pulling it through the insulation, as the rough end might cut the rubber. In some instances adhesion between the wire and rubber is so great that the insulation can not be easily removed after the above treatment. When this is the case the sample is immersed in mercury, which forms an amalgam with the tin coating on the wire. Within one-half hour the amalgamation has usually progressed sufficiently to permit the insulation to be easily removed. When there is a longitudinal bead or fin on the surface of the insulation it should be removed with a small sharp wood plane before the wire is withdrawn.

If the cross section of the test piece is not of rectangular shape, its area is computed from measurements as follows (fig. 2):

(1) When the test piece is cut with a die and buffed, the width is taken as the average of the top and bottom widths of the buffed portion between the gauge marks.

(2) When the test piece is cut with a die and not buffed—

$$A=\frac{arc}{4\,D}(D+d)\ (D-d)$$

where

A = area of cross section in square inches,
D = outside diameter of insulation in inches,
d = diameter of wire or wire core in inches,
arc = width of test piece corresponding to outside diameter D.

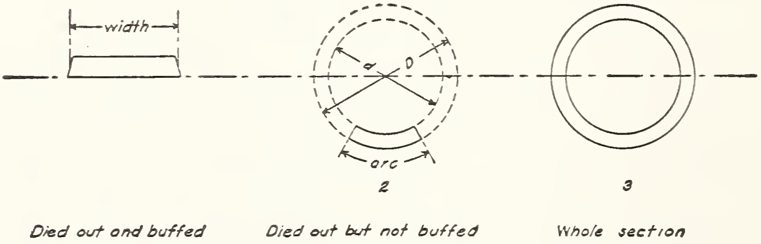

Fig. 2.—*Cross section of test pieces*

(3) When the insulation is tested as a whole, in the form of a hollow cylinder (VI, 10)

$$A=0.7854\ (D+d)\ (D-d)$$

where

A = area of cross section in square inches,
D = outside diameter of insulation in inches,
d = diameter of wire or wire core in inches.

Part 2.—METHODS OF CHEMICAL ANALYSIS

VII. INTRODUCTION

These methods are essentially the methods adopted as standard by the American Chemical Society and the American Society for Testing Materials. In addition, there is included a method which employs a solvent for the vulcanized rubber. This method has proved to be more accurate than an ash determination when carbonates are present in a rubber compound. It is also used in the analysis of asbestos rubber packings and rubberized fabrics.

VIII. DEFINITION OF TERMS

1. ACETONE EXTRACT

If the acetone extraction is made on a vulcanized compound, the acetone removes the rubber resins, the free sulphur, any mineral oils or waxes, and part of any bituminous substances or vulcanized oils that may have been used. This is generally called acetone extract uncorrected. The percentage of free sulphur is determined and deducted from the total extract. The value obtained is known as acetone extract corrected. The corrected figure thus obtained will at times give valuable information regarding the quality of the rubber present. For the best grades of Hevea rubber this should not exceed 5 per cent of the rubber present. A higher extract may indicate the presence of inferior or reclaimed rubbers.

2. CHLOROFORM EXTRACT

The chloroform extraction removes a portion of the bituminous substances and serves as an indication of their presence.

3. ALCOHOLIC-POTASH EXTRACT

The purpose of the alcoholic-potash extraction is to detect the presence of rubber substitutes.

4. FREE SULPHUR

The free sulphur is defined as that which is removed during the acetone extraction.

5. TOTAL SULPHUR

This represents the sulphur that occurs in the compound, either free or chemically combined, unless barium sulphate is present, in which case the amount of sulphur in it is deducted from the amount of total sulphur.

6. ASH

The ash is the residue left after ignition and consists principally of the nonvolatile mineral fillers, together with their reaction products with sulphur.

7. SULPHUR IN ASH

The sulphur in ash consists of the sulphur from the mineral fillers and also part of the sulphur that was with the rubber, but which during ignition enters into combination with mineral fillers.

8. TOTAL COMPOUNDING INGREDIENTS

These consist of the residue obtained when the rubber is dissolved by mineral oil in the solution method of analysis. Total compounding ingredients corrected is the value obtained after deducting sulphur and organic matter present.

9. SPECIAL DETERMINATIONS

Special methods are given for the analysis of compounds that contain glue, carbon, antimony, and waxy hydrocarbons.

10. RUBBER

Up to the present time no simple method has been devised for the direct determination of the amount of rubber present in a vulcanized compound. Therefore an indirect method is proposed which will give satisfactory results in all cases known to-day, except where there are found to be present decomposible compounding ingredients, such as carbonates, cellulose, and high percentages of mineral rubbers. As practically all insulating compounds contain some of these ingredients, the said compounds when specified shall be analyzed in accordance with the Joint Rubber Insulation Committee's method as given in Section XII, 18.

When carbonates, talc, and asbestine are present more accurate results are obtained by the use of the Joint Rubber Insulation Committee's methods given in Section XII, 18, or by the solution method given in Section XII, 22.

If high percentages of mineral rubber are used, no accurate method is known. If cellulose is present the best results are obtained by the method given in Section XII, 19.

IX. PREPARATION OF SAMPLES

Before preparing a sample for analysis the analyst shall, by inspection, assure himself that it has not been contaminated. The sample shall be prepared by taking pieces from various parts of the original sample and separating them from foreign matter.

The rubber shall be ground to the required fineness on a rubber mill or cut with scissors so as to pass a No. 14 sieve.

Hard rubber should be prepared by rasping with a coarse file, cleaning with a magnet, and passing through a No. 14 sieve.

Crude, reclaimed, or unvulcanized rubber shall be sheeted out very thin on an experimental mill and shall be rolled in holland or other cotton cloth to prevent the sample from sticking.

If no mill is available, the sample shall be cut with scissors as fine as possible.

Samples of rubberized cloth shall be prepared by cutting into pieces, 1.5 mm square, and then mixing well.

Cements.—Evaporate to dryness in vacuo and analyze the residue as an unvulcanized sample. Distill a separate sample under reduced pressure if examination of the solvent is desired.

Packings.—The sampling of packings is described under each type of packing listed in Section XIII.

X. REAGENTS AND EQUIPMENT

1. ACETONE

The acetone shall be chemically pure and shall be freshly redistilled over anhydrous sodium carbonate, using the 56 to 57° C. fraction.

2. ALCOHOLIC POTASH SOLUTION

The alcoholic potash solution shall be of normal strength. It shall be made by dissolving the required amount of potassium hydroxide in alcohol which has been purified as follows:

Dissolve 1.5 g of silver nitrate in 3 cc of water and add it to 1,000 cc of alcohol. Dissolve 3 g of potassium hydroxide in the smallest amount of hot water, cool, add it to the alcoholic-silver nitrate solution, and shake thoroughly. Allow the solution to stand for at least 24 hours, filter, and distill. (Alcohol denatured with 10 per cent by volume of methanol may be used in place of ethyl alcohol.)

3. NITRIC ACID-BROMINE SOLUTION

The nitric acid-bromine reagent shall be prepared by adding a considerable excess of bromine to concentrated nitric acid and shaking thoroughly.

4. ZINC-NITRIC ACID SOLUTION

The zinc-nitric acid solution is made by adding 200 g of zinc oxide to 1,000 cc of concentrated nitric acid.

5. BARIUM CHLORIDE SOLUTION

Barium chloride solution shall be made by dissolving 100 g of crystallized barium chloride in 1 liter of distilled water, and adding two to three drops of concentrated hydrochloric acid. If there is any insoluble matter or cloudiness, the solution shall be heated on the steam bath overnight and filtered.

6. STANDARD 0.1 N POTASSIUM PERMANGANATE SOLUTION

Standard 0.1 *N* potassium permanganate solution is made as follows: Dissolve approximately 3.1 g of potassium permanganate in 1 liter of water, and after about one month filter through an ignited asbestos pad. Weigh 0.25 g of pure metallic antimony and transfer it to a 600 cc Erlenmeyer flask. Add 12 to 15 cc of concentrated sulphuric acid, 10 to 12 g of potassium sulphate, and heat until all the antimony is dissolved. Dilute to 250 cc with water, add 20 cc of concentrated hydrochloric acid, cool to 10 to 15° C., and titrate with permanganate solution until a faint pink color is obtained.

$$1 \text{ cc of } 0.1\ N\ \mathrm{KMnO_4} = \frac{\text{Wt. of metallic antimony}}{\text{cc of permanganate}}$$

7. STARCH IODATE PAPER

Starch iodate paper is prepared by impregnating filter paper with a solution obtained by heating 2 g of starch with 100 cc of water and, after solution, adding 0.2 g of potassum iodate dissolved in 5 cc of water.

8. RUBBER SOLVENT

The mineral oil used in the solution method of determining rubber and fillers shall have approximately the following properties: Vicosity at 68° F., 56 seconds; at 100° F., 45 seconds; flash point 270° F.; fire point 350° F.; and specific gravity 0.853, and shall be colorless.

9. ASBESTOS GOOCH CRUCIBLE

Prepare a Gooch crucible in the following manner: Amphibole asbestos is cut fine with shears, digested with 10 per cent caustic-soda solution, washed with water, and then digested with concentrated hydrochloric acid for a few hours on the steam bath. After it has been washed comparatively free from acid by decantation, the asbestos is shaken up with water and the resulting mixture is used in preparing the pad. The Gooch crucible is ignited, and is then ready for use.

10. BLANK ANALYSES

The purity of all materials shall be checked and determined by blank analyses.

XI. PRELIMINARY EXAMINATION OF SAMPLE

1. CARBONATES

Drop a small piece of the sample into a test tube containing concentrated hydrochloric acid saturated with bromine. If a stream of bubbles is given off, the method described in Section XII, 22, should be used.

2. ANTIMONY

Place a portion of the sample in a porcelain crucible and burn off the rubber. Treat the ash with concentrated hydrochloric acid. Dilute the solution to 10 times its volume, filter and saturate with hydrogen sulphide. If a precipitate of antimony sulphide is formed, the method described in Section XII, 11 or 22, should be used.

3. CARBON BLACK

Heat a portion of the sample with concentrated nitric acid until there is no more frothing. If the liquid is black, it indicates the presence of free carbon, and the test for this substance should be made.

4. BARIUM SALTS

A qualitative test for barium can conveniently be made on the residue after filtration in the total sulphur determination as described in Section XII, 6, by dissolving it in 10 per cent hydrochloric acid, filtering and then adding a few drops of 10 per cent sulphuric acid. A white precipitate of barium sulphate indicates the presence of barium in the sample.

5. PARAFFIN

If paraffin is present, it will solidify in the cold acetone extract as a white flucculent precipitate clinging to the sides of the flask.

6. GLUE

Extract a portion of the sample with a mixture of 32 per cent acetone and 68 per cent chloroform by volume for eight hours. Dry the sample and digest for one hour with hot water. Filter, cool, and add a few drops of a freshly prepared 2 per cent solution of tannic acid and allow to stand for a few minutes. If the solution becomes turbid, glue is present and should be determined as described in Section XII, 14.

XII. METHODS OF ANALYSIS

1. SPECIFIC GRAVITY

This shall be determined by the use of a pycnometer, using alcohol in place of water, to eliminate the errors due to air bubbles.

A = Wt. of pycnometer filled with alcohol,
B = Wt. of pycnometer filled with sample and alcohol,
C = Wt. of sample.

$$\text{Sp. gr.} = \frac{C}{C-(B-A)} \times \text{sp. gr. of alcohol.}$$

2. ACETONE EXTRACT

The extraction apparatus used here and for other extractions shall be of the type shown in the Journal of Industrial and Engineering Chemistry, **9** (1917), 314. The flask shall be heated so that the period of filling an empty siphon cup with acetone and completely emptying it will be between 2.5 and 3.5 minutes.

Place 2 g of rubber in a thimble made by folding a filter paper so that it will fit in the extraction cup, which is suspended in a weighed extraction flask. Extract the sample continuously for 8 hours, unless the solution in the thimble is still colored at the end of that time, when the extraction shall proceed for a further period of 4 hours or longer. For hard rubber the extraction period shall be a minimum of 72 hours. Carefully note all characteristics of the acetone extract, both when hot and cold. Distill off the acetone on a steam bath, recovering the acetone if desirable, at as low a temperature as possible. Loss of extract by bumping can be avoided by means of a gentle current of air.

Care must be taken to avoid allowing the flasks to stand on the steam bath after the solvent has been removed, because appreciable quantities of free sulphur may be lost by so doing. Dry the extraction flask and contents in an air bath for one hour at 70° C., cool, and weigh. Call the residue "acetone extract, uncorrected."

$$\text{Percentage of acetone extract, uncorrected} = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

Keep the sample for future determination.

3. CHLOROFORM EXTRACT

The rubber sample (Section XII, 2) is suspended in a second weighed extraction flask and extracted for 4 hours with chloroform. Care should be taken that any small particles of rubber, which are often carried down into the extract, are filtered off. Evaporate off the solvent and dry to constant weight, usually one hour at 70° C., cool, and weigh. The color of the chloroform solution should be recorded. Reserve the rubber for extraction with alcoholic potash.

$$\text{Percentage of chloroform extract} = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

4. ALCOHOLIC-POTASH EXTRACT

Dry the rubber from the chloroform extraction at about 70° C. to remove the chloroform, transfer to a 200 cc Erlenmeyer flask, add 50 cc of alcoholic-potash solution, and heat under a reflux condenser for four hours. Filter into a 250 cc beaker, wash with two portions of 25 cc of boiling alcohol; then with three 25 cc portions of boiling water, and evaporate the filtrate to dryness. Use about 75 cc of distilled water to transfer the residue to a separatory funnel. Acidify the solution with 10 per cent hydrochloric acid, testing with Congo red paper. Extract with four 25 cc portions of ether, unless the fourth portion should be colored, when the extraction must be continued until no further quantity can be removed. Unite the ether fractions and wash thoroughly with distilled water until free from acid (two washings are generally sufficient). Filter the ether solution through a plug of absorbent cotton into a weighed flask, wash with ether, evaporate, dry to constant weight at 70° C., cool, and weigh.

$$\text{Percentage of alcoholic-potash extract} = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

5. FREE SULPHUR

Add to the flask containing the acetone extract, uncorrected (Section XII, 2), to 60 cc of distilled water and 2 to 3 cc of bromine, and cover with a watch glass. Allow the flask to stand 0.5 hour on the side of the steam bath, then heat cautiously over the direct steam bath until the solution is practically colorless, filter into a 250 cc beaker, and dilute to about 175 cc with distilled water. Precipitate with barium chloride and determine in the usual manner.

$$\text{Percentage of free sulphur} = \frac{\text{Wt. of } BaSO_4 \times 0.1373}{\text{Wt. of sample}} \times 100.$$

6. TOTAL SULPHUR

Place 0.5 g of rubber in a porcelain crucible of about 75 cc capacity, add 15 cc of the nitric acid-bromine mixture, cover the crucible with a watch glass, and let it stand for 1 hour in the cold. Heat for an hour on the steam bath, remove the cover, rinse it with a little distilled water, and evaporate to dryness. Add 3 cc of nitric acid, cover, warm a short time on the steam bath, then let it cool. Carefully add in small portions, by means of a glass spatula, 5 g of sodium carbonate (weighed to 0.5 g). The watch glass is to be raised only high enough to permit the introduction of the spatula. The carbonate is allowed to slide down the side of the crucible and is not dropped directly into the acid. Rinse the watch glass with 2 or 3 cc of hot distilled water and stir the mixture thoroughly with a glass rod. Digest for a few minutes, spread the mixture halfway up the side of the crucible to facilitate drying, and dry on a steam bath. Fuse the mixture by heating over a sulphur-free gasoline flame.

Place the crucible in an inclined position on a wire triangle and start the ignition over a low flame. The tendency for the organic matter to burn too briskly is controlled by judicious use of the stirring rod with which the burning portion is scraped away from the rest. When part of the mass is burned white, a fresh portion is worked into it until all of the organic matter is destroyed. It is necessary to hold the edge of the crucible with tongs. Toward the last half of the operation the flame should be increased. It is unnecessary to heat the crucible to redness. With care a crucible can be used for at least 10 or 12 fusions.

After a fusion, allow the crucible to cool, place it in a 400 cc beaker, add sufficient distilled water to cover the crucible (about 125 cc), and digest on the steam bath for two hours, with occasional stirring. If the filtration can not be made on the same day, do not add the water, but allow the fusion to stand overnight.

Filter the solution into a covered 400 cc beaker containing 5 cc of concentrated hydrochloric acid, and wash the residue thoroughly with hot water. (A qualitative test for barium can be made on the residue.) Complete the acidification of the filtrate and washings and add 2 cc of concentrated hydrochloric acid in excess. Cover the beaker and heat the solution on the steam bath. The total volume of the solution should be 300 cc. The solution must be acid to Congo paper in order to insure the complete destruction of the carbonates. Precipitate with barium chloride and determine the sulphur in the usual manner.

$$\text{Percentage of total sulphur} = \frac{\text{Wt. of } BaSO_4 \times 0.1373}{\text{Wt. of sample}} \times 100.$$

7. TOTAL SULPHUR (ALTERNATIVE METHOD)

When this method is used the total sulphur determined represents the sulphur exclusive of that originally present as barytes or formed from barium salts, if present in the compound.

Place a 0.5 g sample in a 500 cc Erlenmeyer destruction flask (Pyrex glass). Add 10 cc of zinc oxide-nitric acid solution and moisten the sample thoroughly. If convenient, the mixture may be allowed to stand overnight. By so doing the sample becomes partly decomposed; this permits the addition of fuming nitric acid with no danger of ignition of the sample. Add 15 cc of fuming nitric acid and whirl the flask rapidly to keep the sample immersed to avoid ignition by too rapid oxidation. With some samples it will be found necessary to cool the flask under a stream of tap water.

When the solution of the rubber is complete, add 5 cc of saturated water solution of bromine and slowly evaporate the mixture to a foamy sirup. (For the determination of total sulphur in unvulcanized mixtures use 3 cc of bromine in place of bromine water.)

If particles of organic matter remain at the end of the evaporation, add a few cc of fuming nitric acid and reevaporate as before. Cool and add a few crystals of potassium chlorate to assist in the oxidation of the sulphur and the decomposition of any nitrates.

Place the flask on asbestos gauze and evaporate the mixture to dryness over a Tirrill burner. Then bake the mixture at the highest temperature of the burner until all nitrates are decomposed and no more nitrogen peroxide fumes can be detected. When the baking is complete, cool the flask, add 50 cc of (1: 6) hydrochloric acid and heat until solution is complete. (In case the original mixture contains barium sulphate or other barium salts, they will be precipitated at this point with consequent loss of sulphur in the case of the latter. If litharge is present in the mixture, lead salts, not otherwise removed, will be eliminated in the final washing with boiling water.) Filter the solution, dilute to 300 cc precipitate with barium chloride, and determine the sulphur in the usual manner.

$$\text{Percentage of total sulphur} = \frac{\text{Wt. of BaSO}_4 \times 0.1373}{\text{Wt. of sample}} \times 100.$$

8. ASH

Wrap a 1 g sample in a filter paper, extract with acetone for four hours, and transfer to a weighed, approximately 50 cc porcelain crucible. Ash the sample in a muffle furnace by heating at the following rate:

Time, minutes,	0	5	10	15	70	75	80	85	145
Temp. °C.,	[illegible]0	100	200	300	300	400	500	550	550

Remove crucible from the furnace, cool, and weigh.

If no furnace is available, distil off the rubber over a very small flame, not allowing it to catch fire, and ignite gently until burnt clean, cool, and weigh.

$$\text{Percentage of ash} = \frac{\text{Wt. of ash}}{\text{Wt. of sample}} \times 100.$$

9. SULPHUR IN ASH

Add 3 cc of nitric acid-bromine mixture to the ash (Sec. XII, 8), cover with a watch glass and heat for one hour, remove the cover, rinse it with a little distilled water, and evaporate to dryness. Complete the determination of sulphur as described under Section XII, 6.

Save the insoluble residue, after filtering the solution of the fusion mixture in water, for testing according to Section XII, 10.

10. BARIUM SULPHATE

The barium sulphate is calculated from the barium in the ash, which is determined as follows: Filter off the insoluble matter after the fusion and extraction in Section XII, 9, wash back into the original beaker with hot water, dissolve the residue in the beaker and any traces on the filter paper with hydrochloric acid, and heat the solution on the steam bath. Filter through the same filter as before and wash thoroughly with hot water. Nearly neutralize the solution with ammonium hydroxide, leaving it slightly acid. Saturate the cold solution with hydrogen sulphide, and when the lead sulphide has settled, filter into a 400 cc beaker and wash thoroughly. The total volume should be not over 200 cc. Precipitate with 10 per cent sulphuric acid and determine the sulphur in the usual manner. Barium sulphate determined above is assumed to have been added as such. Obviously, if barium carbonate is present, it must be determined in order that an undue correction will not be made.

Percentage of sulphur as barium sulphate

$$= \frac{\text{Wt. of } BaSO_4 \times 0.1373}{\text{Wt. of sample}} \times 100.$$

11. TOTAL ANTIMONY

When a qualitative test indicates that antimony is present, weigh out a 0.5 g sample, and transfer to a Kjeldahl flask, add 25 cc of concentrated sulphuric acid and 10 to 12 g of potassium sulphate, place a funnel in the neck of the flask, and heat until the solution becomes colorless. Cool and wash the funnel, dilute the solution to 100 cc with water, and transfer to a 400 cc beaker, dilute to 250 cc with hot water, and precipitate the antimony with hydrogen sulphide. Filter and transfer the precipitate to a Kjeldahl flask, add

15 cc of concentrated sulphuric acid, 10 to 12 g of potassium sulphate, and heat as described above until the solution is colorless. Wash the funnel, dilute the solution to 100 cc with water, add 1 to 2 g of sodium sulphite, and boil until all the sulphur dioxide is driven out.

This is shown when no blue color is obtained with starch iodate paper. Add 20 cc of concentrated hydrochloric acid, dilute to 250 to 275 cc with water, cool to 10 to 15° C., and titrate with standard 0.1 *N* permanganate solution until a faint pink color is obtained. If iron is found to be absent it is not necessary to precipitate the antimony with hydrogen sulphide and the second heating in a Kjeldahl flask may be eliminated.

$$\text{Percentage of antimony} = \frac{\text{Sb (Sec. X, 6)} \times \text{cc of permanganate}}{\text{Wt. of sample}} \times 100.$$

12. ANTIMONY IN THE ASH

This is determined in the ash of a 1 g sample. Transfer the ash to a 600 cc Erlenmeyer flask, add 12 to 15 cc of concentrated sulphuric acid and 10 to 12 g of potassium sulphate, and boil until solution is complete. It may be necessary to warm part of the sulphuric acid in the crucible to transfer any adhering particles to the flask. Rinse with the remaining portion of acid. Then complete the determination as described in section XII, 11.

$$\text{Percentage of antimony as } Sb_2O_4 \text{ in ash} = \frac{\text{Sb (Sec. X, 6)} \times \text{cc of permanganate} \times 1.26}{\text{Wt. of sample}} \times 100$$

13. FREE CARBON

Extract a 0.5 g sample for eight hours with a mixture of 68 per cent chloroform and 32 per cent acetone by volume.

Transfer the sample to a 250 cc beaker and heat on the steam bath until it no longer smells of chloroform. Add a few cc of concentrated nitric acid and allow to stand for about 10 minutes. Add 50 cc more of concentrated nitric acid, taking care to wash down the sides of the beaker, and heat on the steam bath for at least one hour. At the end of this time there should be no more bubbles or foam on the surface. Pour the liquid while hot into a Gooch crucible, taking care to keep as much as possible of the insoluble material in the beaker. Filter slowly with gentle suction and wash well by decantation with hot concentrated nitric acid. Wash with acetone and a mixture of equal parts of acetone and chloroform until the filtrate is colorless. Digest the insoluble material, which has been carefully retained in the beaker, for 30 minutes on the steam bath with 35 cc of 25 per cent sodium hydroxide solution. This treatment with alkali can be omitted if silicates are absent. Dilute to 60 cc with hot distilled water and heat on the steam bath. Filter the solution of alkali and wash well with hot 15 per cent sodium hydroxide solution.

Next wash the residue about four times with hot concentrated hydrocholoric acid. Neutralize the last washing with ammonia and test for the presence of lead with sodium chromate solution. If lead is present continue to wash with hot concentrated hydrochloric acid and finally wash with warm 5 per cent hydrochloric acid. Remove the crucible from the funnel, taking care that the outside is perfectly clean, dry it in an air bath for 1.5 hours at 110° C., cool, weigh, burn off the carbon at a dull red heat, and reweigh. The difference in weight represents approximately 105 per cent of the carbon originally present in the form of lampblack or gas black.

$$\text{Percentage of free carbon} = \frac{\text{Wt. of crucible with carbon} - \text{wt. of crucible after ignition}}{1.05 \times \text{Wt. of sample}} \times 100$$

14. NITROGEN CALCULATED AS GLUE

When glue is found to be present, a correction must be made for the natural protein in the rubber. The average amount of nitrogen is 0.4 per cent. See Section XII, 21, for calculations.

Extract a 2 g sample for eight hours with acetone. Remove the solvent from the sample, and transfer the latter from the filter paper to a 750 cc Kjeldahl flask. Add 25 to 30 cc of concentrated sulphuric acid, 10 to 12 g of sodium sulphate, and about 1 g of copper sulphate. Heat gently until the first vigorous frothing ceases, then raise the heat gradually until the liquid boils. Continue the boiling until the solution becomes clear. Allow the flask to cool, dilute carefully with 150 cc of water, and again allow to cool. Add 100 cc of 50 per cent sodium hydroxide solution, pouring it carefully down the side of the flask, so that it does not mix immediately with the acid solution. Add about 1 g of granulated zinc to prevent bumping and a piece of paraffin the size of a pea to diminish frothing. Connect the flask quickly with a condenser, the delivery tube of which dips into a 500 cc Erlenmeyer flask, containing 50 cc of 0.1 *N* sulphuric acid diluted to about 100 cc. Carefully swirl the flask to mix the contents and start to heat gently, increase the flame as the danger of foaming over diminishes, and finally boil briskly until about one-half of the liquid has passed over into the receiver. Add methyl red solution and titrate the excess acid by means of 0.1 *N* sodium hydroxide solution. A blank determination should be made.

Percentage of nitrogen as glue =

$$\frac{100\ (\text{cc } H_2SO_4 \times \text{normality} - \text{cc NaOH} \times \text{normality})\ (0.014)\ (6.5)}{\text{Wt. of sample}}$$

$$(\text{cc of } H_2SO_4 \times \text{normality} - \text{cc NaOH} \times \text{normality}) \times 4.55.^{1}$$

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[1] Simplified formula when a 2 g sample is used.

15. UNSAPONIFIABLE MATTER

If this determination is made the free sulphur shall be determined on a separate 2 g sample.

Add to the acetone extract obtained from two 2 g samples (Sec. XII, 2) 50 cc of N alcoholic-potash solution, heat on the steam bath under a reflux condenser for two hours, remove the condenser and evaporate to dryness. Transfer to a separatory funnel, using about 100 cc of water, add 25 cc of ether, and shake. Allow the two layers to separate thoroughly, then draw off the water layer. Continue the extraction of the water layer with fresh portions of ether until no more unsaponifiable matter is removed, unite the ether layers, and wash with distilled water. Transfer the ether to a weighed flask, distill off the ether, dry to constant weight at 70° C., cool, and weigh.

$$\text{Percentage of unsaponifiable matter} = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

16. HYDROCARBONS A

To the unsaponifiable matter Section XII, 15, add 50 cc of absolute alcohol, and heat on the steam bath for 0.5 hour. Let the flask stand in a mixture of ice and salt for one hour. Filter off the separated waxy hydrocarbons on filter paper, by applying a gentle suction. Wash with alcohol (95 per cent will do) which has been cooled in an ice-salt mixture. The funnel should be surrounded by a freezing mixture.

Dissolve the precipitate from the filter paper with hot chloroform, and catch the solution in a weighed 100 to 150 cc beaker. Wash the flask with hot chloroform which is added to the same beaker, in order to include any insoluble matter adhering to the walls of the flask. Evaporate off the solvent, dry to constant weight at 70° C., cool, and weigh.

$$\text{Percentage of hydrocarbons } A = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

17. HYDROCARBONS B

Evaporate the alcohol from the flask containing the alcohol soluble unsaponifiable material, add 25 cc of carbon tetrachloride, and transfer to a separatory funnel. Shake with concentrated sulphuric acid, drain off the discolored acid, and repeat with fresh portions of acid until there is no longer any discoloration. After drawing off all the acid, wash the carbon tetrachloride solution with repeated portions of water until all traces of acid are removed. Transfer the carbon tetrachloride solution to a weighed flask, evaporate off the solvent, and dry to constant weight at 70° C., cool, and weigh.

$$\text{Percentage of hydrocarbons } B = \frac{\text{Wt. of extract}}{\text{Wt. of sample}} \times 100.$$

18. RUBBER HYDROCARBONS (JOINT RUBBER INSULATION COMMITTEE)

Add to the flask containing the rubber residue from the alcoholic-potash extraction sufficient water to make the total 125 cc and then add 25 cc of concentrated hydrochloric acid. Heat for one hour at 100° C. Decant the supernatant liquid through a hardened filter paper on a Büchner funnel, 7 cm in diameter, using suction. Wash the residue with 25 cc of hot water and decant. (Although a Büchner funnel is recommended, it is permissible to use a 11 cm hardened filter paper with platinum cone in a 60° funnel.) Perform this entire treatment with water and hydrochloric acid three times. The rubber at this stage should be white and practically free from black specks of undissolved fillers; if not, continue the acid treatment until the black specks disappear. (If carbon is present, all the particles of rubber will be grayish, bluish, or black, depending on the form and quantity of carbon used. Black specks in light particles of rubber usually indicate the presence of lead sulphide which must be removed to prevent the formation of lead sulphate on igniting the residue C.) Add 150 cc of hot water to the flask and allow to stand on a steam bath or hot plate for half an hour and decant through the filter paper, repeating until washings are free from chlorides. Transfer all the rubber in the flask to the filter paper and dry as much as possible by suction. Wash the rubber with 50 cc of 95 per cent alcohol, using suction. Transfer the entire residue to a weighing bottle. Dry at 95 to 100° C. for an hour, cool in a vacuum desiccator under reduced pressure and weigh. Dry for 30 minutes, cool, and weigh, repeating this process until either constant weight is reached or the weight starts to increase. Let this weight be represented by C. The weight originally taken for the acetone extract determination shall be called A. Determine the ash (E) on a portion (D) of this residue (C), and sulphur (H) on the remaining portion (G). Also determine the sulphur (F) on the ash (E). Perform the ash determination as described under Section XII, 8, and the sulphur determination as described under Section XII, 6. Calculations:

$$\text{Percentage of rubber hydrocarbons} = 100\frac{C}{A}\left(1-\frac{H}{G}-\frac{E-F}{D}\right).$$

19. CELLULOSE

Treat 0.5 g of rubber previously extracted with a constant boiling mixture of acetone and chloroform with 25 cc of rubber solvent oil, Section X, 8, in a 250 cc lipped assay flask. Place in an air bath at a temperature of 145 to 150° C. until small particles can be seen distinctly in the supernatant liquid, which requires about three hours. Dilute with 15 cc benzene and 200 cc petroleum ether.

Filter through a Gooch crucible and wash the residue thoroughly with warm acetone. Treat repeatedly with hot 10 per cent hydrochloric acid until the residue is free from acid soluble fillers. Wash the pad well with boiling water, and run small portions of acetone through it until the filtrate is colorless. Wash with alcohol and dry at 105° C. to constant weight. Remove the pad from the crucible with the help of a pair of sharp pointed tweezers, using the under portion of the pad as a swab to clean the sides of the crucible, and place all of this material in a tared weighing bottle. Replace in the drying oven for about 10 minutes, cool and weigh.

Weight of weighing bottle, pad, insoluble fillers, and cellulose minus weight of weighing bottle equals weight of pad, insoluble fillers, and cellulose.

Transfer the contents of the weighing bottle to a 50 cc beaker and pour over it 15 cc of acetic anhydride and one-half cc of concentrated sulphuric acid, and allow to digest for at least one hour. on the steam bath. After the mixture has cooled thoroughly, dilute with 25 cc of 90 per cent acetic acid and filter through a weighed Gooch crucible. To guard against traces of the material being carried through, this filtration, as well as the ones to follow, must be very slow and only gentle suction can be used. Wash with hot, 90 per cent acetic acid, until the filtrate comes through absolutely colorless and then wash about four times more. Wash with acetone about five times. After having taken care that all of the material has been washed out of the beaker in which the acetylation took place, remove the crucible from the funnel, clean the outside thoroughly, and dry for two hours at 150° C. Cool and weigh. The original weight of crucible plus weight of pad, fillers, and cellulose minus weight of crucible after acetylation equals cellulose.

$$\text{Percentage of cellulose} = \frac{\text{Cellulose}}{\text{Wt. of sample.}} \times 100.$$

20. BARIUM CARBONATE

Barytes was determined by the calculation to barytes of all barium found in the sample. Obviously if barium carbonate is present, it must be determined in order that an undue correction will not be made. The determination is as follows: A 1 g sample, in a porcelain boat, is placed in a combustion tube through which passes a current of carbon dioxide. The sample is ashed in the tube. After ignition and cooling, in the atmosphere of carbon dioxide, the boat is removed, the residue finely ground in an agate mortar, transferred to a 250 cc beaker, and treated with 5 to 10 g of ammonium carbonate, 15 to 20 cc of strong ammonia water, and about 50 cc of distilled water. The mixture is boiled for 20 minutes, filtered, and the precipitate thoroughly washed to remove all soluble sulphates. The residue on the filter paper is washed back into the original beaker and about 10 cc of glacial acetic acid with sufficient water to make the total volume about 100 cc is added. This is heated to boiling and filtered through the same paper as before. Hydrogen sulphide is passed into the filtrate to precipitate the lead and the solution is subsequently treated as in the determination of barytes. The final weight of barium sulphate obtained is calculated to barium carbonate.

Percentage of sulphur required to convert barium present as carbonate to sulphate $=\frac{\text{Wt. of } BaSO_4 \times 0.1373}{\text{Wt. of sample}} \times 100.$

The rubber solvent method, Section XII, 22, has been found to be more rapid in the determination of barium carbonate, and as accurate as the above method.

21. CALCULATIONS (XII, 1 TO 20)

Percentage of acetone extract uncorrected equals percentage of acetone extract, uncorrected, minus percentage of free sulphur, and waxy hydrocarbons.

Percentage of waxy hydrocarbons equals percentage of hydrocarbons *A* plus percentage of hydrocarbons *B*.

Calculations for glue: Multiply the percentage of rubber as compounded by 0.004 and 6.5 and call this value *A*. Subtract *A* from the percentage of glue and call this value *B*. Substitute *B* for glue in calculating the percentage of rubber hydrocarbons and then complete the calculations.

Percentage of sulphur as antimony trisulphide equals percentage of total antimony multiplied by $\frac{S_3}{Sb_2}$ equals percentage of total antimony multiplied by 0.400.

Percentage of total antimony as trisulphide equal percentage of total antimony multiplied by $\frac{Sb_2S_3}{Sb_2}$ equals percentage of total antimony multiplied by 1.400.

Percentage of total sulphur corrected equals percentage of total sulphur minus percentage of sulphur as barium sulphate minus percentage of sulphur as antimony trisulphide. When barium carbonate is found the percentage of sulphur necessary to convert it to sulphate must be added to the total sulphur, corrected.

Percentage of ash corrected equals percentage of ash minus percentage of sulphur in ash plus percentage of sulphur as barium sulphate minus percentage of antimony in ash, calculated to Sb_2O_4.

Percentage of rubber hydrocarbon equals 100 minus the sum of the following percentages, except when it is determined by the Joint Rubber Insulation Committee's method

Acetone extract corrected.
Chloroform extract.
Alcoholic-potash extract.
Total sulphur, corrected.
Ash, corrected.
Carbon.
Glue.
Total antimony as trisulphide.

Percentage of rubber hydrocarbon plus 5 per cent of its weight is taken as "rubber as compounded," except when the sum of the percentages of the acetone extract corrected, chloroform extract, and alcoholic-potash extract is less than the figure represented by the arbitrary 5 per cent, as in many high-grade compounds. Percentage of rubber as compounded is then: Percentage of rubber hydrocarbon plus the sum of the percentages of the acetone extract corrected, the chloroform extract, and the alcoholic-potash extract.

Percentage of rubber by volume =

$$\frac{\text{Percentage of rubber as compounded} \times \text{sp. gr. of compound}}{0.94 \text{ (taken as the average sp. gr. of crude rubber)}}$$

Ratio of acetone extract corrected =

$$\frac{\text{Percentage of acetone extract corrected}}{\text{Percentage of rubber as compounded}} \times 100$$

Ratio of sulphur to rubber =

$$\frac{\text{Percentage of total sulphur, corrected}}{\text{Percentage of rubber as compounded}} \times 100$$

Statement of Results

Per cent
Acetone extract corrected.
Waxy hydrocarbons.
Chloroform extract.
Alcoholic-potash extract.
Free sulphur.
Total sulphur, corrected.
Ash, corrected.
Cellulose.

Per cent
Sulphur as barium sulphate.
Total antimony.
Sulphur as antimony trisulphide.
Carbon.
Glue.
Rubber hydrocarbon.
Rubber as compounded.
Rubber by volume

Ratio of acetone extract to rubber as compounded.
Ration of sulphur to rubber as compounded.
Specific gravity.

22. RUBBER SOLVENT METHOD

Weigh two portions (*a* and *b*) of 0.5 to 1 g each of the finely ground sample. Extract with a mixture of 32 parts by volume of acetone and 68 parts of chloroform for a minimum of 8 hours.[2]

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[2] B. S. Tech. Paper 162; Rubber Age and Tire News pp. 445–447; 1920.

(1) If the liquid in the extraction apparatus is still colored at the end of this time, the extraction should be continued. Remove the samples and put each into a 150 cc lipped assay flask, add 20 to 25 cc of the oil, cover with a watch glass and heat in an air bath at a temperature of 150 to 155° C. until solution appears complete and then 15 to 30 minutes more. Solution may be considered complete when the rubber colloid has been broken down and the oil seems quite clear. Remove the flasks from the air bath, cool to about 110° C., and add in a small stream 10 to 15 cc of benzene, while mixing thoroughly; allow to cool and then dilute with sufficient petroleum ether to fill the flasks to within about 2 cm of the top. Mix thoroughly, cover the flasks to prevent evaporation and allow the mixture to stand overnight.

(2) *Treatment of portion a.*—Prepare a Gooch crucible with finely divided asbestos that has been previously treated with strong caustic soda solution and concentrated hydrochloric acid and washed well with water. Ignite the crucible, cool and weigh; call this weight *c*. Filter the mixture by decantation through the crucible, using suction; wash well with petroleum ether, followed by warm acetone, and by a warm mixture of equal volumes of acetone and chloroform if the filtrate is dark. Remove as much as possible of the organic residue by washing; finally wash with hot alcohol. A portion of the fillers will remain in the flask. Dry the crucible and flask with their contents for one hour at a temperature of 105 to 110° C. Cool and weigh. Call the weight of the flask and contents *d* and of the crucible and contents *e*.

(3) Remove the acid-soluble compounding ingredients from the flask and Gooch crucible, collecting the solution in a 400 cc beaker, by adding to the flask and crucible a few cc of boiling alcohol. Allow to soak for two or three minutes and then wash two or three times with boiling water; let the flask cool, add 10 cc of concentrated hydrochloric acid and swirl the flask to bring the acid in contact with the compounding ingredients. Pour the acid from the flask into the crucible and let it stand until no more bubbles rise through the liquid. If carbonates are present, there is danger of loss by excessive frothing. This can be prevented by first adding a few drops of the acid to the crucible, sucking it through the pad. After the violent action has ceased, the remainder of the 10 cc of acid is added. When no more gas is evolved, draw the acid through the pad and again wash with 20 cc of acid, adding a little at a time. Wash well with hot water, and transfer as much as possible of the residue remaining in the flask to the asbestos pad. If by qualitative tests the sample was found to contain antimony, save the filtrate and washings for treatment as described in (4). Dry the flask and crucible for one hour at 105 to 110° C., cool and weigh. Call the weight of the flask *f*, and

that of the crucible containing the organic residue and acid-insoluble fillers, *h*. Burn the organic residue from the asbestos pad by igniting in a furnace at a temperature of 700° C., cool and weigh. Call this weight *k*. If the sample contains barytes, save the contents of the crucibles for treatment as described in (5).

(4) *Determination of sulphur present as antimony trisulphide.*[3]—Dilute the filtrate in the beaker from (3) to about 250 cc and precipitate the antimony with hydrogen sulphide. Filter off the antimony sulphide and wash the precipitate with hydrogen sulphide water to which a little hydrochloric acid has been added. Transfer the filter paper containing the antimony precipitate to a Kjeldahl flask, and add 12 cc of concentrated sulphuric acid and 5 g of potassium sulphate. Heat the solution gently until it is colorless, dilute to 100 cc and add 1 to 2 g of sodium sulphite. Boil until the sulphur dioxide is driven out. Dilute to 250 to 275 cc, cool to 10 to 15° C., and titrate with standard permanganate solution. Calculate the antimony present to antimony trisulphide and express the sulphur present as antimony trisulphide in percentage of the sample and call this *L*.

(5) *Determination of barytes.*—Transfer the contents of the crucible from (3) to a 50 cc porcelain crucible and fuse with 5 g of a mixture of equal weights of sodium carbonate and nitrate. Stir well during the fusion. Cool the crucible, put it into a 250 cc beaker, cover with distilled water and heat on the steam bath until all the solid material has been loosened from the crucible. Filter and wash the residue with warm water to free from sulphate. Wash the residue back into the beaker and dissolve in 10 per cent hydrochloric acid, using about 10 cc excess and keeping the solution warm. Filter off the asbestos, wash well with hot water, and precipitate the barium in the solution with 10 cc of a 10 per cent solution of sulphuric acid. Heat on the steam bath, filter off the precipitate, ignite and weigh the barium sulphate.

Then

$$\frac{100 \times \text{wt. of } BaSO_4 \times 0.1373}{\text{Wt. of sample } a} = \text{percentage of sulphur present as barytes.}$$

Call this value *m*.

(6) *Treatment of portion* b *for sulphur in compounding ingredients.*—Treat portion *b* as described under (2), but omit the weighing of the crucible. After the pad has been dried, transfer it to the flask. The last traces of the fillers can be removed from the sides of the crucible with wads of absorbent cotton moistened with a little warm water. Add to the fillers about 10 cc of bromine water, and swirl the flask to

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[3] S. Collier, M. Levin, and J. A. Scherrer, Determination of Antimony in Rubber Goods, India Rubber Jour., 64, 580; 1921.

moisten the contents. Add 20 cc of concentrated nitric acid saturated with bromine. Allow to stand for 15 minutes and then heat on the steam bath for one hour. Transfer the contents of the flask to a 75 cc crucible and evaporate to dryness. Complete the determination of sulphur by the method described in Section XII, 6. Let the value for the percentage of sulphur in compounding ingredients be represented by n.

Calculations.—The percentage of total inorganic compounding ingredients corrected equals:

$$\frac{100\ (e+d+k)-(c+f+h)}{0.5}+(m+L)-n$$

To determine the percentage of rubber hydrocarbon subtract from 100 the sum of the percentages of acetone, chloroform, and alcoholic potash extracts, free carbon, glue, total sulphur corrected, and total inorganic compounding ingredients corrected.

XIII. ANALYSIS OF PACKINGS

1. F. S. B. SPECIFICATION NO. 93, ASBESTOS-COPPER GASKETS, CORRUGATED

Scrape the asbestos from the corrugations. Place about 1 g of the asbestos in a weighed crucible and weigh. Ignite over a blast lamp until weight is constant. Cool and weigh. This weight minus the original weight of the crucible divided by 0.88 is the weight of the asbestos fibers, on the basis of 12 per cent water of constitution.

2. F. S. B. SPECIFICATION NO. 94, ASBESTOS METALLIC CLOTH SHEET PACKING AND GASKETS FOR HIGH-PRESSURE STEAM

(*a*) Preparation of Sample.—Cut small strips from different parts of the sample.

(*b*) Separation of Rubber Compound from the Wire and Fibers.—Transfer a weighed sample, about 1 g, to a 150 cc lipped assay flask and add 10 to 15 cc of the oil described in Section X, 8.

Heat at 135° C. until the rubber compound and compounding ingredients have formed a colloidal solution—about one-half hour. Pour through a wire-gauze sieve of about 80 mesh. Wash well with petroleum ether and follow with warm benzol and chloroform alternately. Repeat the heat treatment and washing until no more of the fillers are removed, collecting the filtrate and washings containing the rubber compound. Extract the fibers with chloroform for one-half hour and place in a tared crucible, dry at 105° C. for one hour, cool and weigh. The loss in weight represents the rubber compound in the packing.

(*c*) Analysis of the Fibers.—Pour the fibers on a sheet of glazed paper and remove from them all the pieces of wire with the aid of a lens and a pair of tweezers. Brush the fibers back into the same crucible, dry at 105 to 110° C. for one-half hour, cool and weigh. This weight minus the original weight of the crucible is the weight of fibers present in the sample. Ignite the fibers over a blast lamp to constant weight. This weight minus the original weight of the crucible, divided by 0.88, is the weight of asbestos fiber. Cellulose may also be determined by oxidation in a combustion furnace and weighing the carbon dioxide produced. Calculate the percentage of total fibers present.

3. F. S. B. SPECIFICATION NO. 95, ASBESTOS VALVE-STEM PACKING

(*a*) Preparation of Sample.—Prepare the sample for analysis by cutting cross sections of about one-half inch in length from different parts of the packing. Cut these lengths into very small pieces and mix thoroughly.

(*b*) Types 1 and 3.—*Lubricant.*—Extract a weighed sample, about 1 g, for eight hours with a mixture of 32 per cent acetone and 68 per cent chloroform, by volume. Remove the sample from the extraction apparatus and place on a No. 80 wire gauze and wash well with chloroform, agitating the sample to remove the graphite. Place the sample in a tared crucible, dry at 105 to 110° C. for one hour, cool, and weigh. This weight minus the tare weight is the weight of the fibers present, and the original weight of the sample minus the weight of the fibers is the weight of lubricant and graphite. Calculate to percentage of the finished packing.

(*c*) Analysis of the Fibers.—Place about 1 g of the fibers freed of lubricant and graphite in a weighed crucible and weigh. Examine the fibers as described in Section XIII, 2.

(*d*) Type 2.—Transfer a weighed sample, about 1 g, to a 150 cc lipped assay flask and treat as described in Section XIII, 2.

(*e*) Analysis of the Fibers.—Examine about 1 g of the fibers as described in Section XIII, 2.

(*f*) Type 4.—*Analysis of the fibers.*—Examine about 1 g of the fibers as described in Section XIII, 2.

4. F. S. B. SPECIFICATIONS NO. 96, COMPRESSED ASBESTOS SHEET PACKING, GRADES A AND B

(*a*) Acetone Extract Corrected.—Prepare the sample by cutting the material into strips and determine the acetone extract and free sulphur as described in Section XII, 2 and 5.

(*b*) Separation and Determination of Fibers and Rubber Compound.—Extract a 1-g portion of the sample with acetone for a minimum of eight hours. Treat the sample as described in Section XIII, 2, to separate the rubber compound from the fibers and also to examine the fiber.

(*c*) Determination of Rubber.—The rubber in this packing is assumed to be unvulcanized. Evaporate the filtrate to the original volume of the oil used; transfer to a tared 75-cc crucible. Burn off the oil, controlling the rate of combustion to prevent spattering caused by condensed moisture from the air by partially covering the crucible with a wire gauze. The rate of combustion is easily regulated. The ignition is completed in a muffle furnace or over a flame. Cool and weigh the ash. Correct it for sulphur in ash and for sulphur in barytes as described in Section XII, 21.

5. F. S. B. SPECIFICATION NO. 98, DIAPHRAGM PACKING

Follow procedure given in Section XII, 1 to 21.

6. F. S. B. SPECIFICATION NO. 99, FABRIC CONDENSER-TUBE PACKING

No analysis required.

7. F. S. B. SPECIFICATION NO. 100, FIBER PACKING

Cut the packing into narrow strips and place about 1 g in a tared weighing bottle. Dry in vacuo over concentrated sulphuric acid at room temperature to constant weight. The loss in weight represents the weight of water originally present in the packing. Calculate to percentage of the packing.

8. F. S. B. SPECIFICATION NO. 101, FLAX PACKING

See General Specifications for Textiles, issued by the Federal Specifications Board, in effect at date of issuance of proposal.

9. F. S. B. SPECIFICATION NO. 102, FLEXIBLE METALLIC PACKING

Heat a portion of the packing at a temperature of 550° F. for four hours. Examine to see if the metal has melted or oxidized and scales or cracks when bent.

10. F. S. B. SPECIFICATION NO. 103, HIGH-PRESSURE SPIRAL GLAND PACKING

(*a*) Determination of Rubber Core.—Cut off about 1 inch of the packing and weigh. Cut open and remove the rubber core and weigh it. Calculate the percentage of the rubber core to finished packing.

(*b*) Preparation of Sample.—Cut off cross sections about one-half inch long from different parts of the finished packing. Discard the rubber core and cut the remainder into very small pieces and mix well to procure a representative sample of the packing.

(*c*) Acetone Extract (Uncorrected).—Place a 1 g sample in an extraction apparatus and extract with acetone for a minimum of eight hours.

(*d*) FREE SULPHUR.—Determine free sulphur on the extract as described in Section XII, 5.

(*e*) DETERMINATION OF RESIDUAL LUBRICANT.—Extract the sample with 95 per cent alcohol for four hours. Evaporate the solvent on a steam bath and dry the extract for one hour at 70° C. and weigh.

(*f*) DETERMINATION OF FIBERS.—Place the extracted sample in a 150 cc lipped assay flask and separate and examine the fibers as described in Section XIII, 2.

(*g*) RUBBER FRICTION COMPOUND.—The percentage of rubber friction compound exclusive of core is the difference between 100 and the percentages of acetone extract corrected, residual lubricant, fibers and core.

(*h*) ANALYSIS OF THE RUBBER CORE.—See Section XII, 1 to 21.

11. F. S. B. SPECIFICATION NO. 104, LOW-PRESSURE SPIRAL GLAND-PACKING

(*a*) PREPARATION OF SAMPLE.—Prepare thin cross sections of the packing and reduce these to fine particles. Extract a 2 g portion of the sample with petroleum ether. Dry the sample at 60° C. for two hours. Allow to cool and weigh. The loss in weight represents the lubricant.

In order to prepare samples of the rubber compound for analysis, pull apart the layers of frictioned duck and cut them into strips about one-eighth inch wide. Extract the strips with petroleum ether and dry at 60° C. Strip the rubber from the duck to obtain a sample of the rubber compound for analysis.

Analyze rubber compound according to procedure given in Section XII, 1 to 21. If it is impossible to effect a mechanical separation of the rubber and fabric, and thus obtain a sample of the rubber compound, the following method may be used.

(*b*) ACETONE EXTRACT (UNCORRECTED).—Extract the petroleum ether extracted portion of the prepared sample with acetone, according to the procedure given in Section XII, 1 to 21.

(*c*) FREE SULPHUR.—The free sulphur present in the acetone extract is determined according to the procedure given in Section XII, 5.

(*d*) CHLOROFORM AND ALCOHOLIC POTASH EXTRACTS.—Follow procedure given in Section XII, 3 and 4.

(*e*) TOTAL SULPHUR (UNCORRECTED).—Follow procedure given in Section XII, 6 or 7. Correct for sulphur in barytes.

(*f*) DETERMINATION OF FABRIC.—Extract a 1 g portion of the prepared sample with a mixture of 32 per cent acetone and 68 per cent chloroform by volume for a minimum period of eight hours. If oil substitutes are present, follow with an alcoholic potash extraction. Place in a 150 cc lipped assay flask and separate the fibers and rubber compound as described in Section XIII, 2. Dry the fibers at 105° C.

for one hour, cool and weigh. Ignite the fibers and cool and weigh the ash.

(*g*) Determination of Rubber Hydrocarbon.—Transfer the ash from the fibers to the beaker containing the solution of the rubber compound and evaporate the liquid to the original volume of the oil used and transfer to a weighed 75 cc crucible. Burn off the oil, controlling the rate of combustion to prevent spattering caused by condensed moisture from the air by partially covering the crucible with a wire gauze. The rate of combustion is easily regulated. The ignition is completed in a muffle furnace or over a flame. Cool and weigh the ash. Correct it for sulphur in ash and for sulphur in barytes.

12. F. S. B. SPECIFICATION NO. 105, METALLIC PACKING—FIXED RING TYPE

Heat a portion of the packing at a temperature of 600° F. for four hours. Examine to see if metal has melted or oxidized and cracks or scales when bent.

13. F. S. B. SPECIFICATION NO. 106, PLASTIC METALLIC PACKING

Heat a portion of the packing at a temperature of 550° F. for four hours. Examine to see if the metal has melted or oxidized and scales or cracks when bent.

14. F. S. B. SPECIFICATION NO. 107, SEMIMETALLIC PACKING

(*a*) Preparation of Sample.—Discard the metallic member and cut off cross sections about one-half inch long from different parts of the finished packing.

Transfer a weighed sample, about 1 g to a 150 cc lipped assay flask and treat the sample and analyze the fibers as described in Section XIII, 2.

15. F. S. B. SPECIFICATION NO. 108, SOLID METALLIC PACKING

Heat a portion of the packing at a temperature of 600° F. for four hours. Examine to see if metal has melted or oxidized and cracks or scales when bent.

16. F. S. B. SPECIFICATION NO. 109, PACKING RINGS FOR BOILER BLOW-OFF VALVES

(*a*) Determination of Asbestos.—Weigh approximately a 1 g portion of the sample and ash over a blast lamp to constant weight. The weight of the ignited residue divided by 0.88 is the weight of the asbestos.

(*b*) Heat Test.—Heat a portion of the sample for four hours at a temperature of 425° F. Examine to see if the sample shows a tendency to disintegrate.

Place a few small pieces of the packing ring in concentrated hydrochloric acid and allow to stand for one hour. Examine to see if the sample shows a tendency to disintegrate.

17. F. S. B. SPECIFICATION NO. 110, CLOTH INSERTION RUBBER PACKING

Follow procedure given in Section XII, 1 to 21.

18. F. S. B. SPECIFICATION NO. 111, RUBBER PACKINGS AND GASKETS, MOLDED, SHEET AND STRIP

Follow procedure given in Section XII, 1 to 21.

19. F. S. B. SPECIFICATION NO. 112, WIRE INSERTION RUBBER PACKING

Follow procedure given in Section XII, 1 to 21.

20. F. S. B. SPECIFICATION NO. 113, TUCKS PACKING

Follow procedure given in Section XII, 1 to 21.

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