

FED. TEST METHOD STD. NO. 311
January 15, 1969
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FEDERAL TEST METHOD STANDARD

LEATHER, METHODS OF SAMPLING AND TESTING

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INFORMATION SHEET
ON
FEDERAL TEST METHOD STANDARDS

This Federal Test Method Standard is issued in looseleaf form to permit the insertion or removal of new or revised sections and test methods.

All users of Federal Test Method Standards should keep them up to date by inserting revised or new sections and test methods as issued and removing superseded and canceled pages.

New and revised material and cancellations will be issued under Change Notices which will be numbered consecutively and will bear the date of issuance. Change Notices should be retained and filed in front of the Alphabetical Index of the Standard until such time as they are superseded by a reissue of the entire Standard.

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LEATHER, METHODS OF SAMPLING AND TESTING

AUTHORITY. This standard is issued pursuant to the Federal Property and Administrative Services Act of 1949, as amended, and its application to the purchase of commodities referred to herein is mandatory on all Federal agencies.

SECTION 1

SCOPE AND CONTENTS

1.1 Scope. This standard describes the general methods for testing leather and leather products for conformance with the requirements of Federal and Military Specifications. In the event that conflict should occur between the requirements of this standard and those contained in a material specification or other type of procurement document on date of invitation for bid, the procurement document or specification shall govern.

1.2 Contents. The contents of this standard are as follows:

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SECTION 3

DEFINITION OF TERMS

3.1 Scope. The definitions of general terms include some of those encountered by personnel concerned with specifications and procurement of hides, leather and leather products by the Federal Government. Terms adequately defined by unabridged dictionaries are not generally included.

3.2 General terms.

3.2.1 Alligator-grained leather. Leather of various types, such as calf, sheep, or cattleshide embossed to resemble the grain of alligator hide.

3.2.2 Apron leather. Any one of several varieties of leather used in connection with textile machinery and blacksmith aprons. Comber and Gill Box Apron leather is soft, mellow, tough leather, tanned from steer hides, heavily stuffed and boarded or otherwise softened. Rub Roll apron leather is a flexible but firm, dry, strong leather.

3.2.3 Aspergillus niger. One of the most common mold growths found on leather, usually greenish or blackish in color.

3.2.4 Back. A crop with the head trimmed off behind the horn holes. (OZUP in figure 1.)

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3.2.5 Bag leather (Also known as case leather). A general term for leathers used in traveling bags and suitcases. It does not include the light leathers employed for women's fancy handbags. The staple material for bag and case leather at present is leather made from the hides of animals of the bovine species, but heavy sealskins and goatskins are also used.

3.2.6 Barkometer. A hydrometer used for determining the specific gravity of tanning solutions. A specific gravity of 1.000 is equivalent to 0° barkometer (Bk), and each additional degree Bk is equivalent to an increase of 0.001 in specific gravity.

3.2.7 Baseball leather. Leather used for covers of baseballs. The better grades of balls have covers of alum-tanned horsehide front leather. Some cheaper grades are made of kip and sheepskins.

3.2.8 Bate. To treat unhaired hides or skins with a warm aqueous solution of enzymes in order to remove certain undesirable nitrogenous constituents.

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3.2.9 Beam. A convex wooden slab sloping downwards from about waist height, over which a hide is placed for trimming off excess flesh and ragged edges and for unhairing by hand.

3.2.10 Belting butt. A double back with the tail cut off at the butt line. (RUT'S in figure 1)

3.2.11 Belting butt bend. A double bend with the tail cut off at the butt line. (RR'S'S in figure 1)

3.2.12 Belly. That part of the hide below the belly line. (VWP'P in figure 1) For steerhide leather, the belly line (RU) passes through a point at or above the top of the rear break. For cowhide leather, the belly line passes through a point at or above the top of the front break and a point not more than 2-1/2 inches below the top of the rear break.

3.2.13 Bend. A back with the shoulder cut off at right angles to the backbone line at the break of the fore flank. (OYR'P in figure 1)

3.2.14 Biff. To beat a salted hide that has been placed on a rack, in order to shake loose salt from the hair.

3.2.15 Bisulfiting. The treatment of hot solutions of vegetable tanning extracts with sodium bisulfite in order to increase their solubility and rate of take-up by hides.

3.2.16 Bleaching. (1) The process of removing oxidized tannins and insoluble materials from the surface layers of leather, particularly sole leather, in order to prevent crackiness of the grain and to improve color. It is performed by dipping the leather in a weak alkaline solution to render the tannin readily soluble, dipping in water, neutralizing in weak acid solution, and washing. (2) The process of lightening the color of chrome leather by treating with synthetic tannins or precipitating white pigment in the surface of the leather.

3.2.17 Bleeding. The transfer of materials exuded from leather to other material that comes into contact with it. It is usually designated as staining.

3.2.18 Blocking. The adhesion between touching layers of similar or dissimilar material, such as occurs under moderate pressures during storage or use.

3.2.19 Bloom. A light-colored deposit of ellagic acid appearing on the grain surface of leather tanned with certain pyrogallol tannins, such as

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myrabolans, valonia, and dividivi. The appearance may be objectionable for some purposes, but bloom does not significantly affect the other physical properties of the leather.

3.2.20 Blue. Usually in the phrase "in the blue". Applied to hides or skins that have been chrome-tanned but not dyed or fat liquored.

3.2.21 Boarded leather . Leather on which the grain has been accentuated by folding the grain side in and working the leather back and forth. Hand boarding is done with a curved cork board attached to the worker's arm and rolled over the folded skin.

3.2.22 Boardy. Adjective applied to stiff, inflexible leather.

3.2.23 Break. (1) Heavy leather - The places, in the area where the fore shank and hind shank join the body of the hide, where the texture of the leather changes quite sharply from the firm, close weave of the bend to a loose, open texture. (2) Shoe upper leather - The superficial wrinkling formed when the leather is bent, grain inward, with a radius of curvature like that formed at the vamp of a shoe in walking. Adjectives commonly used to describe this characteristic are tight, loose, coarse, and pipey.

3.2.24 Brining. A process of curing hides by washing and soaking in a concentrated salt solution.

3.2.25 Brush coloring. The application of dyestuffs by brushing.

3.2.26 Buck sides. Cattlehide shoe upper leather finished to resemble buckskin.

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3.2.27 Buckskin. Leather from deer and elk skins; used for shoes, gloves and clothing. Only the outer cut of the skin from which the surface grain has been removed may be correctly defined as "genuine buckskin". Leather finished from the split or under cut of deerskin must be described as "split buckskin".

3.2.28 Buffalo. Buffalo leather is made from the hides of domesticated water buffalo of the Far East, not the American bison.

3.2.29 Buffing. (1) Removing minor blemishes from the grain with a knife or abrasive. (See Snuffed top grain) (2) Producing a velvet surface on leather, usually with an emery wheel. (3) Buffing leather is a light cut of the grain portion used for bookbindings, pocketbooks, etc., but not for upholstery.

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3.2.30 Bullhides. Hides from bulls are characterized by thick and rough head, neck and shoulders, and by coarse flanks. Bullhides are often poor in quality and heavy, ranging from 60 pounds up.

3.2.31 Butcher cuts. Damage to hides caused by improper removal from the animal. Damage is usually in the form of cuts or furrows on the flesh side.

3.2.32 Butt. That part of the hide or skin covering the rump or hind part of the animal.

3.2.33 Cabretta. Skin of Brazilian hair sheep used principally for glove leathers. Term probably derived from Spanish "Cabrito", or similar Portuguese or Italian word. (cf. Cape)

3.2.34 Calf leather. Leather made from the skins of young cattle from a few days up to a few months old, the skins weighing up to 15 pounds. Calf leather is finer grained, lighter in weight and more supple than cowhide or kip leather.

3.2.35 Cape (skin or leather). Skin of South African hair sheep. Fine-grain leather, superior to wool sheep for gloves and garments. Loosely applied to all hair sheep, but should be qualified to show origin, if other than South African. (Uncertain whether term is derived from "Caper" (Goat) or from "Cape Town".

3.2.36 Carding leather. A type of side leather used on the cards of textile machinery.

3.2.37 Carpincho leather. Leather from the skin of the carpincho, a large South American rodent. The skin is used in making glove leather, usually chrome tanned and washable. In the glove-leather trade, carpincho is classified as a pigskin. It resembles pigskin in appearance, because of the occurrence of bristle holes in straight-line groups, usually with 5 but may vary between 4 to 7 holes in a group.

3.2.38 Case leather (Also known as bag leather). A general term for leathers used in travelling bags and suitcases. It does not include the light leathers employed for women's fancy handbags. The staple material for bag and case leather at present is leather made from the hides of animals of the bovine species, but heavy sealskins and goatskins are also used.

3.2.39 Chamois leather. A soft pliable absorbent oil tanned leather which is recognized in this country and abroad as being made from sheepskin, from which the outer or grain side has been split prior to tanning, known technically as a flesher.

3.2.40 Chrome retan. Term applied to leather tanned first with chromium salts, then retanned with vegetable extracts.

3.2.41 Chrome retannage. Retannage with chromium salts.

3.2.42 Chrome tannage. Tannage of leather with chromium compounds. Chrome tanned leather is often distinguished from other kinds by its greenish color, particularly of a cut edge.

3.2.43 Cockle. A hard warty growth on sheepskin.

3.2.44 Collagen. The principal fibrous protein in the corium or derma layer of a hide or skin.

3.2.45 Colorado steer. A side-branded steerhide, not necessarily from Colorado.

3.2.46 Comber leather. A steer-hide leather, heavily stuffed and usually hand boarded, used in textile combing machines.

3.2.47 Combination tanned. Formerly, tanned with a blend of vegetable extracts. Today, tanned with two or more types of tanning materials such as chromium compounds and vegetable extracts, or chromium compounds and synthetic tanning materials.

3.2.48 Cordovan. Leather made from the tight firm shell portion of horse butts. Cordovan has very fine pores and a characteristic finish, and is very durable.

3.2.49 Corrected grain. (See Snuffed top grain.)

3.2.50 Country hides. Hides taken off by butchers and farmers. The quality is usually lower than that of packer hides because country hides are removed by less skilled hands and are not cured as well as packer hides.

3.2.51 Cowhide. Term specifically applied to leather made from hides of cows, although the term is sometimes loosely used to designate any leather tanned from hides of animals of the bovine species.

3.2.52 Crop. A side with the belly trimmed off. (OO'P'P in figure 1)

3.2.53 Crushed leather. Chrome-vegetable retanned leather with the grain accentuated by plating or other process.

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3.2.54 Crust. Used as an adjective or in the phrase "in the crust". Refers to leather that has been tanned but not finished. (See Rough)

3.2.55 Curing. Treating raw hides or skins so as to minimize putrefaction and bacterial action, but to enable the skins to be wet back conveniently in preparation for tanning. (See Brining, Dry salting, Dry pickling, Green salting and Pickle.)

3.2.56 Curling temperature. The temperature at which noticeable curling occurs, when gradually heating a leather specimen in water.

3.2.57 Currying. A process of treating tanned hides with oils and greases to prepare them for belting, sole, harness leathers, etc.

3.2.58 Deep buff. The first cut or split underneath the top grain or machine buff on which no traces of the grain remain.

3.2.59 Deerskin. In glove leather, a deerskin tanned and finished with the grain surface intact.

3.2.60 Degrained leather. Leather from which the grain has been removed after tanning, by splitting, abrading, or other process.

3.2.61 Degras, moellon. The direct oxidized oil pressed out of sheepskin after tannage with cod or other oil.

3.2.62 Doeskin. Commercial term for white leather from sheep or lamb-skin, tanned with alum and/or formaldehyde.

3.2.63 Double-dressed. As applied to chamois skins, with the grain removed and buffed or sueded on both surfaces.

3.2.64 Double shoulder. The fore part of the hide cut off at right angles to the backbone line at the break of the fore flank, with the belly cut off and the head cut off behind the horn hole. (R'UT'S' in figure 1)

3.2.65 Drawn grain. Shrunken, shriveled, or wrinkled grain surface of leather.

3.2.66 Drumhead leather. (See Parchment.)

3.2.67 Dry pickling. A method of curing skins from wool sheep with sodium sulfate and sodium chloride.

3.2.68 Dry salting. A method of curing hides in which the hides are first green-salted and then dried.

3.2.69 **Dubbing (Also Dubbin).** A mixture primarily of oils and fats used for restoring fatty matter to military footwear in the field.

3.2.70 **Electrified shearling.** Shearling in which the wool has been straightened by a special process. (Also Electrified lambskin)

3.2.71 **Elk leather.** Trade term used to designate chrome-tanned cattlehide for uppers of work shoes, hunting boots, some children's shoes, and others requiring flexibility and durability. More properly, elk-finished cowhide. Leather from elk hide is more properly called "buckskin".

3.2.72 **Embossed leather.** Leather which has been ornamented with a geometrical or fancy design by heavy pressure in a machine.

3.2.73 **Extract.** A liquid, powder, or solid concentrate of vegetable tannin obtained by the extraction of tannin from natural sources.

3.2.74 **Factory sole leather.** One of the two principal types of sole leather. It is tanned and finished to have more flexibility and compressibility than *finder's* sole leather, and is more suitable for use in shoemaking machinery. (See *Finder's* sole leather)

3.2.75 **Fancy leather.** Leathers made from hides and skins of all kinds which have commercial importance and value primarily because of grain or distinctive finish, whether natural or the result of processing. Such processing may be graining, printing, embossing, ornamenting (including gold, silver, and aluminum finishes), or any other finishing operation enhancing the appeal of the leather.

3.2.76 **Fat liquor.** An emulsion of oils or greases in water, usually with an emulsifying agent, used to lubricate the fibers of the leather.

3.2.77 **Fat wrinkle.** Wrinkles in the grain of leather, caused by fat deposits in the live animal.

3.2.78 **Fiberboard.** A firm, but somewhat flexible, composition material in sheet form, made from new, long vegetable fibers. Used for counters, insoles, midsoles, and heel lifts. The term is often loosely applied to boards made from scrap material or short-fibered stock, such as chip-board, which has inferior physical properties in the uses mentioned. (See also Leatherboard)

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3.2.79 Finder's sole leather. One of the two principal types of sole leather. It has less flexibility and compressibility than factory sole leather and is more suitable for use in shoe repair. (See also Factory sole leather.)

3.2.80 Finish. Materials applied to the grain and sometimes the split surface of the leather to cover blemishes, create smoothness, and give uniformity of color and appearance which may vary from dull to glossy.

3.2.81 Flesh. The inner side of a hide or skin.

3.2.82 Flesher. The flesh split or undercut of a sheepskin, split before tanning. (See Chamois)

3.2.83 Flint dried. Dried in air without other curing.

3.2.84 Formaldehyde tannage. Tannage used especially for white leathers and washable glove leathers.

3.2.85 French kid. Leather tanned from kidskin by an alum or vegetable process.

3.2.86 Frigorifico hides. Cattlehides from South American slaughtering and freezing plants, cured in brine and salted.

3.2.87 Frizing. In tanning Mocha glove leather, a process of removing the grain surface involving long liming for not less than a month, during which the elastic structure of the grain layer is destroyed. (Also Friezing)

3.2.88 Front. The forepart of a hide or skin. Particularly in horsehide leathers, the front is used for garments, baseballs, etc. It is the part left when the butt is cut off about 22 inches from the root of the tail.

3.2.89 Full grain. Having the original grain surface of the skin.

3.2.90 Gasket leather. (See Hydraulic leather)

3.2.91 Gill box leather. A leather used in textile machinery, similar to comber leather.

3.2.92 Glazed (Glace) kid. Chrome-tanned goatskin and kidskin leather, either black or in colors, which has a glazed finish.

3.2.93 Glove leather. Term covering two distinct classes: (1) The leather used for dress gloves (including those for street, riding, driving, and sports wear) made predominantly from sheep and lamb skins and to a lesser degree from deer, pig, goat, kid and Mocha skins. (2) The leather used for

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utilitarian or work gloves made from a variety of hides and skins of which the most important are horsehides, cattlehide splits, calfskins, sheepskins and pigskins.

3.2.94 Glove splits. Split chrome-tanned cattlehide leather used for work gloves.

3.2.95 Grain. The outer or hair side of a hide or skin. Also used as an adjective referring to that side.

3.2.96 Grained leather. Any leather on which the original natural grain has been changed or altered by any method, process or manipulation.

3.2.97 Green salting. A process of curing hides by treating them with salt on the flesh side and stacking in pile to cure for a period of ten days or more.

3.2.98 Grub hole. A hole through the hide caused by the penetration of the grub of the warble fly.

3.2.99 Gusset leather. A soft flexible leather used for gussets in shoes, bags, and cases.

3.2.100 Hair-on leather. Leather tanned without removing the hair from the skins or hide.

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3.2.101 Hand buffs. A term used to describe upholstery leather of the same type as full top grain except that the surface of the hide is lightly snuffed or sandpapered all over. Such snuffing removes only the top of the hair follicles. (Also snuffed top grain, corrected top grain, top grain snuffed.)

3.2.102 Harness leather. A self-explanatory term sometimes so defined as to include collar and saddlery leathers. Harness leather, including the related items mentioned, is practically all made of vegetable-tanned cattlehides except for a considerable quantity of pigskins used for making saddle seats.

3.2.103 Hat leather. Usually sheepskin or calfskin for sweatbands of hats. The grain splits of sheepskin are vegetable-tanned for this purpose.

3.2.104 Head. That portion of the hide from the snout to the flare into the shoulder.

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3.2.105 Heavy leather. A somewhat indefinite term, generally understood to include vegetable-tanned sole, belting, strap and mechanical leathers made from unsplit cattlehides. Also refers to the thick side of leather.

3.2.106 Hide. The pelt of a large animal, such as cow, horse, etc. Also used interchangeably with skin.

3.2.107 Hide grades. Standard hide grades, take-up and delivery practice are given in the booklet, "Approved Standard Practice Governing the Take-Up and Delivery of Domestic Packer Hides", published by the Tanners' Council of America, 411 5th Avenue, New York 16, New York.

3.2.108 Hide powder. Purified, shredded rawhide used as a reagent in the determination of tannins. Standard hide powder is any lot of hide powder officially approved by the American Leather Chemists Association.

3.2.109 Hide substance. The nitrogen content of leather multiplied by the factor 5.62.

3.2.110 Horsehide leather. Leather made from the hide of a horse or colt. (See Cordovan and Front)

3.2.111 Hydraulic leather. A collective term sometimes used for the cattlehide leathers (vegetable, chrome, or combination tannage) with special stuffing added, which are used in pump valves, as piston packing, and so forth.

3.2.112 Indian tanned. Combination tanned with alum and vegetable tannins.

3.2.113 India-tanned. Term applied to hides and skins from India, considered as a semi-tanned raw material and generally retanned in the U.S.A. before finishing.

3.2.114 Iron. A term used for measuring thickness of sole leather. One iron equals 1/48 inch (0.53 millimeters).

3.2.115 Iron tannage. Tannage with salts of iron.

3.2.116 Kangaroo. Leather made from the hide of the kangaroo.

3.2.117 Kid. Originally referring to leathers made from the skins of immature goats, the term is now rather loosely applied to glove and shoe leathers made from goatskins.

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3.2.118 Kip. Skin from a bovine animal in size between a calf and a cow, weighing in green-salted condition approximately from 15 to 30 pounds.

3.2.119 Lace leather. A form of rawhide leather (from cattlehides) for lacing sections of power-transmission belts; sometimes prepared also with an alum and oil, chrome, or combination tanning.

3.2.120 Lambskin leather. Term applied to leather from either lambskins or sheepskins, which are practically indistinguishable after tanning.

3.2.121 Larrigan leather. Oil-tanned light cattlehides, used largely for moccasins.

3.2.122 Latigo leather. A type of lace leather, alum and vegetable tanned, used in saddlery.

3.2.123 Leatherboard. A type of fiberboard in which the fiber content is at least 75 percent leather, usually with asphaltic or resinous binder.

3.2.124 Levant. Term applied to goatskin on which the grain pattern is accentuated in tannage. Goatskin embossed to give a Levant pattern is properly described as "Levant-grained goatskin". Sheep, seal, and other skins bearing this pattern should not be described as "Levant leather" but as "Levant-grained sheepskin," etc.

3.2.125 Lining leather. Any leather used for making shoe linings, which includes sheep, lamb, kid, goat, cattle, calf, and splits.

25 3.2.126 Load. The amount of nonprotein material in vegetable-tanned leather.

3.2.127 Loading. The addition of glucose, magnesium sulfate, or other materials to give leather the physical properties needed for working in modern shoe machinery. (Also known as Filling or Stuffing.)

3.2.128 Machine buffs. That cut of the hide from which a buffing of approximately 1/64 inch (one ounce) in thickness has been removed from the grain. This should leave a portion of the grain on almost the entire hide.

3.2.129 Manufacturer's leather. (See Factory sole leather.)

3.2.130 Matadero hides. Hides from Argentina corresponding to city butcher or smaller packer hides of the United States.

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3.2.131 Mechanical leather. A collective term for many types of leather used in connection with textile and other machinery.

3.2.132 Meter leather. A speciality leather made mainly from sheepskins treated to make it impermeable and used for the measuring bags of gas meters.

3.2.133 Mineral tanned. Tanned with chemical compounds of mineral origin, (chromium, zirconium or alum compounds) without the use of vegetable tanning materials.

3.2.134 Moellon. (See Degras)

3.2.135 Morocco grain. Vegetable-tanned fancy goatskin leather having a distinctive pebbled grain.

3.2.136 Morocco leather. Vegetable-tanned fancy goatskin leather having a distinctive pebbled grain.

3.2.137 Mouton. A sheepskin shearling tanned and finished for use as a fur; usually with wool straightened.

3.2.138 Mukluk leather. Leather usually made from deer, elk and similar skins. It is tanned white with formaldehyde alum or syntans. It is very permeable to moisture vapor and retains its flexibility at low temperatures.

3.2.139 Napa leather. Chrome, alum, or combination tanned sheepskin glove leather, drum colored.

3.2.140 Native hide. A cattlehide without a brand.

3.2.141 Oak tannage. Originally, the tannage of leather entirely (or nearly so) with oak bark, later the tannage with a blend containing oak tannin. Now loosely applied to any tannage of heavy leather with vegetable extracts.

3.2.142 Offal. Parts of hides not used for standard grades of outsole leathers; the heads, shoulders, and bellies of heavy leather.

3.2.143 Oiling off. Coating the surface of leather with oil.

3.2.144 Oil tannage. Tannage with cod oil or other oxidizing oil, usually of marine origin.

3.2.145 Ooze. Traditionally refers to a nap produced on vegetable-tanned leather. Also refers to other tannages sueded or napped on the grain side.

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3.2.146 Ounce. A term used to indicate weight or substance of certain kinds of leather (such as upholstery, bag, and case leather). In theory it is based upon the assumption that one square foot of leather will weigh a certain number of ounces and will uniformly be of certain thickness; hence, a three-ounce leather theoretically would be one square foot of leather weighing three ounces. In practice, this varies because of specific gravity of tanning materials used and for that reason a splitter's gauge has been adopted which controls the commercial thickness of leather when sold by the square foot. An ounce is equivalent in thickness to $1/64$ inch = 0.0156 inch = 15.6 mils = 0.4 millimeters.

3.2.147 Pac leather. Highly water resistant leather used by lumber men, hunters and others for outdoor use.

3.2.148 Packer hides. Hides from meatpacking houses.

3.2.149 Packing leather. (See Hydraulic leather)

3.2.150 Parchment. Traditionally alum-tanned sheepskin or slunk used for special documents, drum heads, lamps, etc.

3.2.151 Patent leather. Leather with a glossy impermeable finish produced by successive coats of drying oils, varnish, or synthetic resins.

3.2.152 Pebbled grain. An embossed-leather grain finish resembling a pebbled surface, ranging from fine pebbled Morocco goat to heavy Scotch grain upper leather.

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3.2.153 Peccary. A wild boar found in Central and South America. The skin is usually chrome tanned and shaved to light weight for glove leathers. It is distinguishable from pigskin and Carpincho skins by the fact that bristle holes occur in straight line groups of three.

3.2.154 Pelt. A raw skin with the hair on. Usually refers to fur animals.

3.2.155 Persians. India-tanned hair sheepskins.

3.2.156 Picker leather. Leathers used for pickers in textile machinery, and having a wide range of properties. Some are hard rawhide buffalo leathers, others glycerine-treated rawhide and still others belting leather.

3.2.157 Pickle. To treat unhaired hides with a solution of salt and acid in order to prepare them for tannage or for temporary preservation until they reach the tannery.

3.2.158 Pigment-finished leather. Leathers finished with compounds containing opaque pigments which more or less conceal the grain pattern. Split leathers are often finished with pigments and embossed to simulate grain.

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3.2.159 Pigskin. Leather made from the skins of pigs or hogs. In the glove leather trade "Pigskin" includes peccary and carpincho.

3.2.160 Pin Seal. Natural grain sealskin tanned for fancy leather. Imitations on other skins should be described as "pin-grain sheepskin", pin-grain goatskin", etc.

3.2.161 Pipeyness. Characteristic of loose grain leather which forms coarse wrinkles on bending with the grain inward.

3.2.162 Plating. Pressing leather with a heated metal plate, usually smooth, under high pressure.

3.2.163 Pocket-shaped. As applied to chamois skins, a skin trimmed in the form of a rectangle with the two corners at one end rounded.

3.2.164 Quebracho. A tanning material extracted from the wood of a South American tree.

3.2.165 Rawhide. Cattlehide that has been dehaired, limed, often stuffed with oil or grease, and has sometimes undergone other preparation, but has not been tanned. It is used principally for mechanical purposes, such as belt lacings, loom pickers, gaskets, pinions, gears, and for hand luggage, shoe laces, snow shoes, etc.

3.2.166 Raw streak. An untanned center layer of leather, visible in cross section as a light-colored streak, especially as applied to heavy leather.

3.2.167 Reconstituted leather. Material composed of collagen fibers, obtained from macerated hide pieces, which have been reconstructed into a fibrous mat.

3.2.168 Retan. A modifying secondary tannage applied after intermediate operations following the primary tannage.

3.2.169 Rigging leather. A strong flexible, vegetable-tanned leather.

3.2.170 Roan. A sheepskin, not split.

3.2.171 Roller leather. Vegetable-tanned sheep or calfskins used for cots or covers on the upper rolls of cotton-spinning machinery.

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3.2.172 Rolling. A tannery operation in which the grain surface is compressed and smoothed under pressure by rollers.

3.2.173 Rough, rough tanned, in the rough. Terms applied to cattlehide leathers tanned but not finished. (See Crust)

3.2.174 Russet. A term of varied meaning in the leather trade, since it connotes both color and tannage. Russet calf is the natural color of unfinished calf leather resulting from tannage by vegetable extracts. Russet harness is a completely finished leather of bright, clean, uniform color and finish. Russet sheepskin originally was leather tanned in cold-leached hemlock bark, used for shoe linings, with color resulting from the hemlock; now sheepskin colored as though tanned by vegetable extracts. Russet upholstery is leather tanned but not finished.

3.2.175 Russia leather. Originally a Russian calfskin shoe leather distinguished by its odor of birch oil. Now in the U.S. A., a fancy leather.

3.2.176 Saddle leather. Vegetable-tanned cattlehide leather for harness and saddlery, usually of a natural tan shade and rather flexible.

3.2.177 Saladero hides. Argentinian hides corresponding to small-packer hides in the U. S. A.

3.2.178 Salt stain. Discoloration on the surface of hides and skins, developed during the curing process.

3.2.179 Scotch grain. A pebbled pattern embossed on cattlehide or calf leather.

3.2.180 Scud. Remnants of epithelial tissue, hair, dirt, etc., left in the hair follicles after unhairing.

3.2.181 Scudding. Removal of scud from unhaird hides by scraping with a blade, either by hand or machine.

3.2.182 Shank. Leg portion of hide pattern (See figure 1)

3.2.183 Sharkskin. Leather made from the top grain of the skins of sharks. It has various natural grain markings. The term should not be applied to leather made from other skins and embossed.

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3.2.197 Snuffed top grain (top grain snuffed). Portions of the grain surface lightly abraded with emery wheel or sandpaper, so as to lessen the effect of grain damage.

3.2.198 Sole leather butt bend. A double bend. (PR'S'T in figure 1)

3.2.199 Spew. Any constituents of leather that come to the surface in the form of a white crystalized deposit or a dark gummy deposit. (Also Spue)

3.2.200 Split. A term used to describe the portion of hide or skin, split into two or more thickness, other than the grain or hair side. Splits are usually named according to their sequence of production, such as "main," "second," or "slab" split (in case of upholstery leather); or for the use to which they are to be put, such as "flexible" (for innersoles), "glove", "waxed" (for cheap shoe-uppers); "bag and case" (finished with pyroxylin or pigment finish), "sole", etc.

3.2.201 Splitting. (1) Cutting leather into two or more layers. (See also Upholstery leather) (2) Cutting a hide into two sides preparatory to tanning.

3.2.202 Spready hide. A hide of large area in proportion to the weight.

3.2.203 Steerhide. (see Hide grades)

3.2.204 Strap bellies. Thin, light-weight, vegetable-tanned cattlehide bellies, rather flexible and with low load, processed for the strap trade.

3.2.205 Stuffing. The process of incorporating grease in leather by drumming the wet leather with warm molten grease and oils.

3.2.206 Sulfite cellulose. A by-product of paper mills, produced in sulfiting wood pulp, used as a tanning material more correctly named lignosulfonate since it does not contain cellulose.

3.2.207 Syntan. A synthetic organic tanning material.

3.2.208 Table dyeing. The application of dyestuff to leather with a brush, the leather being laid on a table. (also called Brush coloring)

3.2.209 Table run. Used to describe leather that has not been sorted and graded before selling by the tanner. (also Tannery run, or T.R.)

3.2.210 Tannery run. (see Table run)

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3.2.211 Tawing. The old English term applied to the process of making leather with alum as distinguished from tanning which was originally confined to vegetable tanning.

3.2.212 Top grain. The grain side of a hide from which nothing except the hair and associated epidermis have been removed by reduction to a specific thickness by shaving, splitting, or other means.

3.2.213 Trim. The removal of parts of a raw hide not suitable for making leather, such as portions from the outer edges of heads, shanks and bellies.

3.2.214 Upholstery leather. A general term for leather processed for use for furniture, airplanes, busses, and automobiles. The staple raw material in this country consists of spready cattlehides, split at least once and in many cases two or three times. The top or grain cuts go into the higher grades and the splits into the lower grades.

3.2.215 Valve leather. (see Hydraulic leather)

3.2.216 Vat dyeing. The application of dyestuffs to leather by the immersion of the leather in a revolving drum containing the dyestuff solution, as contrasted with "Table dyeing."

3.2.217 Veal. A large calfskin, almost as large as kip.

3.2.218 Vegetable tanning. The conversion of rawhides into leather by treating with water solution of tannin extracted from materials of vegetable origin.

3.2.219 Veiny. Appearance of leather characterized by many clearly visible blood vessels mostly on the flesh side, either closed or cut open by buffing or shaving operation.

3.2.220 Vellum. (see Parchment)

3.2.221 Wallaby. Leather from skin of the wallaby, a small or medium-sized species of kangaroo.

3.2.222 Walrus. Leather from the hides of walrus. Walrus hide is very thick and is used for buffing wheels. When split, it is used for bag leather. Split walrus and seal leather are practically indistinguishable, and "walrus leather" in the travelling-goods industry is used to refer to sealskin leather on which a simulated walrus grain is embossed.

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3.2.223 **Waltling shoulder.** The shoulder portion of vegetable-tanned cattlehide leather, tanned with a low load to give the flexibility required for a welt.

3.2.224 **White weight.** The weight of limed and unwashed stock.

3.2.225 **Willow.** (1) Willow grain - refers to boarded leather. (2) In the sporting goods industry, Willow tanned is used to indicate flexible, well-oiled, chrome-tanned cattlehide or horsehide used for gloves.

3.2.226 **Window.** In a chamois skin, a thin portion that transmits light when the skin is viewed against a window or light background.

3.2.227 **Woolskin.** Sheepskin with the wool on.

3.2.228 **Wrinkle.** A permanent crease or furrow on the grain surface of a hide or leather, incapable of removal by rolling or plating.

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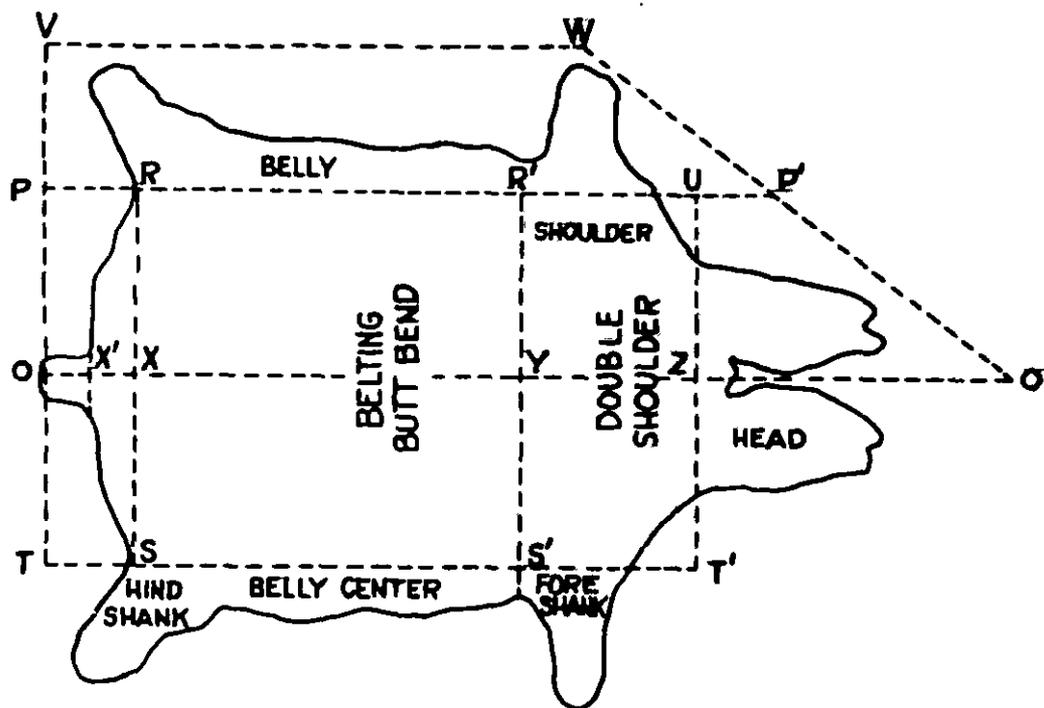


FIGURE 1-LOCATION OF HIDE PARTS

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3.3 Terms applicable to sampling.

3.3.1 Acceptance of a lot. The approval of a lot as conforming to contract or specification.

3.3.2 Composite sample. A portion of leather, which may be the scraps from the cuttings of physical test specimens, that has been taken from each of the sample units constituting the sample. The leather is composited as specified for the purpose of testing a lot for chemical properties.

3.3.3 Defective unit. A unit that fails to conform to one or more of the visual, dimensional, or tactile requirements.

3.3.4 Delivery. The leather or fabricated leather articles presented at any one time for inspection or test.

3.3.5 Examination. An element of investigation, without the use of special laboratory appliances or procedures, of supplies and services to determine conformance to those specified requirements which can be determined by such investigations. Examination is generally nondestructive and includes but is not limited to visual, auditory, olfactory, tactile, gustatory, and other investigations; simple manipulation; gaging; and measurement.

3.3.6 Inspection. The process of measuring, examining, testing, or otherwise comparing the unit of product.

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3.3.7 Lot. The term "lot" shall mean "inspection lot," i.e., a collection of units of product from which a sample is to be drawn and inspected to determine conformance with the acceptability criteria, and is to be accepted or rejected as a whole; it may differ from a collection of units designated as a lot for other purposes (e.g. production, shipment, etc.).

3.3.8 Lot size. The number of units of product in a lot.

3.3.9 Mean. Arithmetical average of a set of numbers.

3.3.10 Normal inspection. Inspection which is used when there is no statistically significant evidence that the quality of the product being submitted is better or poorer than the specified quality level.

3.3.11 Rejection number. A number R , such that if the number of defective units in the sample is equal to or greater than R , the lot shall be rejected.

3.3.12 Rejection of a lot. The disapproval of a lot as not conforming to contract or specification.

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3.3.13 Sample. A sample consists of one or more units of product drawn from a lot, the units of the sample being selected at random without regard to their quality.

3.3.13.1 Sample for examination. A specified number of units taken from a lot for the purpose of visual, dimensional, or tactile inspection.

3.3.13.2 Sample for test. A specified number of sample units taken from a lot for the purpose of testing the lot for all physical and chemical properties for which requirements are specified.

3.3.14 Sample size. The number of sample units in the sample.

3.3.15 Sample unit (for test purposes). The total quantity of material necessary to obtain one test result for each of the properties and characteristics specified in the material specification or procurement document. In testing of small package units, the sample unit may be a package unit randomly selected from the material representing the lot. In testing commodities in which the units are individually too small to provide sufficient material for evaluating all the properties specified in the material specification, the sample unit may be a sufficient amount of the material, taken as an aggregate, to provide the quantity of material required.

3.3.16 Specimen. That portion of a sample unit required for a single measurement of a given property or characteristic.

3.3.17 Testing. An element of inspection which generally denotes the determination by technical means of the properties or elements of supplies, or components thereof, and involves the application of established scientific principles and procedures.

3.3.18 Tightened inspection. Inspection under a sampling plan using the same quality level as for normal inspection, but requiring more stringent acceptance criteria.

3.3.19 Unit of product. A piece of leather in the form in which it is purchased, such as a single hide, side, skin or part thereof; or a single fabricated-leather article in the form in which it is purchased, such as a counter, a pair of shoes, a gasket, etc.

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SECTION 4
SAMPLING FOR INSPECTION

4.1 Scope

4.1.1 This section establishes sampling plans for inspection by attributes of leather and fabricated leather articles. The term "inspection" is used throughout the specification to signify the process of measuring, examining, testing, or otherwise comparing the unit of product. The term "sampling" is used to signify lot-by-lot acceptance sampling. The essential feature is that a product is grouped into lots, each of which is accepted or rejected in its entirety on the basis of the performance of a sample or samples taken from it.

4.2 General sampling procedure.

4.2.1 Formation of lots or batches. Prior to sampling, the product shall be assembled into identifiable lots, batches, or any such other manner as may be specified in the applicable procurement document. Each lot or batch (see 3.3.7) shall, as far as is practicable, consist of units of product of a single type, grade, class, size, and composition, manufactured under essentially the same conditions. Unless otherwise specified in the material specification, the number of units of product comprising a lot shall not exceed the equivalent of 25,000 square feet of leather.

4.2.1.1 The leather in any one lot should be produced:

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- (a) From units of product of similar size and type.
 - (b) From functionally equivalent tanning and finishing materials.
 - (c) From a single production method.
 - (d) From sequential production batches.

4.2.1.2 Presentation of lots or batches. The formation of the lots or batches, the lot or batch size, and the manner in which each lot or batch is to be presented and identified by the supplier shall be designated or approved by the responsible authority. As necessary, the supplier will provide adequate and suitable storage space for each lot or batch, equipment needed for proper identification and presentations, and personnel for all handling of product required for drawing of samples.

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4.3 Sampling for inspection. The sample required for inspection of the lot shall be taken in accordance with a random process, i.e., a process which gives each item in the lot the same chance of being included in the sample. To this end, the sample procedure should conform as far as practicable to the following rules:

Rule 1: Units shall be taken from locations scattered throughout the lot to insure that all the units for a sample will not be taken from the same portion of the lot, such as a single carton, layer, tier, etc.

Rule 2: Units shall be taken without regard to quality.

4.4. Sampling for examination of visual, dimensional, and tactile characteristics.

4.4.1 Sample size. Unless otherwise specified in the material specification, the number of units to be taken from a lot for the purpose of visual, dimensional, or tactile characteristic examination shall be in accordance with table I.

TABLE I.

Sampling for examination of visual, dimensional, and tactile characteristics of nonfabricated and fabricated leather articles.

Lot size	Sample size	Rejection No.	
		Normal	Tightened
8 or less	2	1	1
9 to 15	3	1	1
16 to 25	5	1	1
26 to 50	8	1	1
51 to 90	13	2	2
91 to 150	20	2	2
151 to 280	32	3	2
281 to 500	50	4	3
501 to 1200	80	6	4
1201 to 3200	125	8	6
3201 to 10000	200	11	9
10001 to 35000	315	15	13
35001 and over	500	22	19

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4.4.2 Normal inspection. Normal inspection shall be used at the start of inspection, unless otherwise directed by the procuring activity. When normal inspection is in effect, tightened inspection shall be instituted when 2 out of 5 consecutive lots or batches have been rejected on original inspection.

4.4.3 Tightened inspection. When tightened inspection is in effect, normal inspection shall be instituted when 5 consecutive lots or batches have been considered acceptable on original inspection.

4.4.4 Rejection of lot on the basis of examination of the sample. A lot should not be tested for physical and chemical characteristics if the number of defective units in the sample equals or exceeds the "Rejection Number." If the number of defective units in the sample is less than the "Rejection Number," the lot should be sampled for physical and chemical characteristics to determine its acceptability.

4.5 Sampling for physical and chemical tests. The sample required for testing the lot for physical and chemical characteristics shall be selected from the sample for visual, dimensional, or tactile characteristics. Each item of the sample, regardless of its visual, dimensional, or tactile characteristics, shall be given an equal chance of being included in the sample for testing.

4.5.1 Sampling for testing of nonfabricated leather.

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4.5.1.1 Sample size. Unless otherwise specified in the material specification, 15 units of product shall be selected at random from each lot for the purpose of testing. For lots consisting of less than 15 units of product, each unit shall be sampled for test.

4.5.1.2 Location of test area. An 8 by 8 inch area of the unit of product from which pieces shall be taken for use in preparing specimens for physical and chemical tests is shown in figure 2. The piece taken from the test area shall be of sufficient size and shape to furnish all the specimens required by the material specification for tests. The specimens shall be taken from the 8 by 8 inch test area as close to point "a" as is practicable. The piece shall be marked to indicate the side that is parallel to the backbone. The size and shape of the specimen for test cut from the piece shall be as specified in the applicable test method or in the material specification. Unless otherwise specified in the applicable test method or in the detail specification, the specimen for test shall be cut with the long dimension perpendicular to the backbone.

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4.5.1.2.1 Skins. The test area shall correspond to that for cattleshides as shown in figure 2, except that it shall begin at a point 1 inch from the backbone line and 3 inches from the root of the tail. Pieces for test shall be cut from only one side of the backbone of each skin.

4.5.1.2.2 Hides. The test area shall correspond to that shown in figure 2. Pieces for test shall be cut from only one side of the backbone of each hide.

4.5.1.2.3 Sides, crops, backs, and bends. The test area shall correspond to that shown in figure 2. Double bends and belting butts shall be sampled on only one side of the backbone.

4.5.1.2.4 Bellies. The test area shall correspond to area X of figure 2. The middle of the area shall be approximately midway between D and E. If physical tests are required, the area may be enlarged to 5 by 7 inches.

4.5.1.2.5 Double shoulders. The test area shall correspond to area Y of figure 2. The middle of the area shall be approximately midway between B and E. If physical tests are required, the area may be enlarged to 5 by 7 inches.

4.5.1.2.6 Pieces of bellies and shoulders for testing. When bellies and shoulders are purchased in separate units, pieces for use in chemical tests shall be taken from the areas marked "X" and "Y" respectively in figure 2 and composited as specified in method 6002. When physical tests are required, the "X" and "Y" areas shall be enlarged to 5 by 7 inches and specimens for testing taken from that area.

4.6 Sampling for testing of fabricated leather articles.

4.6.1 Sample size. Unless otherwise specified in the detail specification, the number of sample units to be taken from a lot for the purpose of physical and chemical tests shall be in accordance with table II.

TABLE II

Lot size (units)	Sample size
50 or less	2
51 to 500	3
501 to 3200	5
3201 and over	8

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4.6.1.1 Sampling for testing physical characteristics. Unless otherwise specified in the material specification, specimens for each physical property for which requirements are specified shall be taken as described in the applicable test method.

4.6.1.2 Sampling for testing chemical properties. Unless otherwise specified in the material specification or applicable test method, chemical properties for which requirements are specified shall be determined on the composite sample that has been prepared in accordance with test method 6002.

4.7 Acceptance of lot.

4.7.1 Unless otherwise specified in the applicable procurement document, a lot shall be considered as meeting the requirements of that document if the following conditions are fulfilled:

(1) After examination of the lot, the number of defective units is less than the applicable rejection number.

(2) The results of physical and chemical test determinations performed on the sample unit or composite sample satisfy the requirements set forth in the procurement document.

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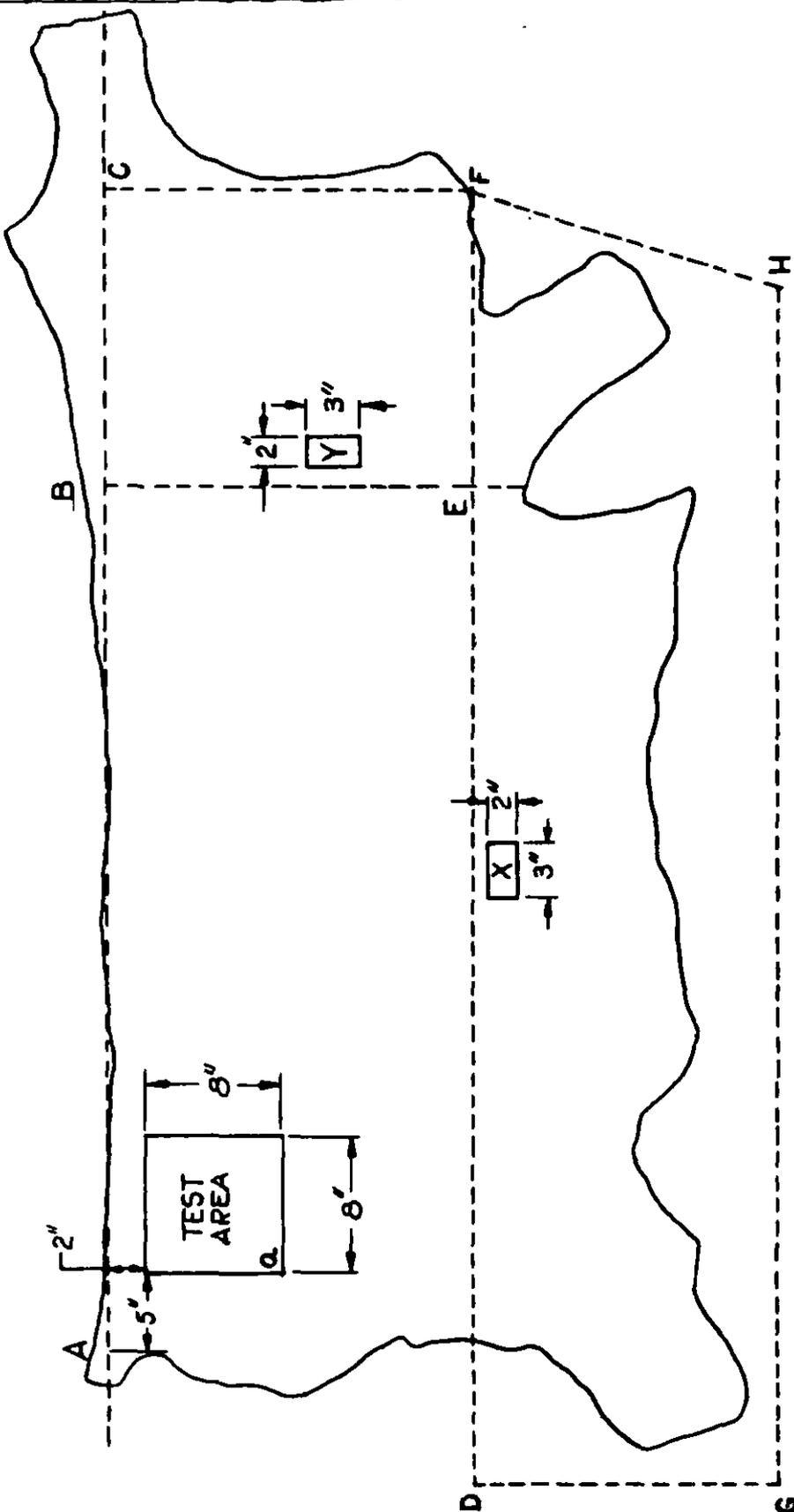


FIGURE 2 - LOCATION OF TEST AREA

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SECTION 5

ATMOSPHERIC CONDITIONS FOR TESTING

5.1 Humidity and temperature conditions for testing. Unless otherwise specified in the applicable test method, material specification, or procurement document, strength and mechanical tests of leather and leather products shall be performed under Standard Atmospheric Conditions and performed on specimens in moisture equilibrium under Standard Atmospheric Conditions. All other types of tests may be performed under ambient conditions or as specified.

5.1.1 Standard Atmospheric Conditions. Standard Atmospheric Conditions for leather and leather products testing are 50 ± 4 percent relative humidity at a temperature of $23^{\circ} \pm 2^{\circ}\text{C}$.

5.1.2 Moisture equilibrium. Moisture equilibrium is considered to have been reached when, after free exposure of the material to air in motion at Standard Atmospheric Conditions as defined above, the change in weight in successive weighings made at 1 hour intervals is no greater than 0.25 percent.

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SECTION 6

GENERAL NOTES

6.1 Content of methods.

6.1.1 Principal subdivisions. The methods are organized under the headings: Scope, Test specimen, Number of determinations, Apparatus, Procedure, Report.

6.1.2 Scope. The property to be measured or evaluated, the material to which the method is applicable, and limitations of the method are stated under "Scope."

6.1.3 Test specimen. The test specimen, its dimensions, the way in which it is to be taken, and its method of preparation is described.

6.1.4 Number of determinations. The number of test specimens required to obtain the results for a property and for a sample unit is found under "Number of determinations."

6.1.5 Apparatus, reagents, and methods cited. The apparatus, reagents, and other materials required to carry out the test are enumerated, as well as applicable methods required and cited to carry out the procedure.

6.1.6 Procedure. Detailed directions for carrying out the test and for calculating the results for a specimen are given.

6.1.7 Report. The precision and manner of expression of the test results is given.

6.2 Numerical requirements in methods.

6.2.1 Forms used. Numerical requirements are given in any of three forms illustrated by the following examples: "approximately 2 grams," "2 grams," and " 2.000 ± 0.002 grams."

6.2.1.1 "Approximately 2 grams". This form of expression implies that the numerical requirement is not critical and may vary within reason. The permissible variation is usually dictated by obvious practical considerations and the nearest readily obtained approximation to the weight or dimensions may be considered satisfactory.

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6.2.1.2 "2 grams". This form of expression implies that the numerical requirement is to be as close to "2 grams" as can be readily measured on the stated material with the usual, ordinary engineering tools.

6.2.1.3 "2.000 \pm .002 grams". This form of expression implies that the numerical requirement in question must be between 1.998 and 2.002 grams.

6.3 Conflict with referenced specification. When the requirements specified in this standard conflict with any requirement of a specification referenced herein, the requirements of this standard shall apply. Nature of conflict between this standard and the referenced specification shall be submitted, in duplicate, to Clothing and Organic Materials Division, U. S. Army Natick Laboratories, Natick, Mass., 01760.

6.4 Specification data.

6.4.1 Detail specifications for leather and leather articles should specify any desired options offered herein and should state which method shall be used for a particular test, such as method 4011 or 4021 for cracking, method 2141 or 2151 for stitch tear, or method 5021 for mildew resistance.

6.5 Raw materials of the tanning industry.

6.5.1 In describing various classes of leather, the name of the animal from which the skin was taken is generally used. The following are groups of animals from which skins are generally taken and dressed into leather:

Cattle
Sheep and lamb
Goat and kid
Equine
Buffalo
Pig and hog
Deer
Kangaroo and wallaby
Aquatic
Miscellaneous

6.6 Finished leather, uses.

6.6.1 Cattle group.

6.6.1.1 Steer, cow, and bull hides produce leather for the following uses:

Shoe soles, heels, counters, welting, innersoles
Boot and shoe uppers
Harness , saddles, skirting for saddles, horse collars
Traveling bags, suit cases, straps
Gloves and garments
Machinery belting
Upholstery for automobiles, furniture, airplanes, and decorative purposes
Fancy leather goods, handbags, belts and wallets
Bookbindings
Aprons
Buffing wheels
Aprons, carders, combers, and pickers on textile machinery
Hydraulic packings, washers and gaskets
Laces for belting, shoes, parachute slings and tags
Lithographic purposes
Footballs and sporting goods
Razor strops
Suspenders
Scabbards
Rawhides
Selfsealing gasoline tanks
Brief cases, envelopes (slide fastener)
Orthopedic purposes
Reinforcements for frame baskets

6.6.1.2 Kipskins from large calves or undersized or small breeds of cattle produce leather for the following uses:

Shoe uppers
Fancy leather goods and luggage
Gloves and garments
Shoe linings

6.6.1.3 Calfskins produce leather for the following uses:

Boot and shoe uppers
Fancy leather goods and handbags
Gloves and garments
Sweat bands for hats
Rawhide and parchment
Military helmets and gas masks
Fine shoe linings
Bookbindings
Grips for golf clubs
Handicrafts

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6.6.2 Sheep and lamb group -

6.6.2.1 Woolen skins, hair skins (cabrettas), etc., produce leather for the following uses:

Shoe linings and uppers, slippers and play shoes
Gloves
Bookbindings
Leather goods and handbags
Aprons
Chamois
Coats and other articles of clothing
Aviators' clothing
Sweat bands for hats
Meters
Parchment
Piano actions
Rollers on textile machinery
Military helmets
Millinery and caps
Sporting goods

6.6.3 Goat and kid group -

6.6.3.1 Skins from this group produce leather for the following uses:

Shoe uppers
Fancy leather goods and handbags
Gloves and garments
Aviators' clothing
Bookbindings
Upholstery

6.6.4 Equine group -

6.6.4.1 Horse, colt, ass, mule, and zebra hides produce leather for the following uses:

Shoes
Gloves and garments
Aviators' clothing
Sporting goods (baseball covers and mitts)
Puttees
Luggage
Razor strops
Belts

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6.6.5 Buffalo group -

6.6.5.1 Domesticated land and water buffalo (excluding American bison) produce leather for the following uses:

Shoe soles and uppers
Buffing wheels
Luggage
Handbags

6.6.6 Pig and hog group -

6.6.6.1 Pig, hog, boar, peccary, and carpincho (Brazilian rodent) skins produce leather for the following uses:

Fancy leather goods and luggage
Gloves
Innersoles, counters, etc.
Saddlery and harness
Shoe uppers

6.6.7 Deer group -

6.6.7.1 Fallow deer, reindeer, antelope, gazelle (dikdik), elk, and caribou skins produce leather for the following uses:

49 Shoe uppers
Gloves
Clothing
Fancy leather goods
Piano actions
Mukluks

6.6.8 Kangaroo and wallaby group

6.6.8.1 Skins from this group produce leather for shoe uppers.

6.6.9 Aquatic group -

6.6.9.1 Seal, sea-lion, and walrus skins produce leather for the following uses:

Luggage
Fancy leather goods
Buffing wheels

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6.6.9.2 Shark, whale, blackfish, dolphin, porpoise, etc., produce leather for the following uses:

Fancy leather goods
Luggage
Shoe uppers

6.6.9.3 Alligator skins produce leather for the following uses:

Shoes
Handbags
Luggage
Fancy leather goods

6.6.10 Miscellaneous group -

6.6.10.1 Ostrich skins produce leather for the following uses:

Fancy leather goods
Luggage

6.6.10.2 Camel, llama, alpaca, and yak skins are tanned in the producing countries usually for local consumption.

6.6.10.3 Lizard, snake, frog, and similar skins produce leather for the following uses:

Shoe uppers
Fancy leather goods

6.6.10.4 Elephant, hippopotamus, and rhinoceros skins are not used to any great extent because of small supply and the long time required to tan it.

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SECTION 7

CONVERSION EQUIVALENTS

7.1 Temperature conversion

7.1.1 Centigrade to Fahrenheit scales

$$\frac{9}{5} \text{ } ^\circ\text{C.} + 32 = \text{ } ^\circ\text{F.}$$

$^{\circ}\text{C.}$	$^{\circ}\text{F.}$								
-20	-4.0	9	48.2	38	100.4	67	152.6	96	204.8
-19	-2.2	10	50.	39	102.2	68	154.4	97	206.6
-18	-0.4	11	51.8	40	104.	69	156.2	98	208.4
-17	1.4	12	53.6	41	105.8	70	158.	99	210.2
-16	3.2	13	55.4	42	107.6	71	159.8	100	212.
-15	5.	14	57.2	43	109.4	72	161.6	101	213.8
-14	6.8	15	59.	44	111.2	73	163.4	102	215.6
-13	8.6	16	60.8	45	113.	74	165.2	103	217.4
-12	10.4	17	62.6	46	114.8	75	167.	104	219.2
-11	12.2	18	64.4	47	116.6	76	168.8	105	221.
-10	14.	19	66.2	48	118.4	77	170.6	106	222.8
- 9	15.8	20	68.	49	120.2	78	172.4	107	224.6
- 8	17.6	21	69.8	50	122.	79	174.2	108	226.4
- 7	19.4	22	71.6	51	123.8	80	176.	109	228.2
- 6	21.2	23	73.4	52	125.6	81	177.8	110	230.
- 5	23.	24	75.2	53	127.4	82	179.6	111	231.8
- 4	24.8	25	77.	54	129.2	83	181.4	112	233.6
- 3	26.6	26	78.8	55	131.	84	183.2	113	235.4
- 2	28.4	27	80.6	56	132.8	85	185.	114	237.2
- 1	30.2	28	82.4	57	134.6	86	186.8	115	239.
0	32.	29	84.2	58	136.4	87	188.6	116	240.8
1	33.8	30	86.	59	138.2	88	190.4	117	242.6
2	35.6	31	87.8	60	140.	89	192.2	118	244.4
3	37.4	32	89.6	61	141.8	90	194.	119	246.2
4	39.2	33	91.4	62	143.6	91	195.8	120	248.
5	41.	34	93.2	63	145.4	92	197.6	121	249.8
6	42.8	35	95.	64	147.2	93	199.4	122	251.6
7	44.6	36	96.8	65	149.	94	201.2	123	253.4
8	46.4	37	98.6	66	150.8	95	203.	124	255.2

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7.1.1 Centigrade to Fahrenheit scales (cont'd)

$$\frac{9}{5} \text{ } ^\circ\text{C.} + 32 = \text{ } ^\circ\text{F.}$$

$^\circ\text{C.}$	$^\circ\text{F.}$								
125	257.	136	276.8	147	296.6	158	316.4	169	336.2
126	258.8	137	278.6	148	298.4	159	318.2	170	338.
127	260.6	138	280.4	149	300.2	160	320.	171	339.8
128	262.4	139	282.2	150	302.	161	321.8	172	341.6
129	264.2	140	284.	151	303.8	162	323.6	173	343.4
130	266.	141	285.8	152	305.6	163	325.4	174	345.2
131	267.8	142	287.6	153	307.4	164	327.2	175	347.
132	269.6	143	289.4	154	309.2	165	329.	176	348.8
133	271.4	144	291.2	155	311.	166	330.8	177	350.6
134	273.2	145	293.	156	312.8	167	332.6	178	352.4
135	275.	146	294.8	157	314.6	168	334.4	179	354.2
								180	356.

7.1.2 Fahrenheit to Centigrade scales

$$(\text{ } ^\circ\text{F.} - 32) \times \frac{5}{9} = \text{ } ^\circ\text{C.}$$

$^\circ\text{F.}$	$^\circ\text{C.}$								
0	-17.78	15	-9.44	30	-1.11	45	7.22	60	15.56
1	-17.22	16	-8.89	31	-0.56	46	7.78	61	16.11
2	-16.67	17	-8.33	32	0.	47	8.33	62	16.67
3	-16.11	18	-7.78	33	0.56	48	8.89	63	17.22
4	-15.56	19	-7.22	34	1.11	49	9.44	64	17.78
5	-15.	20	-6.67	35	1.67	50	10.	65	18.33
6	-14.44	21	-6.11	36	2.22	51	10.56	66	18.89
7	-13.89	22	-5.56	37	2.78	52	11.11	67	19.44
8	-13.33	23	-5.	38	3.33	53	11.67	68	20.
9	-12.78	24	-4.44	39	3.89	54	12.22	69	20.56
10	-12.22	25	-3.89	40	4.44	55	12.78	70	21.11
11	-11.67	26	-3.33	41	5.	56	13.33	71	21.67
12	-11.11	27	-2.78	42	5.56	57	13.89	72	22.22
13	-10.56	28	-2.22	43	6.11	58	14.44	73	22.78
14	-10.	29	-1.67	44	6.67	59	15.	74	23.33

7.1.2 Farenheit to Centigrade scales (cont'd)

$$(^{\circ}\text{F.} - 32) \times \frac{5}{9} = ^{\circ}\text{C.}$$

$^{\circ}\text{F.}$	$^{\circ}\text{C.}$								
75	23.89	111	43.89	147	63.89	183	83.89	219	103.89
76	24.44	112	44.44	148	64.44	184	84.44	220	104.44
77	25.	113	45.	149	65.	185	85.	221	105.
78	25.56	114	45.56	150	65.56	186	85.56	222	105.56
79	26.11	115	46.11	151	66.11	187	86.11	223	106.11
80	26.67	116	46.67	152	66.67	188	86.67	224	106.67
81	27.22	117	47.22	153	67.22	189	87.22	225	107.22
82	27.78	118	47.78	154	67.78	190	87.78	226	107.78
83	28.33	119	48.33	155	68.33	191	88.33	227	108.33
84	28.89	120	48.89	156	68.89	192	88.89	228	108.89
85	29.44	121	49.44	157	69.44	193	89.44	229	109.44
86	30.	122	50.	158	70.	194	90.	230	110.
87	30.56	123	50.56	159	70.56	195	90.56	231	110.56
88	31.11	124	51.11	160	71.11	196	91.11	232	111.11
89	31.67	125	51.67	161	71.67	197	91.67	233	111.67
90	32.22	126	52.22	162	72.22	198	92.22	234	112.22
91	32.78	127	52.78	163	72.78	199	92.78	235	112.78
92	33.33	128	53.33	164	73.33	200	93.33	236	113.33
93	33.89	129	53.89	165	73.89	201	93.89	237	113.89
94	34.44	130	54.44	166	74.44	202	94.44	238	114.44
95	35.	131	55.	167	75.	203	95.	239	115.
96	35.56	132	55.56	168	75.56	204	95.56	240	115.56
97	36.11	133	56.11	169	76.11	205	96.11	241	116.11
98	36.67	134	56.67	170	76.67	206	96.67	242	116.67
99	37.22	135	57.22	171	77.22	207	97.22	243	117.22
100	37.78	136	57.78	172	77.78	208	97.78	244	117.78
101	38.33	137	58.33	173	78.33	209	98.33	245	118.33
102	38.89	138	58.89	174	78.89	210	98.89	246	118.89
103	39.44	139	59.44	175	79.44	211	99.44	247	119.44
104	40.	140	60.	176	80.	212	100.	248	120.
105	40.56	141	60.56	177	80.56	213	100.56	249	120.56
106	41.11	142	61.11	178	81.11	214	101.11	250	121.11
107	41.67	143	61.67	179	81.67	215	101.67		
108	42.22	144	62.22	180	82.22	216	102.22		
109	42.78	145	62.78	181	82.78	217	102.78		
110	43.33	146	63.33	182	83.33	218	103.33		

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7.2 Linear equivalents.-

Meter	Centimeter	Millimeter	Inch	Foot	Yard
1.000	100.00	1000.000	39.37	3.281	1.094
0.010	1.00	10.000	0.394	0.033	0.011
0.0254	2.54	25.400	1.000	0.083	0.028
0.305	30.48	304.801	12.000	1.000	0.333
0.914	91.44	914.400	36.000	3.000	1.000

7.3 Mass equivalents.-

Grams	Ounces	Pounds
1.000	0.035	0.002
28.350	1.000	0.063
453.590	16.000	1.000

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7-4 Relative humidity, percent (based on Centigrade scale at pressure of 29.24 inches).

Air Temp. (t)	Depression of Wet-bulb thermometer (t-t ₁)																				
	t = dry-bulb (air) temperature										t ₁ = wet-bulb temperature										
	.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	10.5
10	94	88	82	77	71	66	60	55	50	44	39	34	29	24	20	15	10	6	-	-	-
11	94	89	83	78	72	67	61	56	51	46	41	36	32	27	22	18	13	9	5	-	-
12	94	89	84	79	74	69	64	59	54	49	45	41	36	32	28	23	19	15	8	-	-
13	95	89	85	79	75	70	65	60	56	51	47	42	38	34	30	26	22	18	11	7	-
14	95	90	85	80	75	71	66	61	57	53	48	44	40	36	32	27	24	20	14	10	6
15	95	90	85	81	76	71	67	63	58	54	50	46	42	38	34	30	26	23	16	13	9
16	95	90	86	81	76	72	68	64	60	55	51	47	43	40	36	32	28	25	19	15	12
17	95	90	86	82	77	73	69	65	61	57	53	49	45	41	38	34	30	27	21	18	14
18	95	91	86	82	77	73	69	65	61	57	53	49	45	41	38	34	30	27	23	20	17
19	95	91	87	82	78	74	70	65	62	58	54	50	46	43	39	36	32	29	26	22	19
20	96	91	87	83	78	74	70	66	63	59	55	51	48	44	41	37	34	31	28	24	21
21	96	91	87	83	79	75	71	67	64	60	56	53	49	46	42	39	36	32	29	26	23
22	96	92	87	83	80	76	72	68	64	61	57	54	50	47	44	40	37	34	31	28	25
23	96	92	88	84	80	76	72	69	65	62	58	55	52	48	45	42	39	36	33	30	27
24	96	92	88	84	80	77	73	69	66	62	59	56	53	49	46	43	40	37	34	31	29
25	96	92	88	84	81	77	74	70	67	63	60	57	54	50	47	44	41	39	36	33	30
26	96	92	88	85	81	78	74	71	67	64	61	58	54	51	49	46	43	40	37	34	32
27	96	92	89	85	82	78	75	71	68	65	62	59	56	52	50	47	44	41	38	36	33
28	96	93	89	85	82	78	75	72	69	65	62	59	56	53	51	48	45	42	40	37	34
29	96	93	89	86	82	79	76	72	69	66	63	60	57	54	52	49	46	43	41	38	36
30	96	93	89	86	83	79	76	73	70	67	64	61	58	55	52	50	47	44	42	39	37
31	96	93	90	86	83	80	77	73	70	67	64	61	58	56	53	51	48	45	43	40	38
32	96	93	90	86	83	80	77	74	71	68	65	62	59	57	54	51	49	46	44	41	39
33	97	93	90	87	83	80	77	74	71	68	65	63	60	57	55	52	50	47	45	42	40
34	97	93	90	87	84	81	78	75	72	69	66	63	61	58	56	53	51	48	46	43	41

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7.5 Tolerance conversion tables

<u>Temperature</u>			<u>Length</u>			<u>Weight</u>		
<u>°C</u>	<u>T</u>	<u>°F</u>	<u>Centimeters</u>	<u>L</u>	<u>Inches</u>	<u>Grams</u>	<u>W</u>	<u>Ounces</u>
0.56	1	1.8	2.54	1	0.40	28.35	1	0.035
1.12	2	3.6	5.08	2	0.80	56.70	2	0.070
1.68	3	5.4	7.62	3	1.20	85.05	3	0.105
2.24	4	7.2	10.16	4	1.60	113.40	4	0.140
2.80	5	9.0	12.70	5	2.00	141.75	5	0.175
3.36	6	10.8	15.24	6	2.40	170.10	6	0.210
3.92	7	12.6	17.78	7	2.80	198.45	7	0.245
4.48	8	14.4	20.32	8	3.20	226.80	8	0.280
5.04	9	16.2	22.86	9	3.60	255.15	9	0.315
5.60	10	18.0	25.40	10	4.00	283.50	10	0.350

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THICKNESS, UNIT

1. SCOPE

1.1 This method is intended for determining the thickness of units of leather such as hides, skins, crops, backs, bends, belting butts, belting butt ends, sole leather bends, single shoulders, double shoulders, bellies, side upper leather, cut soles, counters, glove leather, chamois, etc.

2. TEST SPECIMEN

2.1 The test specimen shall consist of a unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified, five measurements shall be made at points equally spaced over the unit.

4. APPARATUS

4.1 Gage, spring-type, graduated in 0.1 millimeter (0.25 ounce) or 0.5 ounce (1/128 inch), having a flat presser foot 10.2 ± 0.6 millimeters in diameter and a flat anvil 10.2 ± 0.6 millimeters in diameter. The spring shall exert a force of 454 ± 20 grams on the foot when the gage reads 2 ounces and 907 ± 20 grams when the gage reads 12 ounces.

57 4.2 Gage, standard wedge-type, having one leg graduated in a 0.5 iron (1 iron = 1/48 inch) preferably constructed of stainless steel, in which the legs enclose an angle of 4 degrees.

4.3 Gage, micrometer caliper, with ball attachment.

5. PROCEDURE

5.1 Non-fabricated leather.

5.1.1 Leather other than sole leather.

5.1.1.1 Unless otherwise specified in the material specification, measurements shall be made with the gage described in 4.1.

5.1.1.2 The portion of the specimen to be measured shall be placed between the anvil and the presser foot of the gage in such a manner that the portion of the specimen to be measured is in contact with the whole area of the anvil. With the specimen held in this position, the thumb

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lever of the gage shall be compressed so that the gage reads 15 ounces. The lever shall then be released allowing the gage presser foot to snap onto the leather. The thickness shall be measured to the nearest 0.1 millimeter or to the nearest 0.25 ounce. The thickness shall be measured at equally spaced places along and at least 15 centimeters from the backbone. The initial measurement shall be at a point 13 centimeters from the root of the tail, and the final point of measurement shall extend no farther than 13 centimeters into the neck area.

5.1.1.3 If the dimensions are such that the gage will not reach from the edge to the point at which the thickness is desired, the specimen shall be folded upon itself with the flesh inside. The thickness of the folded specimen shall be measured and one half of this measurement shall be taken as the thickness.

5.1.2 Sole leathers.

5.1.2.1 Unless otherwise specified in the material specification, measurements shall be made with the gage described in 4.2.

5.1.2.2 The cut edge of the specimen shall be inserted between the legs of the gage so that the plane of the leather is perpendicular to the plane of the gage and bisects the angle formed by the legs. The gage shall be pushed over the edge of the leather to make firm contact with the leather so that the gage will stay in position when inverted, but not cause any visible deformation of the specimen. The thickness shall be read to the nearest 0.5 iron at the point where the specimen contacts the legs of the gage. The thickness shall be measured as follows:

5.1.2.2.1 Bellies. The specimen shall be gaged at two locations 15 centimeters to either side of the point opposite the center of the main width of the belly.

5.1.2.2.2 Shoulders (double). The specimen shall be gaged at two places on the cut edge opposite the neck and 15 centimeters on each side of the backbone.

5.1.2.2.3 Shoulders (single). The specimen shall be gaged at one place on the cut edge opposite the neck and 15 centimeters from the backbone line.

5.1.2.2.4 Bend butts. The specimen shall be gaged at two places on the cut edge at the shoulder end and 15 centimeters on each side of the backbone.

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5.1.2.2.5 Bends. The specimen shall be gaged at three places along the backbone starting 30 centimeters from the root of the tail and at 15 centimeter intervals from that point toward the shoulder end.

5.1.2.2.6 Backbones, sides and crops. The specimen shall be gaged in no less than five places along the backbone, starting 30 centimeters from the root of the tail and at 15 centimeter intervals from that point toward the shoulder end.

5.2 Fabricated leather articles.

5.2.1 General.

5.2.1.1 Unless otherwise specified in the material specification, measurements shall be made with the gage described in 4.1, or if the specimen is rounded, the micrometer gage described in 4.3 shall be used.

5.2.1.2 Unless otherwise specified in the material specification, the specimen shall be measured as described in 5.1.1.2, except that measurements shall be made at 3 places spaced equidistant along the longitudinal center line of the specimen.

5.2.2 Cut soles.

5.2.2.1 Unless otherwise specified in the material specification, measurements shall be made with the gage described in 4.2.

5.2.2.2 The smooth edge of the specimen shall be placed between the legs of the gage so that the plane of the leather is perpendicular to the plane of the edge and bisects the angle formed by the legs of the gage. The gage shall be pushed over the edge of the specimen far enough to make firm contact with both surfaces of the leather, but not far enough to cause any visible distortion of the specimen. At the point where the specimen contacts the legs of the gage, the thickness shall be read from the scale to the nearest 0.5 iron.

5.2.2.3 Unless otherwise specified in the material specification, measurements shall be made on the toe, inside ball, and outside ball.

6. REPORT

6.1 The thickness of the specimen shall be the average of the results obtained from all measurements made on the specimen. Individual results utilized to obtain the average shall be reported.

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6.2 The thickness of the specimen as determined in 5.1.1 and 5.2.1 shall be reported to the nearest 0.1 millimeter or 0.25 ounce.

6.3 The thickness of the specimen as determined in 5.1.2 and 5.2.2 shall be reported to the nearest 0.5 iron.

NOTE: The gages described in 4.1 and 4.2 may be purchased from The Woburn Machine Co., 201 Main Street, Woburn, Mass. 01801.

The micrometer gage and ball attachment described in 4.3 may be purchased from the L.S. Starrett Co., Athol, Mass. 01331.

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METHOD 1021
January 15, 1969

THICKNESS, SPECIMEN

1. SCOPE

1.1 This method is intended for determining the thickness of specimens of leather used for various physical tests.

2. TEST SPECIMEN

2.1 The specimen shall be of the size and shape required in the applicable physical test method.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the detail physical test method, each specimen shall be measured.

4. APPARATUS

4.1 The apparatus shall consist of a dial micrometer having a flat anvil not less than 10 millimeters in diameter and a flat presser foot 10 ± 0.5 millimeters in diameter which exerts a force of 393 ± 10 grams on the specimen, the force being applied by means of a weight. The surfaces of the anvil and presser foot shall be parallel to within 0.00025 millimeters. The dial shall be graduated to read in 0.0025 centimeter units.

5. PROCEDURE

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5.1 General. The specimen shall be placed on the anvil of the micrometer and the presser foot lowered gently (not dropped) until it contacts the surface of the leather. The thickness of the specimen shall be read from the dial and the value recorded. Unless otherwise specified in the detail physical test method, 3 measurements, equally spaced over the specimen, shall be made.

5.2 Tensile strength specimen. The procedure shall be as described in 5.1, except that the specimen shall be measured at 3 places along the longitudinal center line of the specimen, at the center and approximately 2.54 centimeters on each side of the center.

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6. RESULTS

6.1 Unless otherwise stated in the detail physical test method, the thickness of the specimen shall be the average of the measurements made.

6.2 The thickness of the specimen shall be reported to the nearest 0.0025 centimeter.

NOTES: Apparatus which meets the requirements of this specification may be purchased from:

Frank E. Randall Co.
248 Ash Street
Waltham, Massachusetts 02154

Custom Scientific Instrument Co.
541-43 Devon Street
Arlington, New Jersey 07032

Testing Machines Inc.
431 W. 42nd Street
New York, New York 10036

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METHOD 1111
January 15, 1969

AREA, UNIT, WEIGHT METHOD

1. SCOPE

1.1 This method is intended for determining the area of units of leather. This method is more accurate than method 1121 and should be used in case of dispute as to the method for determining area.

2. TEST SPECIMEN

2.1 The specimen shall consist of a unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be measured.

4. APPARATUS

4.1 Paper of uniform thickness at least as large in area as the specimen to be measured.

4.2 An instrument for cutting the paper.

4.3 Balance accurate to 0.01 gram and weights when required.

4.4 Rule or tape graduated in millimeters.

5. PROCEDURE

5.1 The piece of paper shall be placed flat on a smooth surface. The specimen shall be placed flat on the paper and the area of the specimen traced on the paper. The paper shall be cut to the shape of the specimen, weighed, and the weight recorded to the nearest 0.1 gram as W_1 . A rectangle consisting of more than half of the total area of the weighed paper shall be cut from the weighed paper. The paper rectangle shall be weighed and the weight recorded as W_2 . The dimensions of the paper rectangle shall be measured to the nearest millimeter by means of the graduated rule or tape, the area calculated by multiplying the width by the length, and the value recorded to the nearest square centimeter as A.

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January 15, 1969

5.2 Calculation. The area of the specimen shall be calculated as follows:

$$\text{Area, square centimeters} = \frac{W_1}{W_2} \times A$$

Where:

W_1 = weight of the specimen-shaped paper, grams.

W_2 = weight of the paper rectangle, grams.

A = area of the paper rectangle, square centimeters.

6. REPORT

6.1 The area of the specimen shall be reported to the nearest square centimeter.

METHOD 1121
January 15, 1969

AREA, UNIT, TEMPLATE METHOD

1. SCOPE

1.1 This method is intended for determining the area of large units of leather.

2. TEST SPECIMEN

2.1 The specimen shall consist of a unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be measured.

4. APPARATUS

4.1 A transparent, flexible template graduated in square centimeters. The template shall be large enough to cover the specimen completely.

5. PROCEDURE

5.1 The specimen shall be placed flat on a smooth surface. The template shall be placed smoothly over the specimen. The area shall be determined by counting the number of square centimeters covering the surface of the specimen. Parts of the squares of the template not completely covered by the specimen shall be estimated and the value recorded to the nearest 500 square centimeters.

6. RESULTS

6.1 The area of the specimen shall be reported to the nearest 500 square centimeters.

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METHOD 1211
January 15, 1969

WIDTH AND LENGTH, UNIT

1. SCOPE

1.1 This method is intended for determining the width or length of physical test specimens of regularly shaped units and pieces of all types of leather.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be the prepared test specimen, unit, or piece of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one test specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A steel scale or tape graduated to 0.5 millimeter or finer.

5. PROCEDURE

5.1 Narrow units (100 millimeters or less), pieces, or specimens: The leather shall be placed on a flat surface and flattened without pulling or stretching. The graduated steel scale or tape shall be placed over the specimen without causing any distortion in the width or length of the specimen. Unless otherwise specified in the material specification, three measurements equally spaced along the width or length of the specimen shall be made to the nearest 0.5 millimeter.

5.2 Wide units (over 100 millimeters), pieces, or specimens: The width or length shall be determined as described in 5.1, except that measurements shall be made accurate to the nearest 2.0 millimeters.

6. REPORT

6.1 The width or length of the specimen, unit or piece shall be the average of the values obtained from all of the measurements made on the specimen, unit, or piece, and shall be reported to the nearest 0.5 or 2.0 millimeters as applicable.

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METHOD 1231
January 15, 1969

APPARENT DENSITY OF LEATHER

1. SCOPE

1.1 This method is intended for determining the apparent density of specimens of leather from their area and thickness. The method is unsuitable for very soft leathers, such as chamois, whose thickness cannot be accurately measured by the method cited.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen for testing shall be a die cut disc of leather 68 to 76 millimeters in diameter.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 Round die 68 to 76 millimeters in diameter.

4.1.2 Mallet.

4.1.3 Ruler or steel tape graduated in millimeters.

4.1.4 Analytical balance accurate to 0.001 gram.

4.1.5 Thickness gage as described in method 1021.

4.2 Method cited.

4.2.1 Method 1021. Thickness, Specimen.

5. PROCEDURE

5.1 The specimen shall be weighed to the nearest 0.001 gram.

5.2 The specimen shall be placed on the anvil of the thickness gage and the presser foot lowered gently until it contacts the surface of the leather. The thickness of the leather shall be read from the dial and the value recorded to the nearest 0.25 centimeter. Measurements shall be taken at 4 quadrants of the specimen disc at points equidistant from the rim and center. The average of these measurements shall be recorded as the thickness of the specimen.

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5.3 The diameter of the specimen disc shall be measured by placing a ruler or steel tape across its widest girth and recording the length indicated on the scale to the nearest 0.1 centimeter. This procedure shall be repeated 4 times at radii approximately 45° to each other. The average of these measurements shall be recorded as the diameter of the specimen disc.

5.4 Calculation of results.

5.4.1 The area of the specimen shall be calculated by the following equation:

$$A = 1/4 \pi D^2$$

A = area of test specimen in square centimeters,

D = average diameter in centimeters,

5.4.2 The apparent density shall be calculated as follows:

$$\begin{array}{l} \text{Apparent density,} \\ \text{grams per cubic} \\ \text{centimeter} \end{array} = \frac{\text{Weight of specimen in grams}}{A \times \text{Average thickness in centimeters}}$$

6. REPORT

6.1 The apparent density of the specimen shall be reported to the nearest .01 gram/cubic centimeter.

METHOD 2021
January 15, 1969

**BREAKING FORCE OR TENSILE STRENGTH, AND
ELONGATION: DUMBBELL STRIP METHOD**

1. SCOPE

1.1 This method is intended for determining the breaking force or tensile strength, and elongation of leather.

2. TEST SPECIMEN

2.1 The specimen shall be dumbbell-shaped and shall be cut from the sample unit of leather by means of a die having the dimensions shown in figure 2021. The specimen shall be cut with the long dimension perpendicular to the backbone. Mechanical leather shall be cut parallel to the backbone.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHODS CITED

4.1 Apparatus.

4.1.1 A testing machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches with the long dimension perpendicular to the direction of application of force. The surfaces of the jaws shall be flat and knurled to prevent slipping of the specimen during test.

4.1.1.2 The machine shall be power driven.

4.1.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

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4.1.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load, and shall be uniform at all times.

4.1.1.6 The machine shall be of such capacity that the maximum load required to break the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.1.1.7 If the machine is equipped with a dynamometer head of the compensating type for convenience in eliminating calculations, the head shall have means for making adjustments for variations in thickness of specimens.

4.1.2 Metal die of the shape and dimensions shown in figure 2021 for stamping or cutting the specimen. The die shall be sharp and free from nicks in order to eliminate ragged edges on the specimen.

4.2 Methods cited.

4.2.1 Method 1021. Thickness, Specimen.

4.2.2 Method 1211. Width and Length, Unit.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed on material conditioned as specified in Section 5.

5.1.1 The distance between the edges of the clamps at the start of the test shall be $4 \pm 1/8$ inches. The specimen shall be placed in the clamps of the testing machine with the long dimension parallel to the direction of the application of force and adjusted symmetrically in order that the tension will be distributed uniformly over the cross section. If the tension is greater on one side of the specimen than on the other, the maximum strength of the specimen will not be developed. The force shall be applied and after rupture of the specimen, the breaking force shall be noted from the dial, scale or automatic recorder and the value recorded to the nearest pound as the breaking force of the specimen.

5.2 When tensile strength is specified in the material specification, the thickness of the specimen shall be determined by method 1021, and the width by method 1211.

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5.3 When elongation is specified, it shall be determined on the same specimen and at the same time as the breaking force determination. The load at which elongation is measured shall be as specified in the material specification. If an automatic recorder is not available, the distance between clamps at the beginning of the test shall be measured to the nearest 0.1 inch and recorded as D_1 . When the force required for measuring elongation is reached, the distance between clamps shall be remeasured to the nearest 0.1 inch and recorded as D_2 .

5.4 Calculation of results.

5.4.1 When specified in the material specification, elongation of the specimen shall be calculated as follows:

$$\text{Elongation, percent} = \frac{D_2 - D_1}{D_1} \times 100$$

Where: D_1 = initial distance between clamps, inches.
 D_2 = distance between clamps at the moment the required force is reached, inches.

5.4.2 When specified in the material specification, the tensile strength of the specimen shall be calculated as follows:

$$\text{Tensile strength, pounds per square inch} = \frac{\text{Breaking force (pounds)}}{\text{Thickness (inches) x Width (inches)}}$$

6. REPORT

6.1 The breaking force of the specimen shall be reported to the nearest 1 pound.

6.2 The elongation of the specimen shall be reported to the nearest 1 percent.

6.3 The tensile strength of the specimen shall be reported to the nearest 10 pounds per square inch.

6.4 The load at which the elongation was measured shall be reported.

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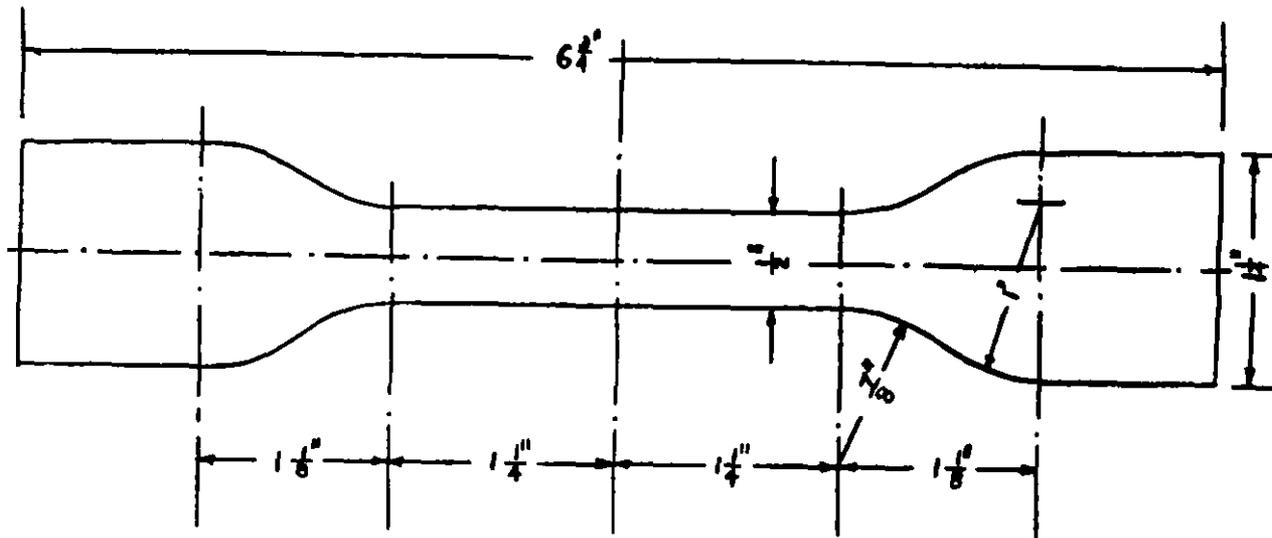


Figure 2021

Shape of specimen for testing for
tensile strength and elongation

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BREAKING FORCE AND ELONGATION, GRAB METHOD

1. SCOPE

1.1 This method is for determining the breaking strength and elongation of light, soft leather; boarded, sueded, or embossed leather; narrow strap, welt, lace and round belt leathers; and other leathers that cannot be tested accurately by method 2021.

2. TEST SPECIMEN

2.1 Non-fabricated leathers.

2.1.1 The specimen shall be a rectangle of leather 3 inches wide by at least 6 inches long. The specimen shall be cut with the long dimension parallel to the backbone.

2.2 Fabricated leathers.

2.2.1 The specimen shall be a rectangle of leather 3 inches wide by at least 6 inches long when possible. When narrower articles are to be tested, such as strap, welting, lace and round belting, the specimen shall be a full width portion at least 6 inches long.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 A testing machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of one jaw of each clamp shall be 1 inch by 1 inch and the face of the other jaw of each clamp shall be 1 inch by not less than 1-1/2 inches with the long dimension perpendicular to the direction of application of force. The surface of the jaws shall be flat and knurled to prevent slipping of the specimen during test.

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4.1.1.2 The machine shall be power driven.

4.1.1.3 The applied tension shall be accurate to + 2 percent up to and including a force of 50 pounds and + 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.1.5 The rate of travel of the power actuated clamp shall be 10 + 2 inches per minute under no load and shall be uniform at all times.

4.1.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to tear the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.1.1.7 If the machine is equipped with a dynamometer head of the compensating type for convenience in eliminating calculations, the head shall have means for making adjustments for variations in thickness.

4.2 Method cited.

4.2.1 Method 1211. Width and Length, Unit.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed on material conditioned as specified in Section 5.

5.2 The specimen shall be placed in the clamps of the testing machine under slight tension with the long dimension parallel to the direction of application of the load. The distance between clamps at the start of the test shall be 3 + 1/8 inches. The specimen shall be adjusted in the clamps uniformly and symmetrically so that it hangs flatly between the jaws. The clamps shall then be tightened and the machine shall be operated until rupture of the specimen. After rupture of the specimen, the breaking force shall be noted from the dial, scale or automatic recorder and the value recorded as the breaking force of the specimen.

5.3 When elongation of the specimen is to be determined, it shall be determined on the same specimen and at the same time as the breaking force determination. The load at which the elongation is to be determined shall be as specified in the material specification. If an automatic recorder

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is not available, the distance between clamps at the beginning of the test shall be measured to the nearest 0.1 inch and recorded as D_1 . When the force required for measuring elongation is reached, the distance between clamps shall be remeasured to the nearest 0.1 inch and recorded as D_2 .

5.4 Calculation of results. The elongation of the specimen shall be calculated as follows:

$$\text{Elongation, percent} = \frac{D_2 - D_1}{D_1} \times 100$$

Where: D_1 = initial distance between clamps, inches.

D_2 = distance between clamps at the moment the required force is reached, inches.

6. REPORT

6.1 The breaking force of the specimen shall be reported to the nearest 1 pound.

6.2 The elongation of the specimen shall be reported to the nearest 1 percent.

6.3 The load at which elongation was measured shall be reported.

6.4 When specimens less than 3 inches in width are tested, the actual width of the specimens as determined by method 1211 shall be reported.

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METHOD 2051
January 15, 1969

BURSTING STRENGTH, PLUNGER METHOD

1. SCOPE

1.1 This method is intended for determining the bursting strength of leather by measuring the load required to force a hemispherically tipped plunger through the specimen. It may be used to test a large variety of leather and leather products. It is particularly applicable to light and medium weight leathers such as shoe uppers and garment leathers.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be a disc of leather 2 inches in diameter. When a group of determinations is desired, large pieces may be used and the clamping area may be overlapped in any direction so that tests can be made with a minimum distance of 1 inch between the bursts.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

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4.1.1 A testing machine wherein the specimen is strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1.1 The machine shall be power driven.

4.1.1.2 The applied tension shall be accurate to + 2 percent up to and including a force of 50 pounds and + 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.1.3 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.1.4 The rate of travel of the power actuated clamp shall be 4 ± 1 inches per minute under no load and shall be uniform at all times.

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4.1.1.5 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to break the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.1.1.6 If the machine is equipped with a dynamometer head of the compensating type for convenience in eliminating calculations, the head shall have means for making adjustments for variations in thickness of specimens.

4.1.2 Burst testing device, as shown in figure 2051D and parts required to adapt the device to the testing machine described in 4.1.1. The assembled device shall be mounted on the testing machine and shall operate in such a manner as to thrust a plunger against the center of the flesh side of a leather specimen.

4.1.2.1 Sample holder plates as shown in figures 2051B and 2051C. The upper and lower gripping surfaces shall be of unpolished (matte) metal which may have fine tool marks or a surface of such nature that will assist in holding the test specimen firmly. The plates shall be mounted in the tester as shown in figure 2051D.

4.1.2.2 Plunger. Unless otherwise specified in the material specification, a plunger having the size and dimensions as shown in figure 2051A shall be mounted in the testing device.

4.2 Method cited.

4.2.1 Method 1021. Thickness, Specimen.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under conditions and on material conditioned as specified in Section 5.

5.2 If the material specification requires calculation of the results in pounds per unit thickness, the thickness of the specimen shall be determined as nearly as possible by the procedure described in method 1021 at the point where the burst measurement will be made. When a larger piece or a strip of leather is specified, the center or point at which the test is specified shall be located and properly marked and the thickness determined at that point before a burst measurement is made on the piece of leather.

5.3 The assembled burst tester as shown in figure 2051D shall be attached to the testing machine. The specimen shall be placed in the clamp with the flesh side facing the plunger. The space between the gripping surfaces

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shall be adjusted in accordance with the thickness of the specimen and the specimen gripped securely by pulling the plunger handle downward. The initial reading of the dial, scale, or automatic recorder shall be observed. The machine shall be set in operation and shall be stopped immediately at burst. The maximum load registered by the machine shall be recorded.

6. REPORT

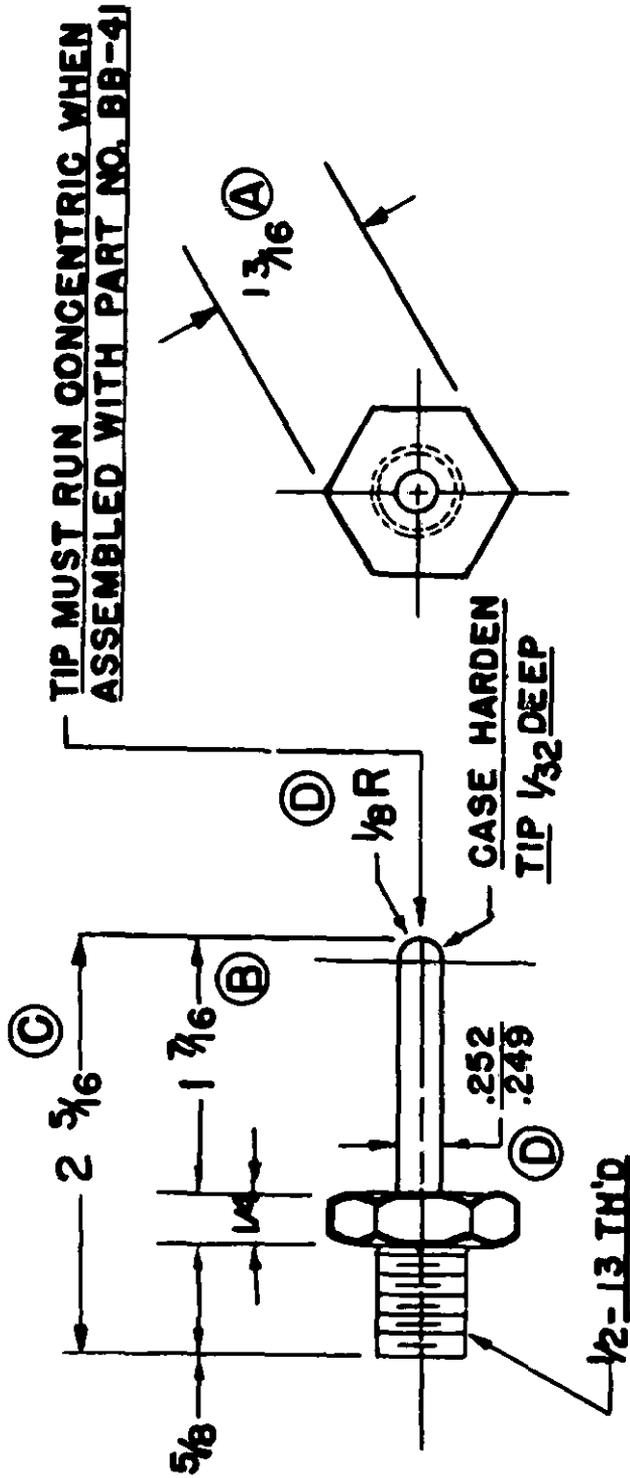
6.1 The bursting load of the specimen shall be reported to the nearest 1 pound.

6.2 When specified, the bursting load per unit thickness shall be calculated to the nearest pound per inch of thickness by dividing the load by the thickness.

NOTE: Apparatus of type described in this method may be purchased from Scott Testers, Inc., P.O. Box 963, Providence, Rhode Island 02901.

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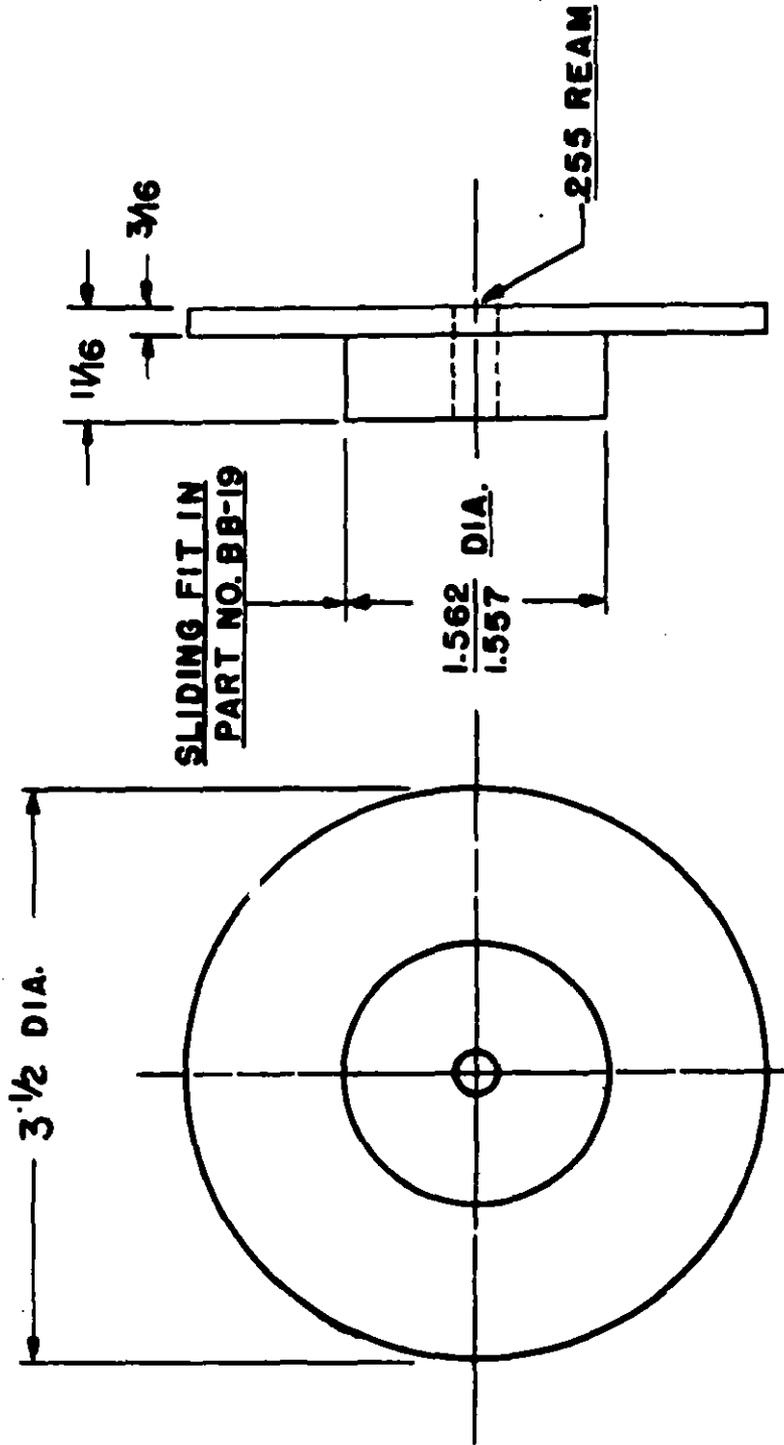
LEATHER INDENTER POINT

PART NO. 40

PART REQUIRED TO MODIFY MODEL-W SCOTT BURST TESTER

FIGURE 2051 A

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SAMPLE HOLDER PLATE
(LOWER)

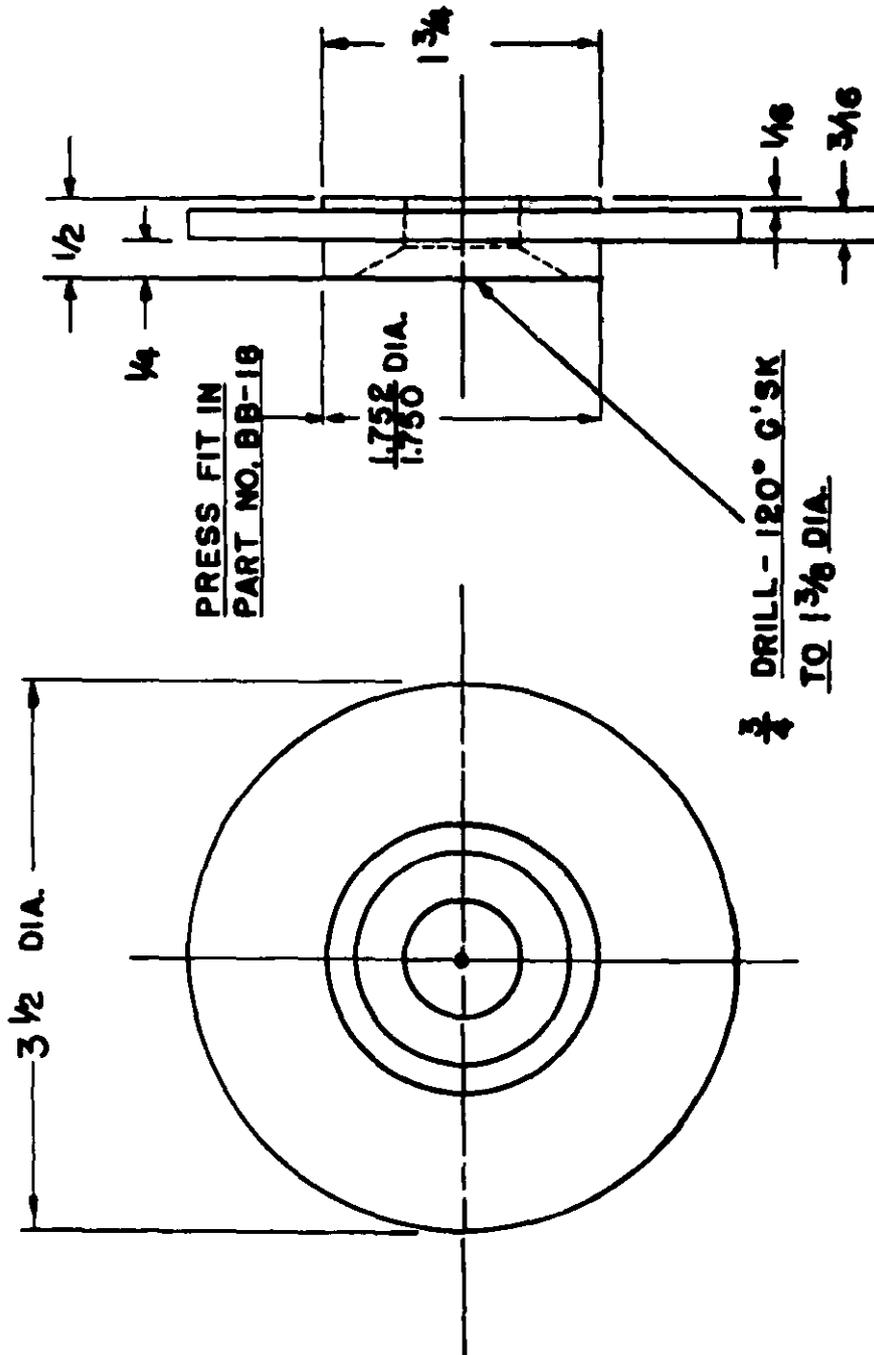
PART NO. BB-4.3

PART REQUIRED TO MODIFY MODEL-W SCOTT BURST TESTER

FIGURE 2051 B

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SAMPLE HOLDER PLATE
(UPPER)

PART NO. - 42
PART REQUIRED TO MODIFY MODEL-W SCOTT BURST TESTER

FIGURE 2051 C

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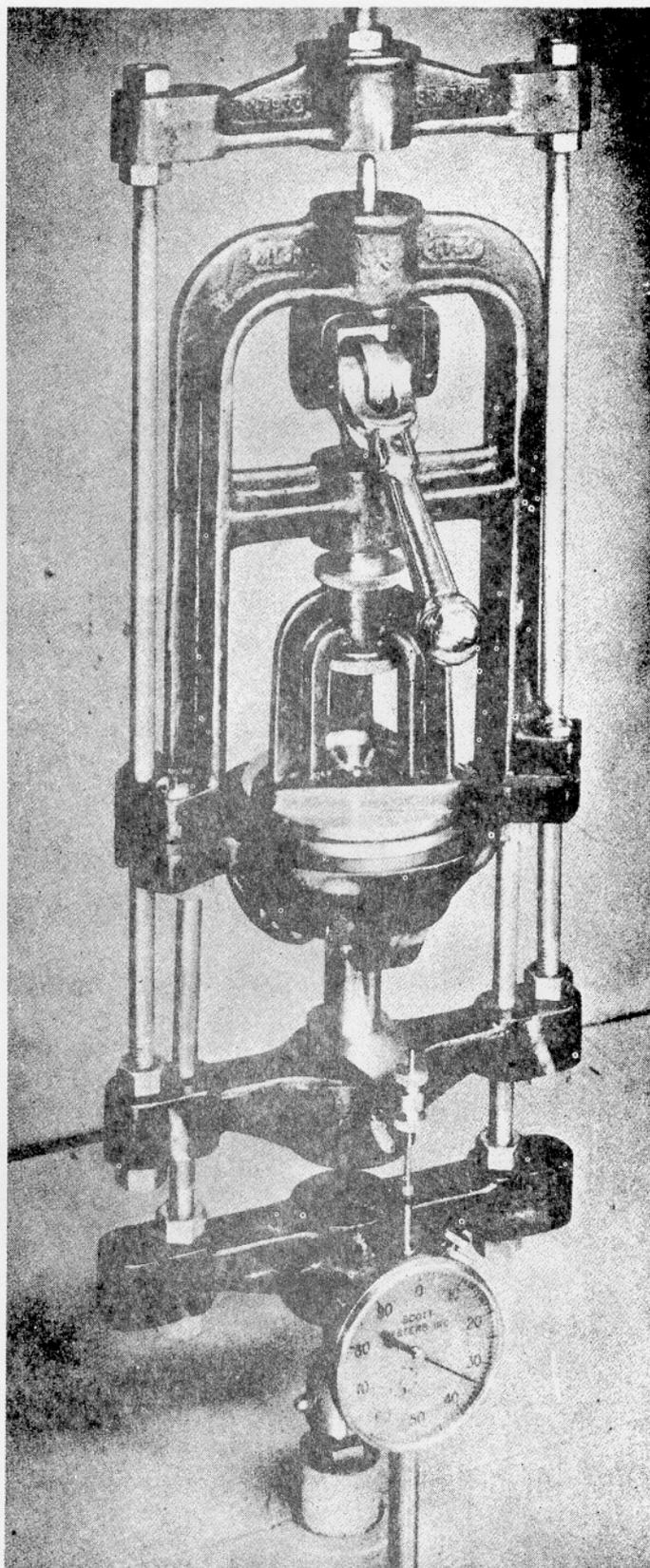


FIGURE 2051

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METHOD 2111

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TEARING STRENGTH, TONGUE

1. SCOPE

1.1 This method is intended for determining the tearing strength of leather by measuring the force required to tear a specimen cut perpendicular to the surface.

2. TEST SPECIMEN

2.1 The specimen for test shall be a die cut rectangle of leather 5 inches in length and 1 inch in width.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A testing machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches with the long dimension perpendicular to the direction of application of force. The surface of the jaws shall be flat and knurled to prevent slipping of the specimen during test.

4.1.2 The machine shall be power driven.

4.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load and shall be uniform at all times.

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4.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to tear the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.2 Metal die for cutting the specimen.

4.3 Punch capable of making a 3/16 inch diameter hole in the specimen.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.2 A hole 3/16 inch in diameter shall be punched in the center of one end of the specimen approximately one inch from the end. The specimen shall be divided into two strips by cutting it lengthwise along the center line from the hole to the end farthest from the hole.

5.3 One strip of the specimen shall be clamped in each of the clamps of the testing machine, approximately 2 inches from the punched hole of the strip. Force shall be applied to the specimen. At the moment of the initial tear of the specimen, the force required to start the tear shall be noted from the dial, scale, or automatic recorder and the value recorded as the tearing strength of the specimen.

6. REPORT

6.1 The tearing strength of the specimen shall be reported to the nearest pound.

METHOD 2121
January 15, 1969

TEARING STRENGTH, SPLIT

1. SCOPE

1.1 This method is intended for determining the tearing strength of leather by measuring the force required to tear a specimen having a split parallel to the surface to form a grain and split tongue. This method is not recommended for use in procurement.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 5 inches in length and 1 inch in width die cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A test machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

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4.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches, with the long dimension perpendicular to the direction of application of force. The surface of the jaws shall be flat and knurled to prevent slipping of the specimen during test.

4.1.2 The machine shall be power driven.

4.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load and shall be uniform at all times.

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January 15, 1969

4.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to tear the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.2 A knife or other instrument for splitting the specimen.

4.3 A die having suitable dimensions for cutting the specimen described in 2.1.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 The specimen shall be split completely across the width and parallel to the surface so as to separate the grain and flesh sides into two strips of approximately equal thickness and about 3-1/2 inches in length.

5.3 One strip of the specimen shall be clamped in one clamp of the testing machine and the other strip shall be clamped in the opposite clamp of the machine approximately 2 inches from the base of the strips. Force shall be applied to the specimen. At the moment of initial tear of the specimen, the force required to start the tear shall be noted from the dial, scale, or automatic recorder and the value recorded as the tearing strength of the specimen.

6. REPORT

6.1 The tearing strength of the specimen shall be reported to the nearest 1 pound.

METHOD 2131
January 15, 1969

TEARING STRENGTH, BUCKLE

1. SCOPE

1.1 This method is intended for determining the tearing strength of leather when tested in contact with a buckle.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 8 inches in length and 1 inch in width cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A test machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the movable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches with the long dimension perpendicular to the direction of application of force. The surfaces of the jaws shall be flat and knurled to prevent slipping of the specimen during test.

4.1.2 The machine shall be power driven.

4.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load and shall be uniform at all times.

METHOD 2131
January 15, 1969

4.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to tear the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.2 A punch for making a 3/16 inch diameter hole in the specimen.

4.3 A 1-1/4 inch harness buckle with tongue 0.175 + 0.050 inch in diameter attached to a strap. The strap shall be at least 5 inches in length and equipped with a keeper.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 A hole 3/16 inch in diameter shall be punched in the center line of the specimen approximately 3 inches from one end. The tongue of the buckle shall be fitted into the hole in the specimen and the 3 inch end passed under the keeper on the buckle strap. The other end of the specimen shall be clamped in one of the clamps of the testing machine so that the hole is approximately 3 inches from the clamp. The free end of the strap attached to the buckle shall be clamped in the other clamp of the testing machine. Force shall be applied to the machine and at the moment of initial tear of the specimen, the force required to start the tear shall be noted from the dial, scale, or automatic recorder and the value recorded as the tearing strength of the specimen.

6. REPORT

6.1 The tearing strength of the specimen shall be reported to the nearest 1 pound.

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METHOD 2141
January 15, 1969

TEARING STRENGTH, STITCH, SINGLE HOLE

1. SCOPE

1.1 This method is intended for determining the stitch-tearing strength of leather with a tear originating from one hole. It is particularly applicable to heavy leather.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 2 inches in length by 1 inch in width die cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus

4.1.1 A testing machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

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4.1.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches with the long dimension perpendicular to the direction of application of force. The surfaces of the jaws shall be flat and knurled to prevent slipping of the specimen during test. The jaws of one of the clamps of the machine shall be covered with a material suitable for protecting the gripping surfaces from damage by contact with a metal yoke. (See 4.1.2).

4.1.1.2 The machine shall be power driven.

4.1.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

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4.1.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

4.1.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load and shall be uniform at all times.

4.1.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to break the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.1.1.7 If the machine is equipped with a dynamometer head of the compensating type for convenience in eliminating calculations, the head shall have means for making adjustments for variations in thickness of specimens.

4.1.2 A metal yoke having the shape and dimensions shown in figure 2141.

4.1.3 A steel rod 0.095 ± 0.001 inch in diameter and at least $5/8$ inch in length.

4.1.4 A punch for making a $1/8$ inch diameter hole in the specimen.

4.1.5 A die having suitable dimensions for cutting the specimen specified in 2.1.

4.2 Method cited.

4.2.1 Method 1021 - Thickness, Specimen.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 When specified in the material specification, the thickness of the specimen shall be determined as described in method 1021. One measurement shall be made in the adjacent area near the end of the specimen where the hole will be made. A hole $1/8$ inch in diameter shall be punched in the center line of the measured end of the specimen, the center of the hole being $5/32$ inch from the end. The end of the specimen containing the hole shall be placed between the two arms of the yoke and a $5/8$ inch length steel rod passed through the holes of the yoke and the specimen. The end of the yoke shall be clamped in the covered clamp of the testing machine. The other end of the specimen shall be clamped in the other clamp of the testing machine. Force shall be applied to the specimen at the rate of 10 ± 2

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inches per minute. At the moment of initial tear of the specimen, the force required to start the tear shall be noted from the dial, scale, or automatic recorder and the value recorded as the stitch tearing strength (single hole) of the specimen.

6. REPORT

6.1 The tearing strength of the specimen shall be reported to the nearest 1 pound.

6.2 When specified in the material specification, the thickness of the specimen shall be reported with the tearing strength of the specimen.

6.3 When the tearing strength per unit thickness is specified in the material specification, the value obtained by dividing the tearing strength by the thickness of the specimen shall be reported.

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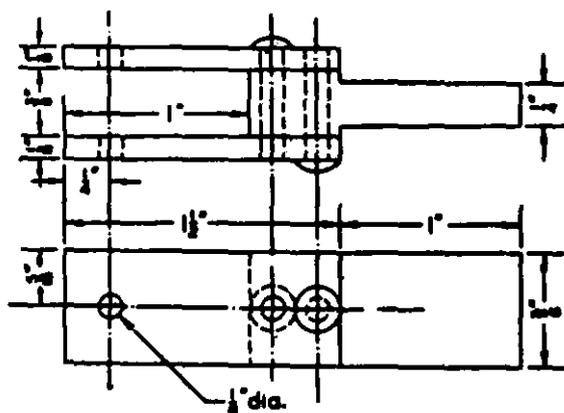


FIGURE 2141 - Metal yoke for testing stitch tearing strength, single hole method.

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METHOD 2151
January 15, 1969

TEARING STRENGTH, STITCH, DOUBLE HOLE

1. SCOPE

1.1 This method is intended for determining the stitch tearing strength of leather using a double-hole tear. It is particularly applicable to light-weight leathers.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 2 inches in length and 1 inch in width cut from the sample unit of leather. The specimen shall be cut with the long dimension perpendicular to the backbone.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus

4.1.1 A testing machine wherein the specimen is held between two clamps and strained by a uniform movement of a pulling clamp. The machine shall meet the following requirements:

4.1.1.1 The design of each clamp shall be such that one jaw is an integral part of the rigid frame of the clamp and the other jaw is on a part hinged or swiveled to the moveable member of the clamp. The face of the jaws of each clamp shall be 1 inch by 1-1/2 or more inches with the long dimension perpendicular to the direction of application of force. The surfaces of the jaws shall be flat and knurled to prevent slipping of the specimen during test. The jaws of one of the clamps of the machine shall be covered with a material suitable for protecting the gripping surfaces from damage by contact with a metal wire. (See 4.1.2.)

4.1.1.2 The machine shall be power driven.

4.1.1.3 The applied tension shall be accurate to ± 2 percent up to and including a force of 50 pounds and ± 1 percent over 50 pounds, and shall be indicated by a dial, scale, or automatic recorder.

4.1.1.4 The load indicator shall record or indicate the point of maximum load after rupture of the specimen.

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4.1.1.5 The rate of travel of the power actuated clamp shall be 10 ± 2 inches per minute under no load and shall be uniform at all times.

4.1.1.6 The machine, when used for a given specimen, shall be of such capacity that the maximum load required to break the specimen is not greater than 85 percent nor less than 15 percent of the rated capacity.

4.1.1.7 If the machine is equipped with a dynamometer head of the compensating type for convenience in eliminating calculations, the head shall have means for making adjustments for variations in thickness of specimens.

4.1.2 A piece of steel wire 0.041 ± 0.001 inch in diameter and not less than 4 inches in length. The wire shall conform to the requirements for type I size 1, large paper clips as contained in Federal Specification FF-C-436.

4.1.3 A punch (as shown in figures 2151A, 2151B, and 2151C) or other suitable instrument for making two holes $5/64$ inch in diameter in the specimen.

4.2 Method cited.

4.2.1 Method 1021 - Thickness, Specimen.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 Two holes each $5/64$ inch in diameter shall be punched in one end of the specimen; the centers of the holes shall be $1/4$ inch from the end, $1/4$ inch apart, and located equidistant from the center line (lengthwise) of the specimen.

5.3 When specified in the material specification, the thickness of the specimen shall be determined as described in method 1021 except that one measurement shall be made in the specified hole location prior to making the holes. The steel wire 0.041 ± 0.001 inch in diameter shall be made into a U-shape and passed through the two holes so that both ends project from the flesh side of the specimen and the bent wire is in contact with the grain side of the specimen. The ends of the wire shall be clamped in the covered clamp of the testing machine. The free end of the specimen shall be clamped in the other clamp of the testing machine. Force shall be applied to the

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specimen at the rate of 10 ± 2 inches per minute. At the moment of initial tear of the specimen, the force required to start the tear shall be noted from the dial, scale or automatic recorder and the value recorded as the stitch tearing strength (double hole) of the specimen.

6. REPORT

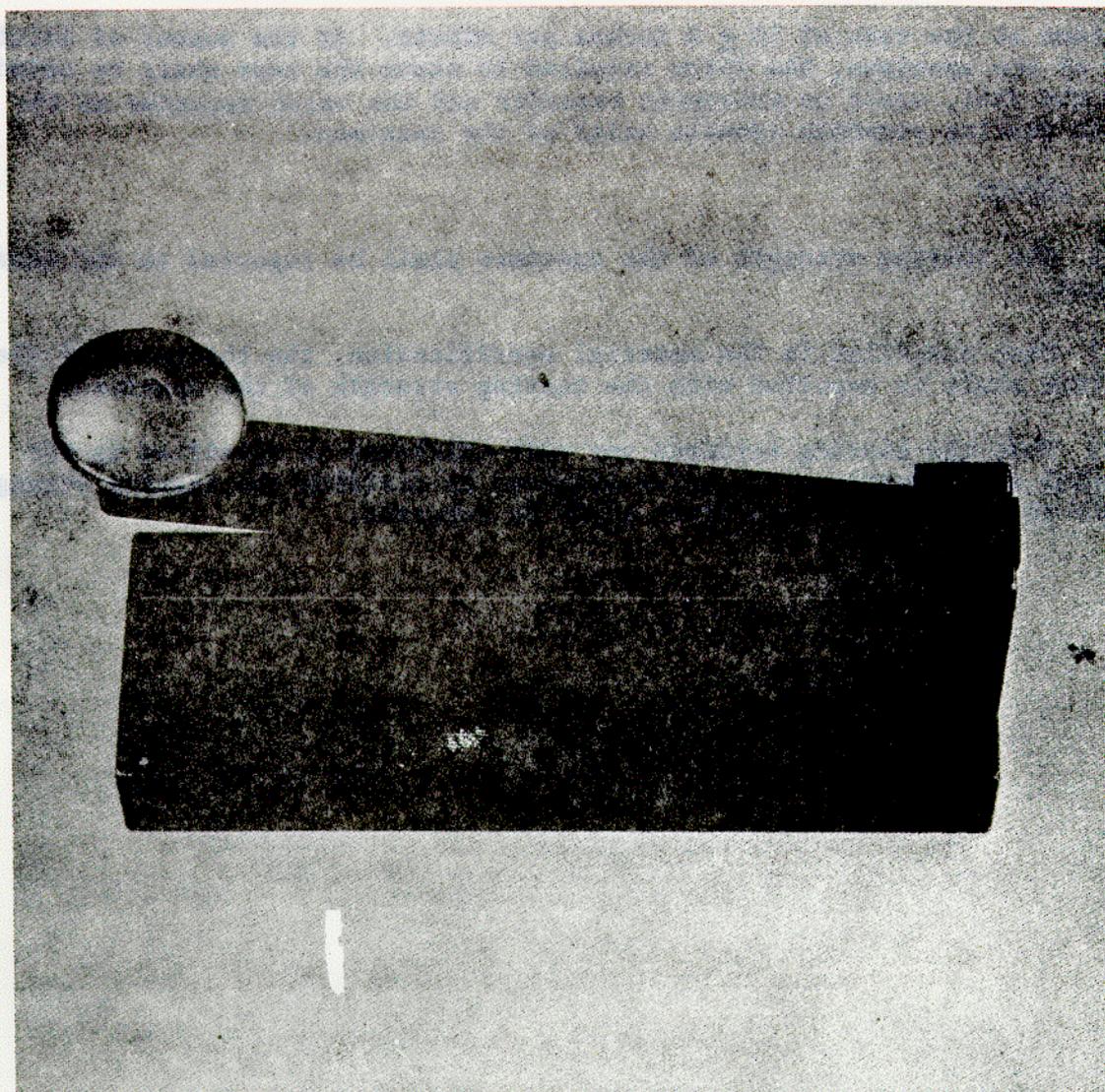
6.1 The tearing strength of the specimen shall be reported to the nearest 1 pound.

6.2 When specified in the material specification, the thickness of the specimen shall be reported with the tearing strength of the specimen.

6.3 When the tearing strength per unit thickness is specified in the material specification, the value obtained by dividing the tearing strength by the thickness of the specimen shall be reported.

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001

FIGURE 2151 A
PUNCH FOR TEAR TEST

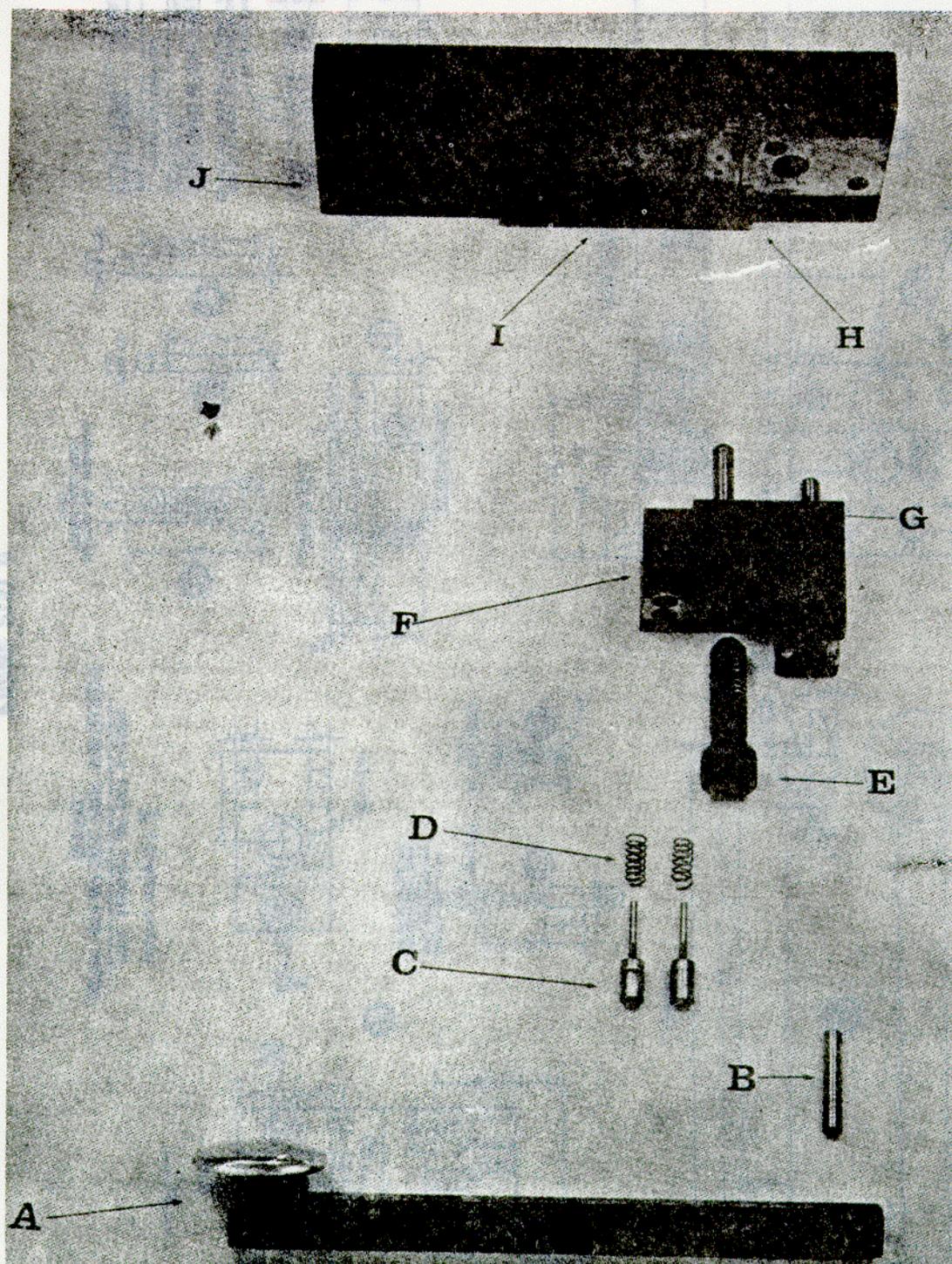
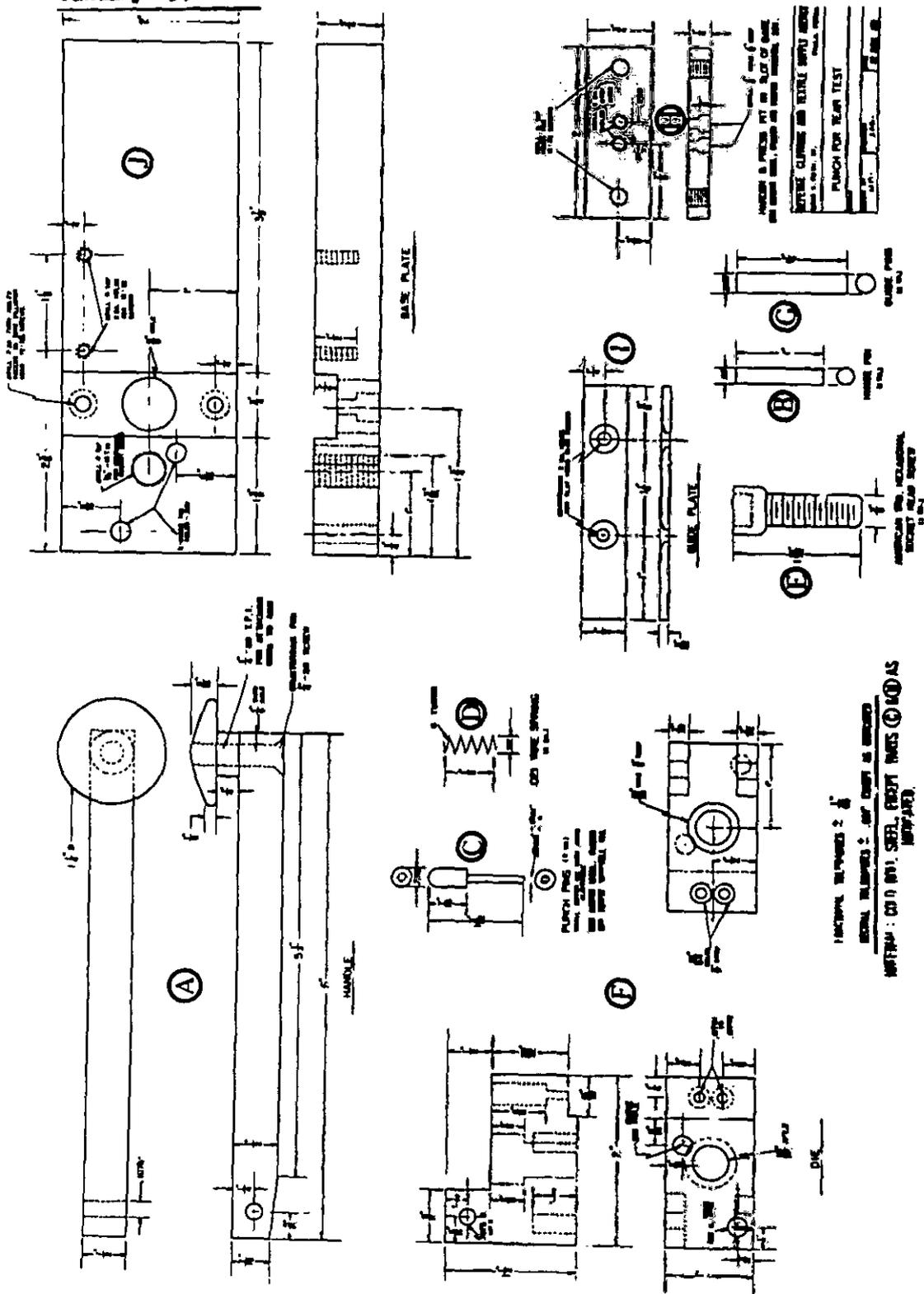


FIGURE 2151 B

PUNCH FOR TEAR TEST

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METHOD 2161
January 15, 1969

TEARING STRENGTH, SLOT TEAR

1. SCOPE

1.1 This method is intended for determining the tearing strength of leather at a slot cut perpendicular to its surface. It is designed to measure the load required to tear light leathers such as shoe uppers, gloves, and upholstery.

2. TEST SPECIMEN

2.1 The test specimen shall be a die cut rectangle of leather cut from the test unit with the die described in 4.1.2. Unless otherwise specified in the material specification, the specimen shall be cut with the long dimension parallel to the backbone.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHODS CITED

4.1 Apparatus

4.1.1 Two specimen holders of the same type having the shape and dimensions shown in figure 2161A. Use of the new style or old style holders is permitted.

4.1.2 A die conforming to the shape and dimensions shown in figure 2161B, which cuts the specimen and simultaneously cuts the required slot in the specimen.

4.1.3 Testing machine described in method 2021.

4.2 Methods cited.

4.2.1 Method 1021 - Thickness, Specimen.

4.2.2 Method 2021 - Breaking Force or Tensile Strength, and Elongation; Dumbbell Strip Method.

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5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 When specified in the material specification, the thickness of the specimen shall be determined as described in method 1021.

5.3 The specimen holders shall be clamped between the jaws of the testing machine and shall be adjusted so that the hooked ends of the holders are almost touching each other. The slot in the specimen shall be slipped over the protruding end of the holders so that the long dimension is at a right angle to the applied force and secure on the holders. Force shall be applied to the specimen as described in method 2021 until the specimen is torn apart. The force required to tear the specimen shall be noted from the dial, scale, or by means of an automatic recorder and the value recorded as the tearing strength of the specimen.

6. REPORT

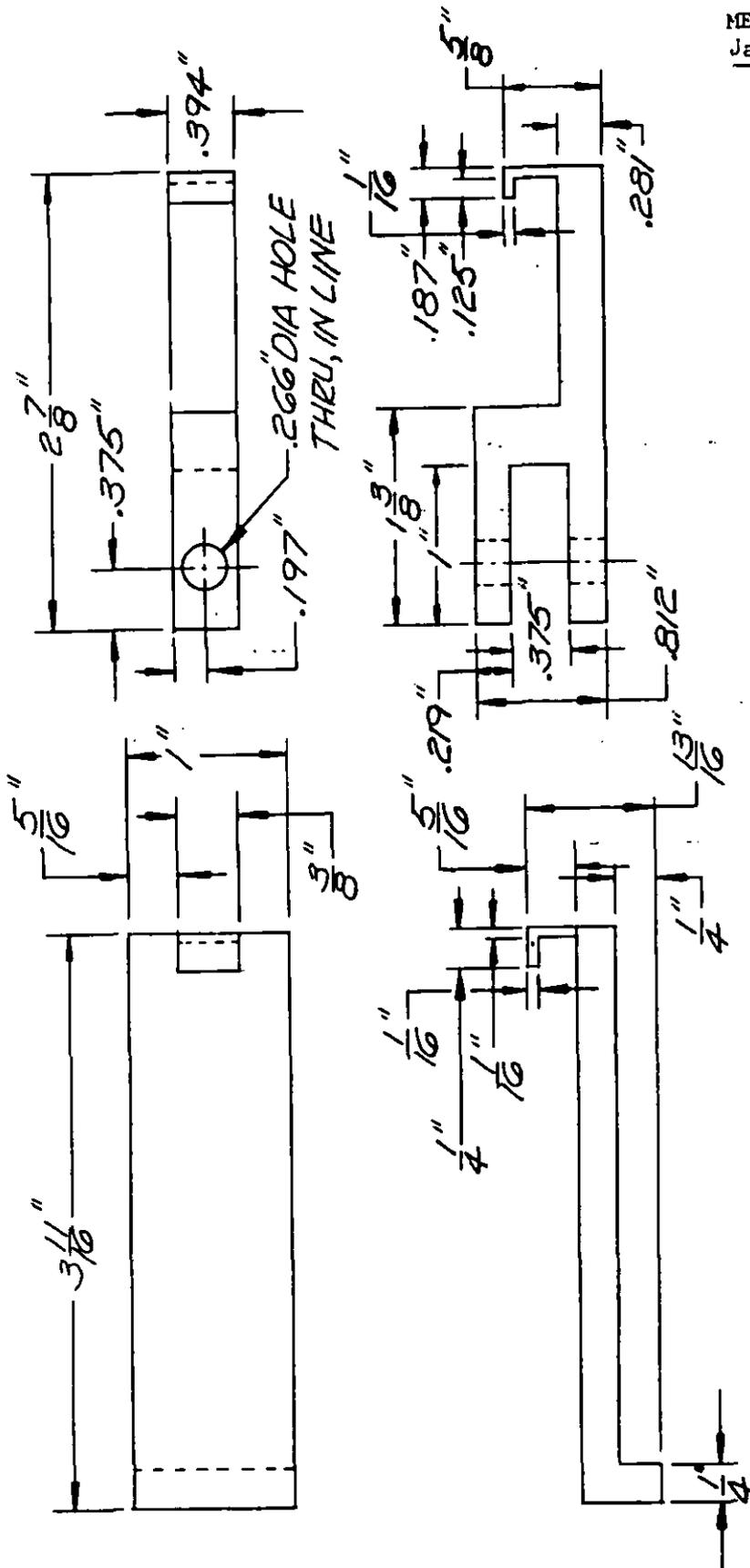
6.1 The tearing strength of the sample shall be reported to the nearest 1 pound.

6.2 When the tearing load per unit thickness is specified, the value obtained by dividing the tearing load by the thickness of the specimen shall be reported.

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OLD STYLE

NEW STYLE
2 REQD

FIGURE 2161A. NEW AND OLD STYLE SLOT TEAR CLAMPS

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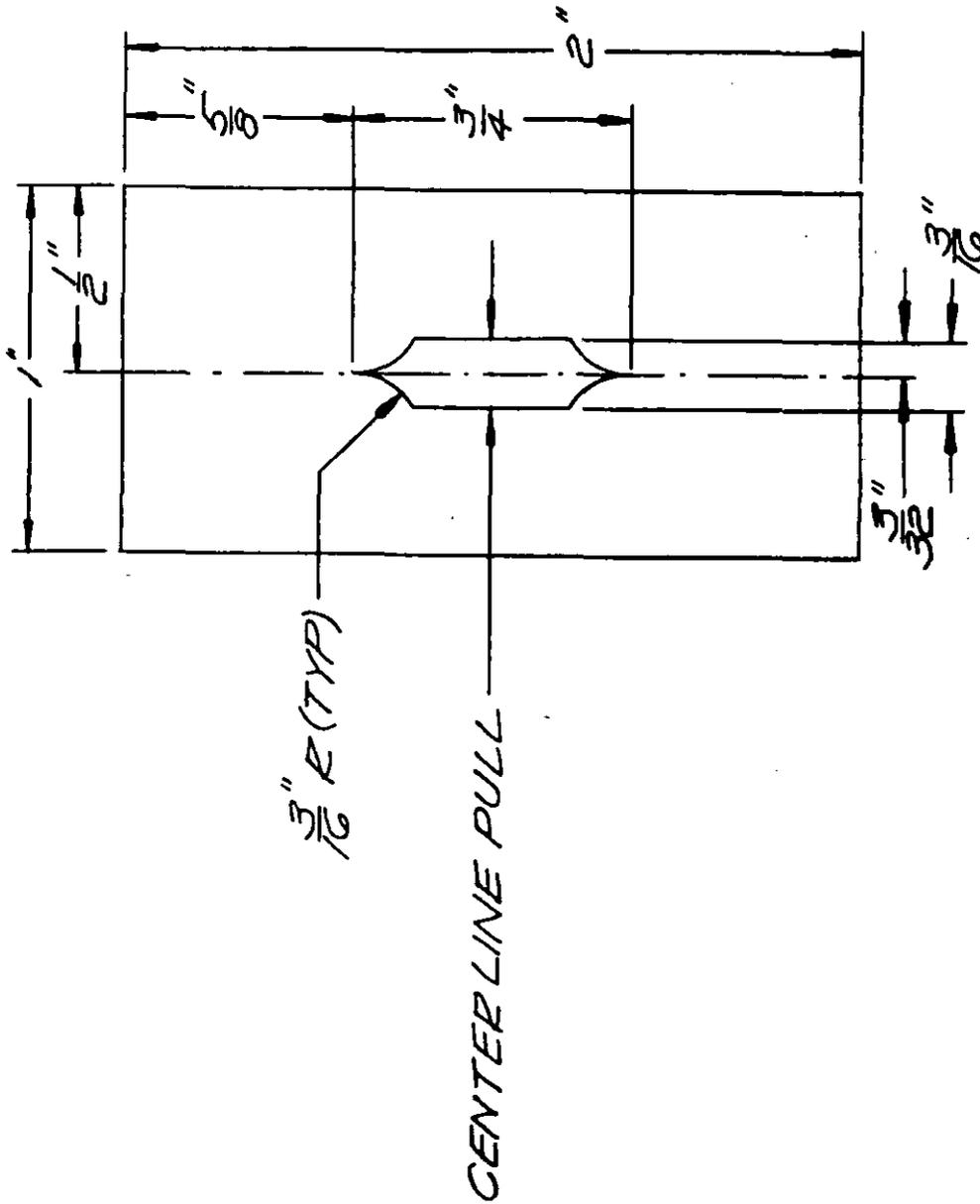


FIGURE 2161B.
SHAPE AND DIMENSIONS OF CUTTING EDGES OF
DIE FOR CUTTING SLOT TEAR TEST SPECIMENS
4-5-37A

METHOD 2211
January 15, 1969

RESISTANCE TO GRAIN CRACK, MULLEN TEST

1. SCOPE

1.1 This method is intended for determining the resistance of leather to grain cracking. It is limited to light leathers such as shoe uppers, garments, gloves, and upholstery.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be a square of leather 3 inches by 3 inches cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus

4.1.1 The standard testing apparatus shall be a motor driven machine wherein the test specimen is held firmly as hydraulic pressure is applied to one side of the specimen until cracking occurs. A hand driven tester meeting the corresponding requirements of the motor driven machine may be used except in cases of dispute wherein a motor driven machine shall be used. (See note.)

4.1.1.1 The testing machine shall permit the leather to be clamped between two circular plane clamps not less than 3 inches in diameter and having coaxial apertures of 1.22 ± 0.03 inches in diameter.

4.1.1.2 The surfaces of the clamp between which the specimen is placed shall be unpolished (matte) and may have fine spiral tool marks not over 0.010 inch in depth. The edges of the circular openings of the clamp surfaces shall be rounded to a radius of not over 0.025 inch to prevent cutting action.

4.1.1.3 The upper half of the clamp shall be connected to the clamping mechanism through a swivel joint to insure an even clamping pressure. The lower half of the clamp shall be firmly attached to the frame of the test apparatus.

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4.1.1.4 The testing machine shall contain a rubber diaphragm 0.035 ± 0.002 inch thick to contain the pressure transmitting fluid. The diaphragm shall be clamped under the lower clamping plate so that the center of its upper surface is below the plane of the clamping surface before pressure is exerted. The diaphragm shall be inspected frequently for permanent distortion.

4.1.1.5 The testing machine shall be equipped with a Bourdon tube, maximum reading-type pressure gage graduated in pounds and accurate throughout the entire range of its scale to within a value equal to 1 percent of its maximum capacity. The capacity of the gage shall be such that the individual readings will be not less than 25 percent nor more than 75 percent of the total capacity of the gage.

4.1.1.6 The apparatus shall be equipped with means of applying controlled hydraulic pressure to the underside of the diaphragm. The pressure shall be generated by a piston forcing a fluid (usually glycerine) into a pressure chamber of the apparatus at the rate of 170 ± 10 milliliters per minute. (In the manually operated machine, this shall correspond to approximately 120 revolutions per minute of a handwheel turning the displacement screw).

4.1.1.7 Means shall be provided for stopping, at the instant of cracking of the specimen, any further application of the loading pressure and for holding unchanged, the contents of the pressure chamber until the gross cracking pressure and tare diaphragm pressure indicated on the gage have been recorded.

4.1.1.8 The testing machine shall be equipped with a dial gage to measure the extension of the leather specimen. The gage shall be mounted on the machine through the screw shaft or on the sides of the clamp on the platform. The gage shall be calibrated to read directly to the nearest 0.001 inch. The machine shall be equipped with a flat anvil and a pressure foot. 101

4.2 Method cited.

4.2.1 Method 1021 - Thickness, Specimen.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 The thickness of the specimen for test shall be determined as described in method 1021. Three measurements shall be taken on the part of the specimen which will form the dome during the test and the average recorded as the thickness of the specimen. The specimen shall be mounted so that the flesh side is

METHOD 2211

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in contact with the rubber diaphragm of the testing machine. It shall be securely fastened in the clamps in such a manner that the leather will not be damaged. If stretch is to be measured, the presser foot of the extension gage shall be placed on the flat surface of the specimen and the gage set at zero. Pressure shall then be applied to the specimen until it cracks. At the moment of the first crack, the machine shall be stopped and the applied pressure and extension recorded to the nearest scale division. The upper clamp shall be completely released allowing the tare of the diaphragm to register on the pressure gage. Record the tare to the nearest scale division.

5.3 Calculation of results. The difference in pounds-per-square-inch between the gross cracking pressure and the tare of the diaphragm shall be the cracking pressure of the specimen.

5.3.1 When specified in the material specifications, the cracking strength of the specimen shall be calculated as follows:

$$\text{Cracking strength (pounds per square inch per inch of thickness)} = \frac{P}{T}$$

Where: P = gross cracking pressure of the specimen, minus the tare of the diaphragm (pounds per square inch).

T = thickness of the unstretched specimen (inches).

5.3.2 When specified in the material specification, stretch shall be calculated as follows:

$$\text{Stretch, percent} = 100 (2.56 h^2)$$

h = extension of the specimen, inches.

6. REPORT

6.1 The diaphragm cracking strength of the specimen shall be reported to the nearest pound per square inch per inch of thickness.

6.2 When specified, the stretch of the specimen shall be reported to the nearest 0.01 percent.

NOTE: A diaphragm bursting machine of the type described in this method may be obtained from B. F. Perkins & Son, Inc., Holyoke, Mass., and the E. J. Cady Company, 654 N. Harlem Avenue, River Forest, Illinois.

Any machine that operates on the same principle as that described in 4.1.1 and has coaxial apertures of 1.22 ± 0.03 inches in diameter in the clamping surface, is a valid machine for this test.

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METHOD 3011
January 15, 1969

COLORFASTNESS (RESISTANCE TO RUBBING), AATCC CROCKMETER

1. SCOPE

1.1 This method is intended for determining whether or not color may be transferred from the surface of leather to other surfaces by rubbing. Dry and wet pieces of cloth are rubbed against the leather and the resistance to the transfer of color determined by examining the cloth for staining. Three methods are provided for evaluating the amount of color transfer.

2. TEST SPECIMEN

2.1 Unless otherwise specified, the specimen shall consist of two rectangular portions of leather each not less than 5 inches by 2 inches cut from the sample unit of leather. One portion is required for testing with a dry cloth and one for testing with a wet cloth.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND MATERIALS

4.1 Crockmeter - A suitable apparatus is shown in figure 3011. The machine consists of a base piece covered with abrasive cloth securely fastened to the test apparatus on which the leather specimen rests during the test. Attached to the base is a slide piece equipped with a cylindrical "pressure foot" 5/8 inch in diameter with edges slightly rounded. A spiral clip is provided to fasten the crock cloth over the foot. The pressure foot is moved back and forth by means of a hand crank for a distance of $4 \pm 1/2$ inches, pressing the cloth against the leather specimen with a force of $16 \pm 1/2$ ounces.

4.2 Color transfer cloth.

4.2.1 White - Unless otherwise specified in the material specification, the color transfer cloth shall be a bleached, desized, 96 by 100, combed yarn, cotton lawn cloth, weighing 4 yards to the pound and cut into 2 inch squares. The cloth shall contain no bluing or optical bleach.

4.2.2 Blue - When specified, the color transfer cloth shall be vat dyed, 96 by 100, combed yarn, cotton lawn cloth, Blue 186, weighing 4 yards to the pound and cut into 2 inch squares.

4.3 Distilled water.

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4.4 Munsell Neutral Value Matte Scale 1-9 or AATCC color transference chart (see note), when evaluation by method C is specified in the material specification.

4.5 A household type wringer equipped with smooth rubber squeeze rolls 2-1/8 to 2-1/2 inches in diameter and not less than 11 inches nor more than 16 inches long. The rubber rolls shall have a Shore durometer hardness of 70 to 80 (A scale). The load exerted on the specimen shall be applied uniformly by means of a dead weight attached to the top roller. The total load of the roller, means of attaching the weight, and the weight itself shall be 60 pounds. The rolls shall be power-driven at such a speed that the specimen shall pass through the rolls at the rate of 1 inch per second.

4.6 Blotting Paper - The blotting paper shall conform to Federal Specification NNN-P-35 Paper, Blotting, (Laboratory).

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 Unless otherwise specified in the material specification, the specimen shall be mounted on the base of the testing machine with the finished side up.

5.3 Unless otherwise specified in the material specification, the evaluation of the resistance to crocking shall be by method B, as described in 5.7.2.

5.4 When a standard or comparison sample of leather is required in the material specification, the evaluation of the resistance to crocking shall be by method A as described in 5.7.1.

5.5 Dry test - The specimen shall be mounted flat and fastened securely on the abrasive cloth of the testing machine so that its long dimension will run in the direction of the rubbing. A piece of test cloth shall be mounted on the pressure foot so that it is securely held in place by the spiral clip without forming wrinkles across the face of the pressure foot. The pressure foot with the test cloth shall be lowered onto the specimen. The testing machine shall be set in operation and slid back and forth 20 times (10 cycles) each stroke covering the same surface for a distance of $4 \pm 1/2$ inches. This shall consist of 10 complete turns of the crank at a rate of approximately 1 turn per second. The test cloth shall then be removed and evaluated as specified.

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January 15, 1969

5.6 Wet test - A square of color transfer cloth shall be wet completely in distilled water and squeezed to remove the excess water. The wet cloth shall then be passed through the wringer with weight attached while being pressed between two sheets of blotting paper. (This procedure should yield a moisture content of 65 ± 5 percent based on the original bone dry weight of the cloth.) The moistened cloth shall be mounted immediately over the end of the pressure foot and the test carried out immediately as described in 5.5 using a new specimen of leather. At the end of the rubbing operation, the cloth shall be removed and dried in air.

5.7 Evaluation - Staining of the dry and wet cloths shall be considered in rating the resistance to crocking, and the results shall be evaluated by method A, B or C. Appreciable change in color means color transfer that is immediately noticeable without comparing the cloth used in the test with a piece of original cloth. If closer inspection or a change of angle of light is required to make apparent a slight change in color, the change is not considered appreciable.

5.7.1 Method A, standard or comparison sample of leather - The crock cloths from the dry and wet tests on the test specimen shall be compared with the crock cloths from the dry and wet tests on the standard or comparison specimens and rated as follows:

Satisfactory - Equal or superior to the standard or comparison specimens in resistance to crocking.

Unsatisfactory - Inferior to standard or comparison specimens in resistance to crocking.

5.7.2 Method B, direct comparison - The crock cloths of the test specimens shall be compared with the unstained crock cloth and any staining shall be rated as follows:

Excellent - No appreciable staining of the wet or dry cloth.

Good - Slight staining of the wet cloth but no appreciable staining of the dry cloth.

Fair - Appreciable staining of the wet cloth but no appreciable staining of the dry cloth.

Poor - Appreciable staining of both the wet and dry cloths.

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5.7.3 Method C, EVALUATION SCALES - The test crock cloth shall be compared with the Munsell Neutral Value Matte Scale or AATCC color transference chart and evaluated as follows:

5.7.3.1 When white crock cloth is required.

Excellent - No perceptible staining of the dry or wet cloth.

Good - Staining of the wet cloth not to be lower numerically than Munsell value 8.5. No appreciable staining of the dry cloth.

Fair - Staining of the wet cloth not to be lower numerically than Munsell value 6.5. Staining of the dry cloth not to be lower numerically than Munsell value 8.5.

Poor - Staining of the wet cloth lower numerically than Munsell value 6.5. Staining of the dry cloth lower numerically than Munsell value 8.5.

5.7.3.2 When blue crock cloth is required.

Excellent - No perceptible staining of the dry or wet crock cloth.

Good - Staining of the dry or wet crock cloth not to numerically exceed Munsell value 4.5.

Fair - Appreciable but not objectionable staining of the dry or wet crock cloth not to numerically exceed Munsell value 6.5.

Poor - Objectionable staining of the dry or wet crock cloth.

6. REPORT

6.1 When a standard or comparison sample of leather is specified, the resistance to crocking shall be reported as "pass" (satisfactory) or "fail" (unsatisfactory).

6.2 When no standard sample has been established, unless otherwise specified in the detail specification, the resistance to crocking shall be reported as "pass" or "fail". When failure is recorded, the adjective rating "good", "fair" or "poor" shall also be recorded.

6.3 When specified in the material specification, the resistance to crocking shall be reported to the nearest Munsell Neutral Value Scale value.

6.4 The wet and dry crocking resistance of each specimen shall be reported separately.

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NOTES: Apparatus which meets the requirements of this specification may be purchased from the Executive Secretary, AATCC National Headquarters, P. O. Box 886, Durham, North Carolina; and Atlas Electric Devices Company, 4114 North Ravenswood Avenue, Chicago, Illinois.

The Munsell Neutral Value Matte Scale may be purchased from the Munsell Color Company, 2441 North Calvert Street, Baltimore 18, Maryland.

White crock cloth which meets the requirements of this method may be purchased from Testfabrics, Inc., 55 Van Dam Street, New York, New York 10013.

Blue crock cloth which meets the requirements of this method may be obtained from the Sample Loan Unit, Code DPSC-PCMS, Defense Personnel Support Center, 2800 South 20th Street, Philadelphia, Pennsylvania 19101.

The Color Transference Chart may be purchased from the AATCC National Headquarters, P. O. Box 12215, Research Triangle Park, North Carolina 27709.

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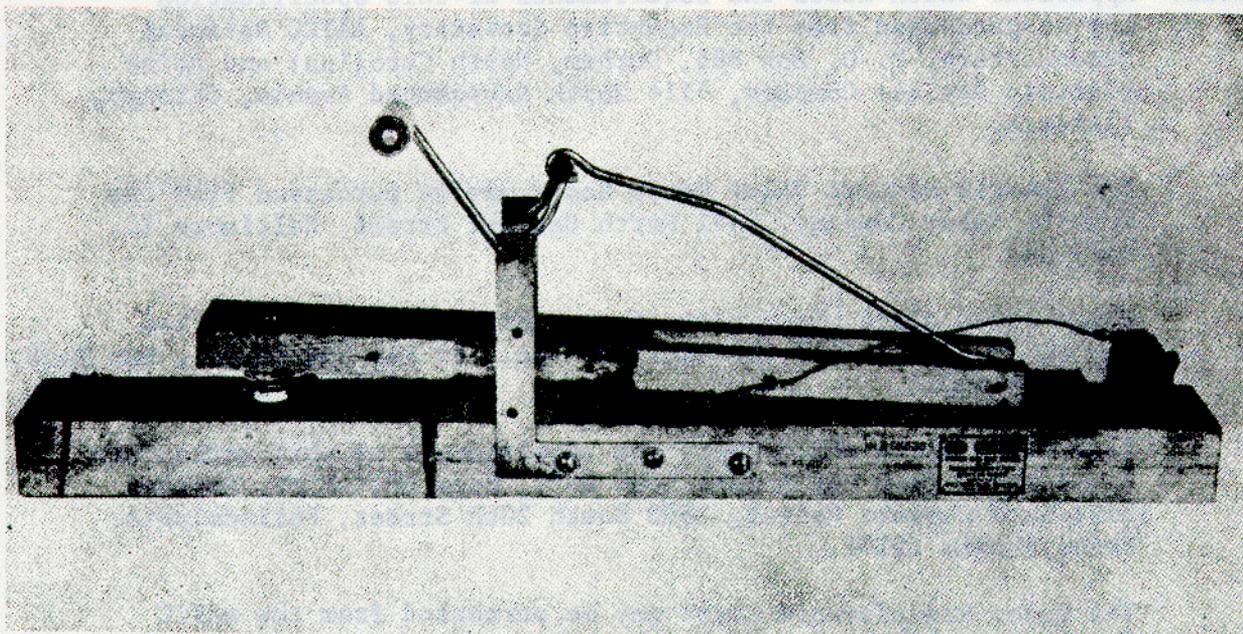


Figure 3011
CROCK METER

FED. TEST METHOD STD. NO. 311

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METHOD 3021
January 15, 1969

STAINING

1. SCOPE

1.1 This method is intended for determining whether leather bleeds (exudes) at elevated temperatures as indicated by staining produced on wet cloth in contact with the leather.

2. TEST SPECIMEN

2.1 The specimen shall consist of two squares of leather each approximately 10 ± 2 centimeters square cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND MATERIALS

4.1 Apparatus

4.1.1 Two glass plates approximately 15 by 15 by 0.6 centimeters.

4.1.2 A forced circulating-air oven capable of maintaining the required temperature to within $\pm 2^{\circ}\text{C}$.

4.2 Materials.

4.2.1 Unless otherwise specified in the material specification, the color transfer cloth shall be a pad of white cloth approximately 5 centimeters square, made of 4 layers of desized, bleached, 96 by 100, combed yarn, cotton lawn cloth weighing 4 yards to the pound. The cloth shall contain no bluing or optical bleach.

4.2.2 When specified, the color transfer cloth shall be a pad, approximately 5 centimeters square made with 4 layers of vat dyed, 96 by 100, combed yarn, cotton lawn cloth, Blue 186, weighing 4 yards to the pound.

4.2.3 Distilled water.

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5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the test shall be conducted at a temperature of $70^{\circ} \pm 2^{\circ}\text{C}$ for a period of 1 hour.

5.2 Unless otherwise specified, the grain side of the leather shall be placed in contact with the wet pad.

5.3 The pad, as specified, shall be thoroughly wet out in distilled water and squeezed to remove the excess water. The wet pad shall be placed between the two pieces of leather so that the edge of the pad will not be closer than 2.5 centimeters to the edges of the leather pieces at any point with the pad in contact with the surfaces of the leather. The specimen with pad between the pieces of leather shall be placed between two glass plates and the whole assembly placed in an oven and maintained at the required temperature for the required time. At the end of the test period, the pad shall be removed from the specimen and examined for stain.

5.4 Evaluation. Unless otherwise specified in the material specification, the test pad shall be compared with the unstained crock cloth and any staining shall be rated as follows:

Excellent - No perceptible staining of the wet pad.

Good - Perceptible but not appreciable staining of the wet pad.

Fair - Appreciable but not objectionable staining of the wet pad.

Poor - Objectionable staining of the wet pad.

Appreciable change in color means a change that is immediately noticeable without comparing the cloth used in the test with a piece of the original cloth. If closer inspection or a change of angle of light is required to make apparent a slight change in color, the change is not considered appreciable.

6. REPORT

6.1 Unless otherwise specified in the material specification, staining shall be reported as excellent, good, fair, or poor.

NOTES: White crock cloth which meets the requirements of this method may be purchased from Testfabrics, Inc., 55 Van Dam Street, New York, New York 10013.

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Blue crock cloth which meets the requirements of this method may be obtained from the Sample Loan Unit, Code DPSC-PCMS, Defense Personnel Support Center, 2800 South 20th Street, Philadelphia, Pennsylvania 19101.

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METHOD 3031
January 15, 1969

COLORFASTNESS (RESISTANCE TO RUBBING), PRECISION CROCKMETER

1. SCOPE

1.1 This method is intended for determining whether or not color may be transferred from the surface of leather to other surfaces by rubbing. Dry and wet pieces of cloth are rubbed against the leather and the resistance to the transfer of color is determined by examining the pieces of cloth for staining. Three methods are provided for evaluating the amount of color transfer.

2. TEST SPECIMEN

2.1 Unless otherwise specified, the specimen shall consist of two rectangular portions of leather each not less than 4 inches by 4 inches cut from the test unit of leather. One portion is required for testing with a dry cloth and one for testing with a wet cloth.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND MATERIALS

4.1 Crockmeter consisting of a table upon which the specimen is placed, a hold down device capable of being locked or held down by the operator after the specimen is placed on the table, and a detachable pressure foot which slides into a chassis. (See figure 3031.)

4.1.1 The pressure foot shall have a neoprene pad $\frac{5}{8}$ inch in diameter with a rounded edge ($\frac{1}{64}$ inch radius) cemented in a recess in the face of the foot.

4.1.2 The chassis shall be held to the apparatus by means of two moveable parallel levers, each with four stainless steel ball bearings. Unless otherwise specified in the material specification, the assembled chassis and pressure foot shall bear down on the specimen with a load of 1 pound.

4.1.3 The chassis shall be connected to an electric motor which makes the pressure foot describe a circle of 1-1/2 inches diameter on the specimen while rotating on its own axis in the opposite direction. After 20 revolutions at a speed of 60 rpm, the pressure foot shall have been rotated one complete revolution.

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4.1.4 A counter or signalling device shall be mounted on the apparatus so as to indicate the passing of 20 revolutions of the pressure foot.

4.2 Color transfer cloth.

4.2.1 White. Unless otherwise specified in the material specification, the color transfer cloth shall be a bleached, desized, 96 by 100, combed yarn, cotton lawn cloth, weighing 4 yards to the pound and cut into 2 inch squares. The cloth shall contain no bluing or optical bleach.

4.2.2 Blue. When specified, the color transfer cloth shall be vat dyed, 96 by 100, combed yarn, cotton lawn cloth, Blue 186, weighing 4 yards to the pound and cut into 2 inch squares.

4.3 Distilled water.

4.4 Munsell Neutral Value Matte Scale 1-9 (see note) when evaluation by Method C is specified in the material specification.

4.5 Wringer. A house hold type wringer equipped with smooth rubber squeeze rolls 2-1/8 to 2-1/2 inches in diameter and not less than 11 inches nor more than 16 inches long. The rubber rolls shall have a Shore durometer hardness of 70 to 80 (A scale). The load exerted on the color transfer cloth shall be applied uniformly by means of a dead weight attached to the top roller. The total load of the roller, means of attaching the weight, and the weight itself shall be 60 pounds. The rolls shall be power driven at such a speed that the specimen will pass through the rolls at the rate of 1 inch per second.

4.6 Blotting paper. The blotting paper shall conform to Federal Specification NNN-P-35 Paper, Blotting, (Laboratory).

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the specimen shall be mounted on the table of the testing machine with the finished side up.

5.2 Unless otherwise specified in the material specification, the evaluator of the resistance to crocking shall be by method B, as described in 5.6.2.

5.3 When a standard or comparison sample of leather is required in the material specification, the evaluation of the resistance to crocking shall be by method A as described in 5.6.1.

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5.4 Dry test. The specimen shall be mounted flat on the table of the crockmeter and secured with the hold down device. A piece of test cloth shall be mounted on the pressure foot so that it is securely held in place by a rubber ring without forming wrinkles across the face of the pressure foot. The pressure foot with the test cloth shall be lowered onto the specimen. The testing machine shall be set in operation and stopped when the pressure foot has completed 20 completed revolutions. The test cloth shall then be removed and evaluated as specified.

5.5 Wet test - A square of color transfer cloth shall be wet completely in distilled water and squeezed to remove the excess water. The wet cloth shall then be passed through the wringer with weight attached while being pressed between two sheets of blotting paper. (This procedure should yeild a moisture content of 65 ± 5 percent based on the original bone dry weight of the cloth.) The moistened cloth shall be mounted immediately over the end of the pressure foot and the test carried out immediately as described in 5.4 using a new specimen of leather. At the end of the rubbing operation, the cloth shall be removed and dried in air.

5.6 Evaluation. Staining of the dry and wet cloths shall be considered in rating the resistance to crocking, and the results shall be evaluated by method A, B, or C. Appreciable change in color means color transfer that is immediately noticeable without comparing the cloth used in the test with a piece of original cloth. If closer inspection or change of angle of light is required to make apparent a slight change in color, the change is not considered appreciable.

5.6.1 Method A, standard or comparison sample of leather. The crock cloths from the dry and wet tests on the test specimen shall be compared with the crock cloths from the dry and wet tests of the standard or comparison specimens and rated as follows:

Satisfactory - Equal or superior to the standard or comparison specimens in resistance to crocking.

Unsatisfactory - Inferior to standard or comparison specimens in resistance to crocking.

5.6.2 Method B, direct comparison. The crock cloths of the test specimens shall be compared with the unstained crock cloth and any staining shall be rated as follows:

Excellent - No appreciable staining of the wet and dry cloths.

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Good - Slight staining of the wet cloth and no appreciable staining of the dry cloth.

Fair - Appreciable staining of the wet cloth but no appreciable staining of the dry cloth.

Poor - Appreciable staining of both the wet and dry cloths.

5.6.3 Method C, Evaluation Scale. The crock cloth of the test specimens shall be compared with the Munsell Neutral Value Matte Scale, or AATCC Color Transference Chart and evaluated as follows:

5.6.3.1 When white crock cloth is required.

Excellent - No perceptible staining of the wet or dry cloth.

Good - Staining of the wet cloth not to be lower numerically than Munsell value 8.5. No appreciable staining of the dry cloth.

Fair - Staining of the wet cloth not to be lower numerically than Munsell value 6.5. Staining of the dry cloth not to be lower numerically than Munsell value 8.5.

Poor - Staining of the wet cloth lower numerically than Munsell value 6.5. Staining of the dry cloth lower numerically than Munsell value 8.5.

5.6.3.2 When blue crock cloth is required.

Excellent - No perceptible staining of the dry or wet crock cloth.

Good - Slight staining of the dry or wet crock cloth, not to exceed Munsell value 4.5.

Fair - Appreciable but not objectionable staining of the dry or wet crock cloth, not to exceed Munsell value 6.5.

Poor - Objectionable staining of the dry or wet crock cloth.

6. REPORT

6.1 When a standard or comparison sample of leather is specified, the resistance to crocking shall be reported as "pass" (satisfactory) or "fail" (unsatisfactory).

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6.2 Unless otherwise specified in the detail specification, when no standard sample has been established, the resistance to crocking shall be reported as "pass" or "fail." When failure is reported, the adjective rating "good," "fair," or "poor" shall also be reported.

6.3 When specified in the material specification, the resistance to crocking shall be reported as the nearest Munsell Neutral Value Scale value.

6.4 The wet and dry crocking resistance of each sample unit shall be reported separately.

NOTES: Apparatus which meets the requirements of this specification may be purchased from Mul Tech Industries, Lee Avenue, Box JJ, Southampton, Long Island, New York 11968.

The Munsell Neutral Value Matte Scale may be purchased from the Munsell Color Company, 2441 North Calvert Street, Baltimore 18, Maryland.

The white crock cloth which meets the requirements of this method may be purchased from Testfabrics, Inc., 55 Van Dam Street, New York, New York 10013.

The blue crock cloth which meets the requirements of this method may be obtained from the Sample Loan Unit, Code DPSC-PCMS, Defense Personnel Support Center, 2800 South 20th Street, Philadelphia, Pennsylvania 19101.

The Color Transference Chart may be purchased from the AATCC National Headquarters, P. O. Box 12215, Research Triangle Park, North Carolina 27709.

A wringer meeting the requirements of this method is available from Atlas Electric Devices Company, 4114 North Ravenswood Avenue, Chicago, Illinois 60613.

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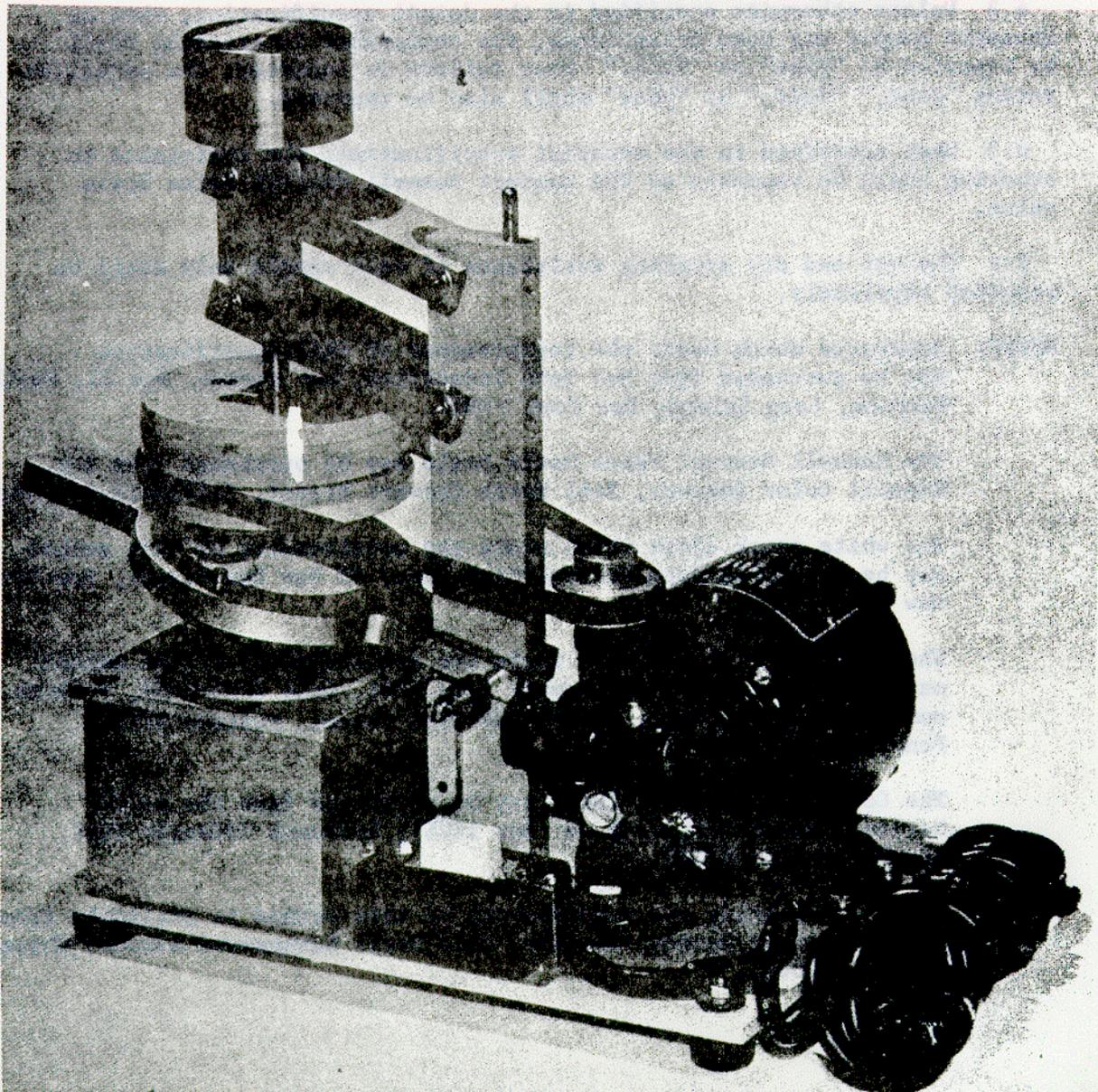


FIGURE 3031
PRECISION CROCKMETER

FED. TEST METHOD STD. NO. 311

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METHOD 3111
January 15, 1969

TACKINESS

1. SCOPE

1.1 This method is intended for determining the tendency of cloth to adhere to leather.

2. TEST SPECIMEN

2.1 The specimen shall be a 10 ± 2 centimeter square of leather cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND MATERIALS

4.1 Apparatus

4.1.1 A forced circulating air oven capable of maintaining the required temperature to within $\pm 2^{\circ}\text{C}$.

4.1.2 Cheesecloth conforming to type 1, class 2 of Federal Specification CCC-C-440, Cloth, Cheesecloth, Cotton, Bleached and Unbleached.

4.1.3 A suitable device for maintaining the required pressure on the specimen for the time specified in 5.1.

4.1.4 Glass or plastic flat plate large enough to cover the specimen. The plate shall be not more than 4 millimeters thick.

5. PROCEDURE

5.1 Unless otherwise specified in the detail specification, the test shall be carried out at a temperature of $50 \pm 2^{\circ}\text{C}$ under a pressure of 70 ± 2 grams per square centimeter for a period of 5 minutes.

5.2 The specimen shall be placed in the oven and brought to the required temperature. With the specimen remaining in the oven, a piece of cheese cloth shall be spread over the finished surface of the leather. A glass or

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plastic plate shall be placed over the cheesecloth and the required pressure applied to the whole assembly. At the end of the exposure period, the pressure shall be removed, and the specimen and cloth shall be taken from the oven and cooled to $23^{\circ} \pm 1^{\circ}\text{C}$. The cloth shall be slowly pulled from the leather while observing any tendency to adhere to the leather.

6. REPORT

6.1 Any tendency of the cloth to adhere to leather shall be reported as pass or fail.

METHOD 3121
January 15, 1969

BLOCKING

1. SCOPE

1.1 This method is intended for determining the resistance of leather to blocking under specific conditions of temperature, humidity, and pressure.

2. TEST SPECIMEN

2.1 The test specimen shall be a rectangle approximately 2.5 centimeters by 10 centimeters, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 American Medical museum jar (with lid) having inner dimensions of 16 centimeters high, 10 centimeters long, and 8 centimeters wide. There shall be three indentations at the top of each 8 centimeter side.

4.2 Petri dish 100 millimeters by 15 millimeters, with cover.

4.3 Weight, 2000 grams.

4.4 Forced circulating air oven capable of maintaining the specified temperature.

5. PROCEDURE

5.1 The specimen shall be perforated 2 millimeters from one end and hung on a paper clip that has been opened to its full length. No more than five specimens shall be hung on one clip. The clip holding the specimen shall be set into the indentations of the jar. No more than three clips shall be placed in a jar. The jar shall be filled with tap water at $23 \pm 2^{\circ}$ C to a level no higher than 1.25 centimeters from the bottom of the hanging specimen. The lid shall then be placed on the jar and the specimen allowed to condition for four hours.

5.2 After conditioning, the specimen shall be removed from the museum jar and quickly (to preserve conditioning) folded grain to grain to form a 2.5 centimeter by 5 centimeter rectangle. The folded specimen shall then be placed in an inverted (rim up) petri dish cover. The bottom plate of the dish shall then be placed concentrically within the inverted cover plate so as to cover the specimen evenly.

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METHOD 3121

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5.3 A 2000 gram weight shall be placed on top of the inverted bottom plate. The whole assembly shall then be placed in an oven at $80 \pm 3^{\circ}\text{C}$ for two hours and then removed. The weight and cover plate shall be removed from the assembly and the specimen allowed to cool for 30 minutes.

5.4 The cooled and folded specimen shall be examined by slowly pulling the fold apart by hand. The specimen shall be examined for ease of separation, grain damage, and finish peeling.

6. REPORT

6.1 The resistance of leather to blocking shall be reported as pass or fail. Specimens which separate with ease and which show no grain damage pass the test. Specimens which separate with difficulty, requiring force to separate the fol., and which show finish peeling fail the test.

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METHOD 3202

January 15, 1969

FINISH STABILITY

1. SCOPE

1.1 This method is intended for determining the resistance of leather finishes to cracking, flaking, or becoming tacky after aging.

2. TEST SPECIMEN

2.1 The specimen for test shall be a rectangle of leather approximately 10 ± 2 centimeters square cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Forced circulating air oven capable of maintaining a temperature between 55° and 60°C .

5. PROCEDURE

5.1 The specimen shall be placed in the circulating air oven at 55° to 60°C , by hanging so that complete circulation of the air around the specimen is possible. The specimen shall be exposed for $20 \pm 1/4$ hours and then allowed to cool. After cooling, the aged leather shall be doubled over with the grain side in, the folded edge creased slightly, and the finished grain surface rubbed together. The grain surface shall then be examined for cracks, parting of the finish from the leather, and tackiness.

6. REPORT

6.1 The presence of cracks, parting of the finish from the leather, or tackiness shall be reported.

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METHOD 3203

January 15, 1969

CORROSIVE EFFECTS OF LEATHER

1. SCOPE

1.1 This method is intended for determining the corrosive effects of leather when in contact with metals and metal alloys.

2. TEST SPECIMEN

2.1 The specimen for test shall be a rectangle of leather approximately 2.5 centimeters in width and 8 centimeters in length, and split to a uniform thickness.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Desiccator, containing a saturated solution of ammonium dihydrogen phosphate, to maintain a relative humidity of 92.5 ± 2.5 percent.

4.1.2 Four plates of the specified metal to be tested, approximately 5 centimeters by 10 centimeters, of uniform thickness, and having a surface roughness of 12 ± 4 microinches RMS finish.

4.1.3 Weight, 12 ± 2 kilograms.

4.1.4 Glass plate of same approximate dimensions as metal plates.

4.2 Reagents.

4.2.1 Hydraulic fluid, conforming to Military Specification MIL-H-6083.

4.2.2 Naphtha, conforming to Military Specification MIL-N-15178A.

4.2.3 Ammonium dihydrogen phosphate.

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METHOD 3203
January 15, 1969

5. PROCEDURE

5.1 Qualitative test.

5.1.1 The specimen for test shall be conditioned for 72 hours in a desiccator at 92 ± 2.5 percent relative humidity and $23^{\circ} \pm 1^{\circ}\text{C}$.

5.1.2 The metal plates shall be polished with a non-abrasive cloth and washed in naphtha solvent.

5.1.3 The conditioned specimen and metal plates shall be immersed in the hydraulic fluid and the excess allowed to drip off. The specimen shall then be placed between the two metal plates and placed in the desiccator. The metal plates shall be held in close contact with the specimen by means of a 12 ± 2 kilogram weight, with a glass plate between the metal plate and the weight to prevent reaction between dissimilar metals. Two control plates shall also be prepared in the same manner and placed in the desiccator so that they do not touch each other or the test specimen. The desiccator shall be closed, and the specimens and plates shall be exposed to an atmosphere of 92.5 ± 2.5 percent relative humidity and $23^{\circ} \pm 1^{\circ}\text{C}$ for fourteen days.

5.1.4 At the end of the 14 day period, the test plates and specimen shall be removed from the desiccator. The plates shall be washed with naphtha solvent, polished with a non-abrasive cloth, and then examined for staining as evidenced by discoloration or corrosion as evidenced by visible pitting and/or erosion of the metal when compared with the control plates.

5.2 Quantitative test.

5.2.1 The metal plates shall be polished and washed as specified in 5.1.2, dried thoroughly in a circulating air oven, and weighed to the nearest 0.001 gram. Record the weight of the test plates as W_1 and that of the control plates as C_1 .

5.2.2 The procedure described in 5.1.3 shall be followed, except that the weighed metal plates shall be used. At the end of the 14 day period of exposure, the plates shall be washed in naphtha solvent, polished with a non-abrasive cloth, dried thoroughly, and then weighed to the nearest 0.001 gram. Record the weight of the test plates as W_2 and that of the control plates as C_2 .

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5.2.3 Calculations. The percent of corrosion caused by the leather shall be calculated as follows:

$$\text{Corrosion, percent} = \left[\frac{W_1 - W_2}{W_1} \times 100 \right] - \left[\frac{C_1 - C_2}{C_1} \times 100 \right]$$

Where: W_1 = original weight of the test plates.

W_2 = weight of the test plates after testing.

C_1 = original weight of the control plate.

C_2 = weight of the control plate after testing.

6. REPORT

6.1 Qualitative. The absence or presence of staining or corrosion caused by the specimen shall be reported.

6.2 Quantitative. The corrosion of metal caused by the leather shall be reported to the nearest 0.1 percent.

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METHOD 3211
January 15, 1969

RESISTANCE TO PERSPIRATION

1. SCOPE

1.1 This method is intended for determining the effect of perspiration on leather. The leather is subjected to treatment with artificial perspiration, and the cracking measured after bending the material through 180 degrees.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 15 centimeters in length and 5 centimeters in width, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus

4.1.1 A forced circulating air oven capable of maintaining the required temperature within $\pm 2^{\circ}\text{C}$.

4.1.2 A mandrel 5 centimeters in diameter.

4.2 Reagents.

4.2.1 Salt solution with a pH of 7.9 ± 0.2 , consisting of the following:

- Sodium chloride. 10 grams
- Ammonium carbonate 4 grams
- Anhydrous disodium phosphate 1 gram
- Distilled water to make one liter of solution

If necessary, the pH of the solution shall be adjusted with ammonium carbonate or lactic acid.

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5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the test shall be conducted at a temperature of $50^{\circ} \pm 2^{\circ}\text{C}$ for a period of 24 hours.

5.2 The specimen shall be immersed in the artificial perspiration solution at $23^{\circ} \pm 1^{\circ}\text{C}$ until its weight increases approximately 20 percent. The wet specimen shall be immediately transferred from the solution to the constant-temperature air oven and maintained at the required temperature for the required period of time. The aged specimen shall be removed from the chamber and cooled to $23^{\circ} \pm 1^{\circ}\text{C}$. The specimen shall be bent slowly, with the grain side out, through 180 degrees over a mandrel 5 centimeters in diameter. The bent specimen shall be examined for cracks.

5.3 The specimen shall be graded according to its resistance to cracking, as follows:

Poor - Cracks completely across the specimen.

Fair - Cracks approximately one-half across the specimen.

Good - Cracks only along the edge of the specimen.

Very Good - No cracks formed.

6. REPORT

6.1 The specimen shall be reported as having poor, fair, good, or very good resistance to cracking.

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METHOD 3221
January 15, 1969

RESISTANCE TO PERSPIRATION (WHITE CHROME LEATHER)

1. SCOPE

1.1 This method is intended for determining the effect of perspiration on upper leather. The procedure has been designed in particular for chrome-tanned white shoe upper leather. The leather is subjected to treatment with a formulation of artificial perspiration for breakdown of chrome-tanned leather. Resistance to grain cracking shall be used as the criterion of deterioration.

2. TEST SPECIMEN

2.1 The specimen shall consist of two square pieces of leather having minimum dimensions of 3 by 3 inches or two discs of leather having a minimum diameter of 3 inches.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS, REAGENTS, AND METHOD CITED

4.1 Apparatus.

4.1.1 A circulating air oven, capable of maintaining the required temperature within $\pm 2^{\circ}\text{C}$.

4.1.2 A wide-mouth 2 quart bottle.

4.1.3 A glass tray 1.5 inches deep.

4.1.4 A Mullen Tester with 1500 pound per square inch gauge.

4.2 Reagents.

4.2.1 A solution of artificial perspiration with a pH of 7.5 consisting of the following:

Sodium chloride, grams	9.0
Urea, grams	1.67
Sodium lactate-60 percent solution (60 grams sodium lactate in 40 grams distilled water)	86.0
Disodium phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$), grams	0.165
Distilled water to make 1 liter of solution	--

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The pH of the solution shall be adjusted with lactic acid or ammonium carbonate depending on whether acid or base is needed to bring pH to 7.5.

4.3 Method cited.

4.3.1 Method 2211 - Resistance to Grain Crack, Mullen Test.

5. PROCEDURE

5.1 The control specimen shall be tested on the Mullen Tester for grain crack strength by method 2211. The specimen for treatment shall be placed in the glass tray and covered with artificial perspiration to a depth of 0.75- 1.00 inches. The leather shall be worked in the solution by folding it grain in and rolling the fold while applying pressure with the fingertips. As many as three cycles of flexing may be required to wet the leather. After one hour the piece shall be removed and suspended in the 2-quart bottle over 2 ounces of water. The bottle containing the treated specimen shall be completely sealed and then placed into a circulating air oven at 70°C for 48 hours. At the end of this period of time, the specimen shall be removed from the bottle and hung up to dry at room temperature for 16 hours. The specimen shall then be conditioned for 48 hours and then tested on the Mullen Tester (Note 1) for grain crack by the same procedure as that used for the control.

5.2 Calculation of results. The percent change in the grain crack strength of the treated specimen shall be calculated as follows:

$$\text{Percent change in grain crack strength} = \frac{C-E}{C} \times 100$$

Where C = grain crack strength of the control specimen
 (pounds per square inch)

E = grain crack strength of the treated specimen
 (pounds per square inch)

6. REPORT

6.1 The change in grain crack strength of the specimen shall be reported to the nearest pound per square inch.

6.2 The percent change in grain crack strength of the specimen shall be reported to the nearest 0.01 percent.

NOTE: 1. Certain specimens may be so deformed as to make the Mullen determination impossible. Such specimens shall be reported as having zero grain crack strength.

METHOD 4011

January 15, 1969

CRACKING, VISE

1. SCOPE

1.1 This method is intended for determining the resistance of leather to cracking on being bent by mechanical means.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather approximately 15 centimeters in length and 2.5 centimeters in width, cut from each sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Vise. The vise shall have plain jaws not less than 7 centimeters wide.

4.2 Mandrel. The mandrel shall be approximately 15 centimeters in length, and have a diameter as specified in the material specification.

4.3 Metal bar. The metal bar shall be approximately 15 centimeters long, 1.9 centimeters in width and shall have a thickness equal to the specified diameter of the mandrel.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions on a material conditioned as specified in Section 5.

5.2 The specimen shall be bent into a loop with the grain side out. The mandrel of the specified diameter shall be placed in the loop of the specimen. The metal bar having a thickness equal to the diameter of the mandrel shall be placed on top of the mandrel. The whole assembly shall be placed in the vise with the loop downward as shown in figure 4011. The specimen shall be bent around the mandrel and against the bar by slowly closing the vise jaws until the mandrel and bar cannot be moved by hand. The grain side of the specimen shall be examined for cracks in the area around the bend.

METHOD 4011

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6. REPORT

6.1 The presence of cracking shall be reported.

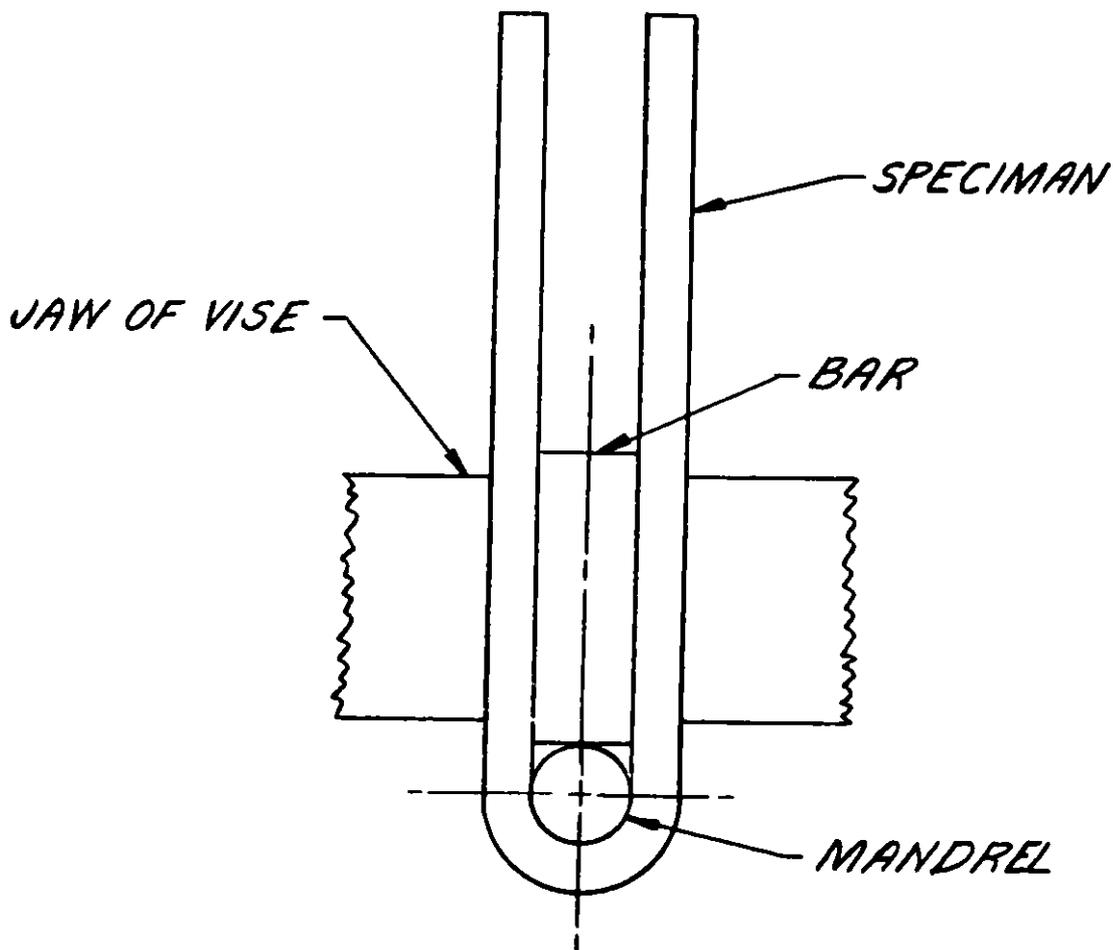
6.2 The diameter of the mandrel used shall be reported.

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METHOD 4011

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FIGURE 4011

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CRACKING, HAND

1. SCOPE

1.1 This method is intended for determining the resistance of leather to cracking on being bent by hand.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather approximately 15 centimeters in length and 2.5 centimeters in width, cut from the sample unit.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 The apparatus shall consist of a mandrel approximately 15 centimeters in length and having a diameter as specified in the material specification.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 The specimen shall be bent slowly by hand over a mandrel of the specified diameter. The bend shall be made through an angle of 180° with the grain side of the leather out. The grain side of the specimen shall be examined for cracks in the area around the bend.

6. REPORT

6.1 The presence of cracking shall be reported.

6.2 The diameter of the mandrel shall be reported.

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METHOD 4111

January 15, 1969

PIPING

1. SCOPE

1.1 This method is intended for determining the pipeyness of leather when bent.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 15 centimeters in length and 2.5 centimeters in width, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 The apparatus shall consist of a mandrel approximately 15 centimeters in length and having a diameter as specified in the material specification.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed on material conditioned as specified in Section 5.

5.2 The specimen shall be bent slowly by hand over a mandrel of the specified diameter. The bend shall be made through an angle of 180° with the grain side of the leather next to the mandrel. The mandrel shall be removed, the specimen held in the bent position, and the edges and grain side examined for piping.

6. REPORT

6.1 The absence or presence of piping in the specimen shall be reported.

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METHOD 4211

January 15, 1969

STIFFNESS

1. SCOPE

1.1 This method is intended for determining the stiffness of leather by measuring the force required to bend the material through a given angle.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather 4.0 inches in length and 0.5 inch in width, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A stiffness testing machine. A suitable apparatus is shown in figure 4211.

4.1.1 The machine shall contain a vise, for clamping one end of the specimen, mounted on a horizontal shaft which is driven at about 0.2 revolutions per minute by a motor. A pendulum weight system equipped with a set of detachable weights shall be attached to the shaft. The force applied to turn the shaft is regulated by changing the weights on the pendulum. Weights shall be supplied that give maximum bending moments from 0.100 to 5.000 inch - pounds in steps of 0.260 inch-pounds.

4.1.2 The machine shall be equipped with a load scale consisting of a pointer moving over a scale that measures the force applied to turn the shaft. The scale shall be graduated in percent of maximum bending moment, 100 percent corresponding to 90° swing of the pendulum.

4.1.3 The machine shall be equipped with an angular deflection scale graduated in degrees. A pointer that moves over the scale shall be attached to the shaft. The scale shall measure the angle through which the specimen is bent.

4.1.4 The machine shall contain a pin that can be fixed in different positions to regulate the length of the span between the vise and the free end of the specimen that rests against the pin to give spans of 0.25, 0.50, 1.00 and 2.00 inches.

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4.1.5 The machine shall be equipped so that the motor can be thrown in and out of gear, the vise moved to the original position by hand, and the pointers adjusted to zero positions.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 The machine shall be placed in test position and leveled. A suitable moment weight shall be placed on the pendulum and then, if necessary, the load scale adjusted to indicate zero. The moment weight applicable to a given specimen shall be determined by trial. The pin shall be adjusted to give the specified span. The motor shall be started and kept running throughout the test since its vibration minimizes friction effects in the weighting system.

5.3 One end of the specimen shall be clamped firmly in the vise with the grain side of the leather down and the long side parallel to the edge of the dial plate.

5.4 The apparatus shall be adjusted by hand so that the free end of the specimen touches the pin and shows a 1 percent load reading. Both pointers shall then be adjusted to zero readings and subsequent load readings reduced by one division.

5.5 The motor engaging lever shall be held down and the apparatus maintained in action until the angular deflection scale indicates that the specimen has been bent through the required number of degrees. At that instant, the load scale reading shall be read from the position of the pointer on the load scale and the value recorded as the load scale reading.

5.6 Calculations:

5.6.1 When specified in the material specification, the stiffness shall be calculated in one or more of the following ways:

5.6.1.1 Bending moment, inch-pounds. The bending moment shall be that applied when the specimen is bent to a 60° angular deflection and shall be recorded as follows:

$$\text{Bending moment, inch-pounds} = \frac{\text{Load scale reading} \times \text{Moment weight}}{100}$$

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5.6.1.2 Load, pounds. The load in pounds shall be that applied when the specimen is bent to a 60° angular deflection and shall be calculated as follows:

$$\text{Load, pounds} = \frac{\text{Load scale reading} \times \text{Moment weight}}{100 \times \text{Span length (inches)}}$$

6. REPORT

6.1 The load scale reading of the specimen shall be reported to the nearest scale division.

6.2 When specified in the material specification, the bending moment shall be reported to the nearest 0.001 inch-pound.

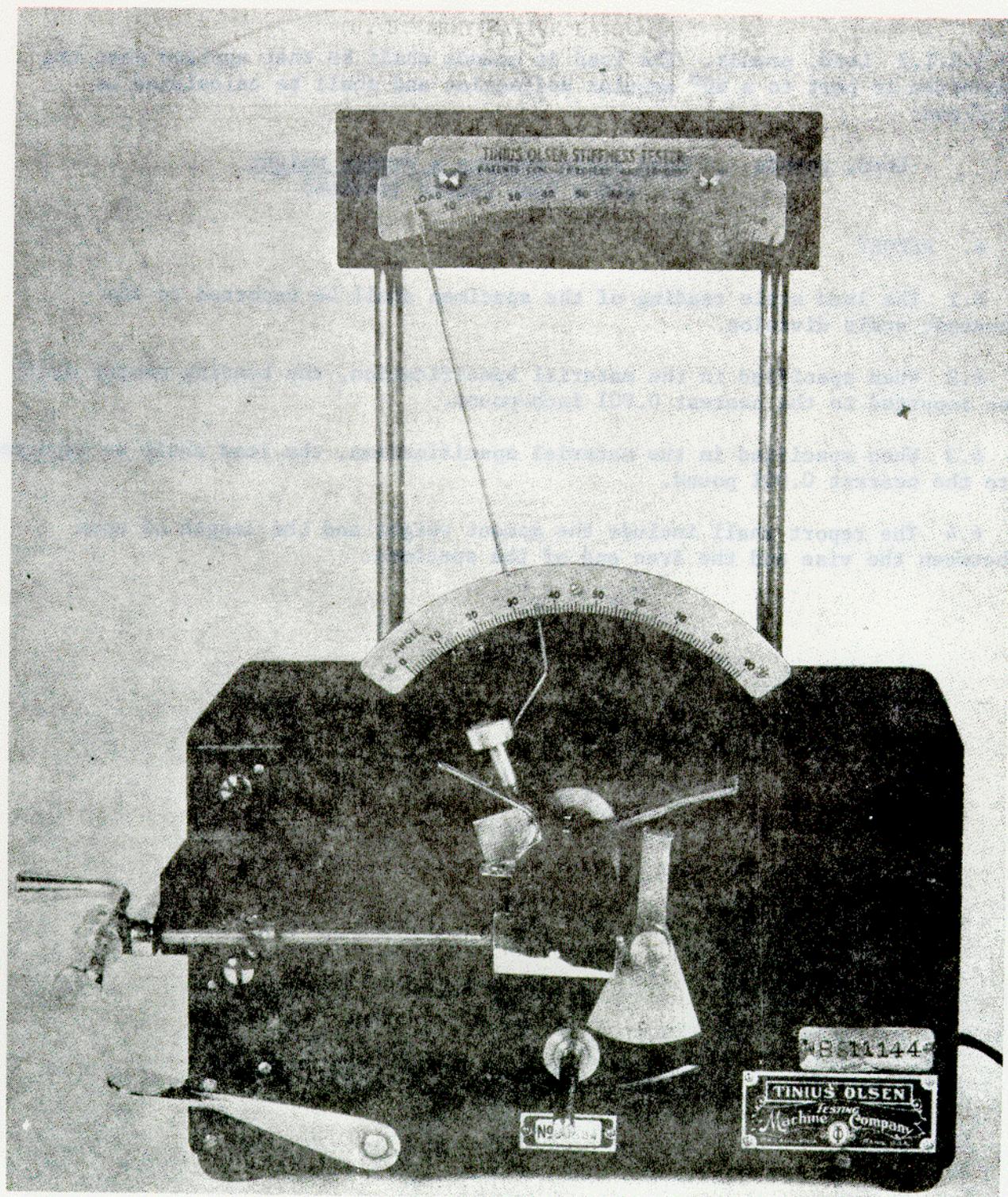
6.3 When specified in the material specification, the load shall be reported to the nearest 0.001 pound.

6.4 The report shall include the moment weight and the length of span between the vise and the free end of the specimen.

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Figure 4211 - Stiffness Testing Machine

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COMPRESSIBILITY

1. SCOPE

1.1 This method is intended for determining the change in thickness of a specimen on being subjected to a specified pressure.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be a piece of leather $2 \pm 1/16$ inches square, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 Compression apparatus - Any type of compression apparatus, that will permit applying a pressure of 3000 ± 100 pounds per square inch at a rate not to exceed 200 pounds per square inch per second and maintain this pressure for a specified period of time, may be used. The apparatus shall be equipped with two flat metal plates larger than the test specimen and a device for measuring the applied force.

4.1.2 A stop watch or other timing device.

4.2 Method cited.

4.2.1 Method 1021, - Thickness, Specimen.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed on material conditioned as specified in Section 5.

5.2 Unless otherwise specified in the material specification, the test shall be carried out using a total force of 3000 ± 100 pounds per square inch for a period of 3 minutes.

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5.3 The thickness of the specimen shall be determined as described in Method 1021, except that 4 measurements shall be made and the average recorded as thickness T_1 of the specimen. One measurement shall be made at the middle of each of the four sides approximately 0.5 inch from the edge.

5.4 The specimen shall be placed in the compression machine and pressure applied at a rate not to exceed 200 pounds per square inch per second until the specified total force is reached. The force shall remain on the specimen for the specified period of time. At the end of this period, the force shall be released, the specimen removed from the machine, the thickness immediately determined at the same points as described in 5.3, and the average recorded as T_2 .

5.5 Calculation. The percent compression of the specimen shall be calculated as follows:

$$\text{Compression, percent} = \frac{T_1 - T_2}{T_1} \times 100$$

Where: T_1 = thickness of the original uncompressed specimen, inches.

T_2 = thickness of the compressed specimen, inches.

6. REPORT

6.1 The percent compression of the test specimen shall be reported to the nearest 0.1 percent.

METHOD 5021

January 15, 1969

MILDEW RESISTANCE; DIRECT INOCULATION,
PURE CULTURE, NON-STERILE SPECIMEN METHOD

1. SCOPE

1.1 This method is intended for evaluating the efficiency of fungicides in preventing damage. The test is not very severe, but may be used as a test for eliminating inefficient fungicides and determining the uniformity of distribution. The procedure is not recommended for use in procurement documents. In the interest of standardizing on test procedures, this method is included for use in research areas where an initial screening test for prospective mildew inhibitors is desired.

2. TEST SPECIMEN

2.1 The specimen for test shall be 2 squares approximately 5 by 5 centimeters cut from the leather to be tested.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Autoclave capable of maintaining an exhaust temperature of $122^{\circ} \pm 2^{\circ}\text{C}$ at a pressure of 15.5 ± 0.5 pounds per square inch.

4.1.2 Incubation chamber capable of maintaining the required temperature within $\pm 2^{\circ}\text{C}$.

4.1.3 Three 10 centimeter Petri dishes.

4.2 Reagents.

4.2.1 Culture medium having the following composition:

Ammonium nitrate (NH_4NO_3)	3.0 grams
Potassium dihydrogen phosphate (KH_2PO_4)	2.5 grams
Potassium monohydrogen phosphate (KHPO_4)	2.0 grams

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Magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$)	2.0 grams
Agar	20.0 grams
Distilled water to make	1000.0 milliliters

Adjust pH to between 6.4 to 6.8 with HCl or NaOH if necessary.

4.2.2 Potato dextrose agar.

4.2.3 Organism. Aspergillus niger QM-458, TC-215-4247, or ATCC 6275 (see Note 1).

5. PROCEDURE

5.1 The culture medium described in 4.2.1 shall be melted and poured into two Petri dishes and left undisturbed until the agar has hardened. The Petri dishes with the agar shall then be sterilized in an autoclave for 20 minutes at a steam pressure of 15.5 ± 0.5 pounds per square inch and an exhaust temperature of $122^\circ \pm 2^\circ\text{C}$ after which the agar shall be allowed to harden.

5.2 Scrapings from a ripe fruiting culture of Aspergillus niger (10 - 14 days old) which completely covers a surface equivalent to a 10 centimeter Petri dish shall be added to an Erlenmeyer flask containing 100 milliliters of water. The organism suspension should be made up with a non-toxic wetting agent such as sodium lauryl sulfate at 0.05 percent concentration.

5.3 When the agar has hardened, the specimen shall be dipped in a fresh organism suspension. Each square of the specimen shall be placed in contact with the agar in a separate culture dish and the dishes covered.

5.4 The inoculated specimen shall be incubated for seven days at a temperature of $29^\circ \pm 1^\circ\text{C}$. At the end of the incubation period, the specimen shall be examined for the presence of any growth of Aspergillus niger.

5.5 A viability test shall be conducted at the same time as the test specimen to verify the viability of the test organism. A Petri dish containing potato dextrose agar inoculated with the organism shall be incubated in the same incubator with the test specimen. If an abundant growth of the test organism does not occur, the test shall be considered inconclusive and shall be repeated.

6. REPORT

6.1 The amount of Aspergillus niger growth on the specimen shall be reported as none, slight, or heavy, as applicable.

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NOTE 1: Pure cultures of the Aspergillus niger organism may be obtained from the following sources:

QM-458 - U. S. Army Natick Laboratories, Kansas Street, Natick, Massachusetts 01760.

ATCC-6275 - American Type Culture Collection, 2029 M Street, N.W., Washington, D. C. 20036.

TC-215-4247 - Cotton Division, U. S. Plant Industry Station, Beltsville, Maryland 20705.

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METHOD 5041
January 15, 1969

MILDEW RESISTANCE, TROPICAL CHAMBER METHOD

1. SCOPE

1.1 This method is intended for determining the resistance of leather to fungus growth under hot, moist tropical conditions. The procedure is not recommended for use in procurement documents. However, in the interest of standardizing on test procedures, this method is included for use in research areas where an initial screening test for prospective mildew inhibitors is desired.

2. TEST SPECIMEN

2.1 The specimen for test shall be any convenient size suitable for testing.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from each sample unit shall be tested.

4. APPARATUS, MATERIAL AND REAGENTS

4.1 Apparatus

4.1.1 Tropical chamber capable of maintaining a temperature of $30 \pm 2^{\circ}\text{C}$ and a relative humidity of 95 ± 5 percent. The chamber shall have sufficient circulation of air to insure uniform conditions of humidity throughout the test area and to remove volatile compounds that may have a tendency to inhibit growth. The chamber shall be large enough to accommodate the test specimens without crowding.

4.2 Material

4.2.1 Unbleached greige cotton duck 2 centimeters by 15 centimeters.

4.3 Reagents

4.3.1 Potassium dihydrogen phosphate (KH_2PO_4)

4.3.2 Ammonium nitrate (NH_4NO_3)

4.3.3 Magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$)

4.3.4 Glycerol

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5. PROCEDURE

5.1 The specimen of leather shall be placed in a tropical chamber maintaining conditions of $30^{\circ} \pm 2^{\circ}\text{C}$ and 95 ± 5 percent relative humidity. The specimen shall be allowed to hang in a position such that it is not in contact with any other specimen and a free circulation of the moist air around the specimen is obtained. The specimens shall be inspected each week for mildew growth. Resistance to fungus growth is considered adequate if no appearance of growth is visible after five weeks exposure to the above conditions.

5.2 A viability control shall be placed in the chamber adjacent to the test strips at the same time as the specimens being tested. The control shall be a piece of unbleached greige cotton duck approximately 2 centimeters by 15 centimeters. The strip shall be dipped in a solution containing 1.5 grams potassium dihydrogen phosphate, 1.5 grams ammonium nitrate, 0.25 gram magnesium sulfate, and 10 ml. glycerol per liter. The strip shall be blotted between paper towels and hung to dry before placing in the chamber. Failure of the viability control strip to develop mold within two weeks indicates inadequate molding conditions in the chamber.

6. REPORT

6.1 The appearance of fungus growth shall be reported.

METHOD 6001

January 15, 1969

CHEMICAL TESTS, GENERAL

1. SCOPE

1.1 These methods are intended for use in qualitative and quantitative analysis of various types of leather and some of their components. Leather is, in general, a highly heterogeneous system of chemical components, some of which are difficult to characterize quantitatively. The methods of analysis, in particular with vegetable-tanned leather, are highly empirical, and precise duplication of results depends upon close control of the conditions prescribed in the methods.

1.2 Because of the empirical features of some of these methods, uncertainties attend their extension to types of leather prepared with new products with which there has been no analytical experience. Caution should be exercised in interpreting the results obtained on applying these methods to these leathers. This caution is particularly applicable to the analysis of leather tanned with zirconium, iron salts, lignosulfonates, or synthetic tannins; leather containing fillers such as rubber and nitrogen-containing resin; modified oils and plastic finishes. The methods are, however, applicable to all leathers covered by Federal specifications.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen for chemical analysis shall be a specified weight of the composite sample described in method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two determinations shall be made on the composite sample prepared from the sample for test.

4. APPARATUS AND REAGENTS

4.1 Apparatus

4.1.1 Unless otherwise specified in the material specification, all volumetric glassware used in the chemical tests shall be in accordance with Circular 434 on testing of Glass Volumetric Apparatus, issued by the National Bureau of Standards.

4.1.2 The following general laboratory apparatus shall be available. Special apparatus is described in the material specification:

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- 4.1.2.1 Analytical balance and weights.
- 4.1.2.2 Baths, water, steam.
- 4.1.2.3 Beakers.
- 4.1.2.4 Burettes.
- 4.1.2.5 Burners, gas.
- 4.1.2.6 Circulating air oven.
- ~~4.1.2.7 Crucibles.~~
- 4.1.2.8 Crucible tongs.
- 4.1.2.9 Desiccators and desiccants.
- 4.1.2.10 Disks.
- 4.1.2.11 Filter paper.
- 4.1.2.12 Flasks, (Erlenmeyer, suction, volumetric, etc.)
- 4.1.2.13 Funnels.
- 4.1.2.14 Furnace, muffle.
- 4.1.2.15 Graduated cylinder.
- 4.1.2.16 Hot plate.
- 4.1.2.17 Pipets.
- 4.1.2.18 Test tubes.
- 4.1.2.19 Thermometers.
- 4.1.2.20 Watch glasses.

4.2 Reagents.

4.2.1 Unless otherwise specified in the material specification, all reagents used in the chemical tests shall conform to the specifications recommended by the Committee on Analytical Reagents of the American Chemical Society.

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4.2.2 The following general laboratory reagents shall be available: Special reagents are described in the individual methods.

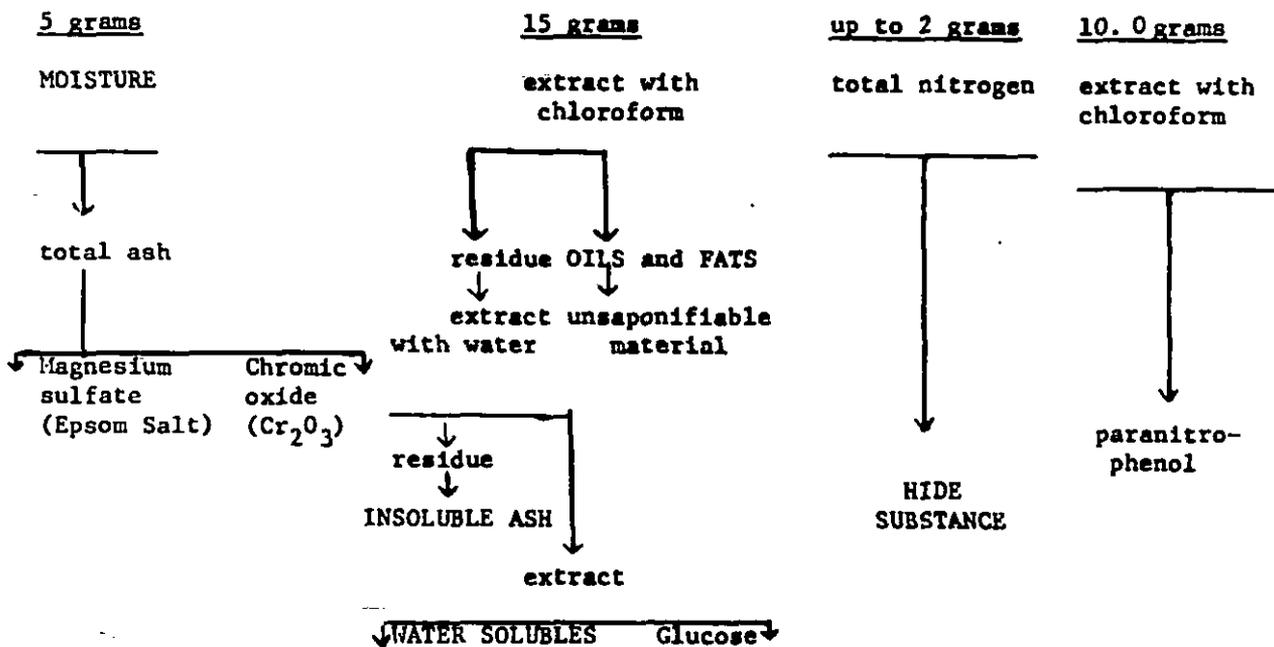
- 4.2.2.1 Acetone.
- 4.2.2.2 Alcohol, ethyl and methyl.
- 4.2.2.3 Ammonium hydroxide, specific gravity 0.91.
- 4.2.2.4 Carbon tetrachloride.
- 4.2.2.5 Chloroform.
- 4.2.2.6 Distilled water.
- 4.2.2.7 Ethyl ether.
- 4.2.2.8 Hydrochloric acid, specific gravity 1.19.
- 4.2.2.9 Nitric acid, specific gravity 1.42.
- 4.2.2.10 Sodium hydroxide.
- 4.2.2.11 Sulfuric acid, specific gravity 1.83.
- 4.2.2.12 Petroleum ether.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the procedures outlined in the individual methods shall be followed. The following flow chart is presented for the convenience of the analyst. It shows the relationship and general separation of the various components that are determined. The sequences listed involve one specimen for more than one determination and have proven to be economical of both time and material. In the chart, the items in capital letters are necessary for the calculation of the degree of tannage.

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5.2 The room temperature referenced throughout Group 6000 shall be $23^{\circ} \pm 5^{\circ}C$.

6. REPORT

6.1 Results of the tests shall be reported as required in detail test methods.

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METHOD 6002
January 15, 1969

PREPARATION OF COMPOSITE SAMPLE

1. SCOPE

1.1 This method is intended for use in preparing a representative composite sample of leather in a form suitable for chemical analysis.

2. COMPOSITE SAMPLE

2.1 The composite sample shall consist of approximately equal portions from each sample unit of the sample for test.

2.2 Cut soles. Scrap obtained in cutting soles, when such cutting is done in the presence of a Government inspector, may be composited, prepared as described in 4.1, and used as the specimen for chemical analysis.

3. APPARATUS

3.1 The apparatus shall be as follows:

3.1.1 Scissors, knife, or other tool.

3.1.2 Balance. The balance shall be capable of weighing to an accuracy of 1 gram.

3.1.3 Wiley mill (or equal).

3.1.4 Sieve. The sieve shall have a 4 millimeter size mesh.

3.1.5 Bottle or other container.

4. PROCEDURE

4.1 Each portion from each sample unit shall be weighed on a balance and adjusted to within 1 gram of each other. All portions taken shall be cut into pieces approximately 1/2 inch square, mixed well, and ground in a Wiley mill (or equal) (see note) until all the material has passed through a 4 millimeter sieve. The ground material shall be mixed well and stored in a tightly stoppered container. Specimens for the tests in group 6000 shall be taken from this composite sample.

NOTE: Utmost care should be utilized in assuring that the mill is thoroughly cleaned prior to use to avoid contamination of the composite sample.

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METHOD 6111

January 15, 1969

ALDEHYDE, QUALITATIVE

1. SCOPE

1.1 This method is intended for determining the presence of aldehyde in chamois leather.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall consist of approximately 0.5 grams of leather from the composite sample (see Method 6002).

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from the composite sample shall be tested.

4. REAGENTS AND METHOD CITED

4.1 Reagents.

4.1.1 Rosaniline hydrochloride solution prepared by dissolving one gram of rosaniline hydrochloride in 500 milliliters of distilled water, and then adding 125 milliliters of a 10 percent solution of sodium sulfite and 10 milliliters of concentrated hydrochloric acid (specific gravity 1.19). The resulting solution shall be filtered, made up to 1 liter volume with distilled water, and stored in an amber bottle. If the solution does not become colorless, it shall be remade using fresh sulfite.

4.1.2 Sodium sulfite, 10 percent solution (12.5 grams anhydrous sodium sulfite dissolved in distilled water and diluted to 125 milliliters).

4.1.3 Hydrochloric acid, 1 volume of acid (specific gravity 1.19) to 10 volumes of distilled water.

4.2 Method cited

4.2.1 Method 6002 - Preparation of Composite Sample.

5. PROCEDURE

5.1 The specimen shall be transferred to a test tube, 7.7 milliliters of 1 to 10 hydrochloric acid added, and the mixture heated to boiling. The test tube and contents shall be cooled to room temperature. One milliliter of rosaniline hydrochloride solution shall then be added, and the mixture set aside for $3 \pm 1/4$ hours at room temperature. The appearance of a grayish-blue color which changes to a violet color during the 3-hour period indicates the presence of aldehyde in the specimen.

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January 15, 1969

6. REPORT

6.1 The presence or absence of aldehyde in the sample for test shall be reported.

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METHOD 6211
January 15, 1967

MOISTURE CONTENT, DRY AIR METHOD

1. SCOPE

1.1 This method is intended for determining the moisture content of leather by utilization of a series of dessicants in conjunction with an oven. In the interest of standardization of testing requirements, it is recommended that this method not be used in procurement documents.

2. TEST SPECIMEN

2.1 The specimen shall consist of approximately 5 grams of leather from the composite sample as described in method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 Two weighing bottles with a 45 milliliter capacity, an inside diameter of 40 millimeters, a height of 50 millimeters, and a tight fitting glass top.

4.1.2 Apparatus for drying air, consisting of a train of suitable towers containing successively (1) sulfuric acid (specific gravity 1.83), (2) anhydrous calcium sulfate (3) anhydrous magnesium perchlorate, and (4) anhydrous calcium sulfate.

4.1.3 An oven capable of maintaining the specified temperature.

4.1.4 Desiccator and desiccant.

4.1.5 Balance accurate to 0.001 gram.

4.2 Method cited.

4.2.1 Method 6002 - Preparation of Composite Sample.

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5. PROCEDURE

5.1 The specimen shall be placed in the weighed bottle, weighed to the nearest 0.001 gram and the weight of the specimen recorded as W_1 . The bottle with the top removed and containing the specimen shall be placed in an oven and maintained at a temperature of $99^{\circ} \pm 1^{\circ}\text{C}$ for a period of $24 \pm 1/4$ hours. During this period, a stream of air shall be passed through the drying train (4.1.2) and into the oven at a rate that will replace the air in the oven approximately once in every 3 hours. At the end of the drying period, the top shall be placed on the bottle and the assembly transferred to the desiccator, cooled to room temperature, immediately weighed, and the weight of the dried specimen recorded as W_2 .

5.2 Calculation of results.

5.2.1 The moisture content of the specimen shall be calculated as follows:

$$\text{Moisture, percent} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 = weight of the original specimen, grams.

W_2 = weight of the dried specimen, grams.

6. REPORT

6.1 The moisture content shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.05 percent. Individual results utilized in obtaining the average shall be reported.

METHOD 6221
January 15, 1969

MOISTURE CONTENT, OVEN METHOD

1. SCOPE

1.1 This method is intended for determining the moisture in leather by drying in a circulating air oven.

2. TEST SPECIMEN

2.1 The specimen shall consist of approximately 5 grams of leather from the composite sample prepared as described in method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 A forced circulating-air oven capable of maintaining the specified temperature.

4.1.2 Two weighing bottles with a 45 milliliter capacity, an inside diameter of 40 millimeters, a height of 50 millimeters and a tight-fitting glass top.

4.1.3 Balance accurate to the nearest 0.005 grams.

4.1.4 Desicca or

4.2 Method cited.

4.2.1 Method 6002- Preparation of Composite Sample.

5. PROCEDURE

5.1 The specimen shall be placed in a weighed bottle, weighed to the nearest 0.005 gram and the weight of the specimen recorded as W_1 . The bottle with the top removed and containing the specimen shall be placed in a circulating air oven and maintained at a temperature of $99^{\circ} \pm 1^{\circ}\text{C}$ for a period of $16 \pm 1/4$ hours. At the end of the drying period, the bottle shall be stoppered, transferred to the desiccator, cooled to room temperature, immediately weighed and the weight of the specimen recorded to the nearest 0.005 gram as W_2 .

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January 15, 1969

5.2 Calculation of results. The moisture content of the specimen shall be calculated as follows:

$$\text{Moisture, percent} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 = weight of the original specimen, grams.

W_2 = weight of the dried specimen, grams.

6. REPORT

6.1 The moisture content shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

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METHOD 6311
January 15, 1969

CHLOROFORM EXTRACTION

1. SCOPE

1.1 This method is intended for determining the amount of materials in leather which are soluble in chloroform.

2. TEST SPECIMEN

2.1 The test specimen shall consist of approximately 4 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHOD CITED

4.1 Apparatus.

4.1.1 Extraction apparatus as shown in figure 6311. The condenser and cover shall be made of glass or block tin or other corrosion resistant metal.

4.1.2 Extraction thimble, size B (22 millimeter diameter, 65 millimeter height, 1 millimeter thick) seamless, fat free: or filter paper, ashless 15 centimeter, No. 40 Whatman or equivalent.

4.1.3 Absorbent cotton.

4.2 Reagents.

4.2.1 Unless otherwise specified, chloroform U S P, that has not been used previously, shall be the solvent for extraction.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6221 - Moisture Content, Oven Method.

5. PROCEDURE

5.1 The specimen shall be weighed to the nearest 0.001 gram and the value recorded as W_2 . The weighed specimen shall be transferred to the extraction thimble or carefully wrapped in filter paper. If a thimble is used, a plug

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of cotton shall be placed on top of the leather. The thimble and leather, or filter paper and leather, shall be placed in the siphon cup and the whole assembly suspended under the condenser.

5.2 The extraction flask shall be dried in an oven for one hour at $100^{\circ} \pm 1^{\circ}\text{C}$, cooled in a desiccator and weighed to the nearest 0.001 gram. Sixty milliliters of chloroform shall be transferred to the flask and the apparatus assembled as shown in figure 6311. Sufficient heat shall be applied to the flask so that the siphon cup will be filled and emptied once every 2 to 3 minutes. After extracting for two hours, the flask shall be removed and the chloroform evaporated on a steam bath. After the solvent has been removed, the flask shall be immediately removed from the steam bath, dried in an oven at $100^{\circ} \pm 1^{\circ}\text{C}$ for 2 hours and then cooled to room temperature in desiccator. The flask and contents shall then be weighed and the weight of the extracted material shall be recorded as W_1 .

5.3 Unless otherwise specified in the material specification, the moisture content of the leather in the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221.

5.4 Calculation of results. The chloroform-soluble material in the specimen shall be calculated as follows:

$$\text{Chloroform-soluble material (moisture-free basis), percent} = \frac{W_1 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Where: W_1 = weight of the chloroform extract, grams

W_2 = weight of the conditioned specimen before extraction, grams

6. REPORT

6.1 The chloroform-soluble material in the specimen shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

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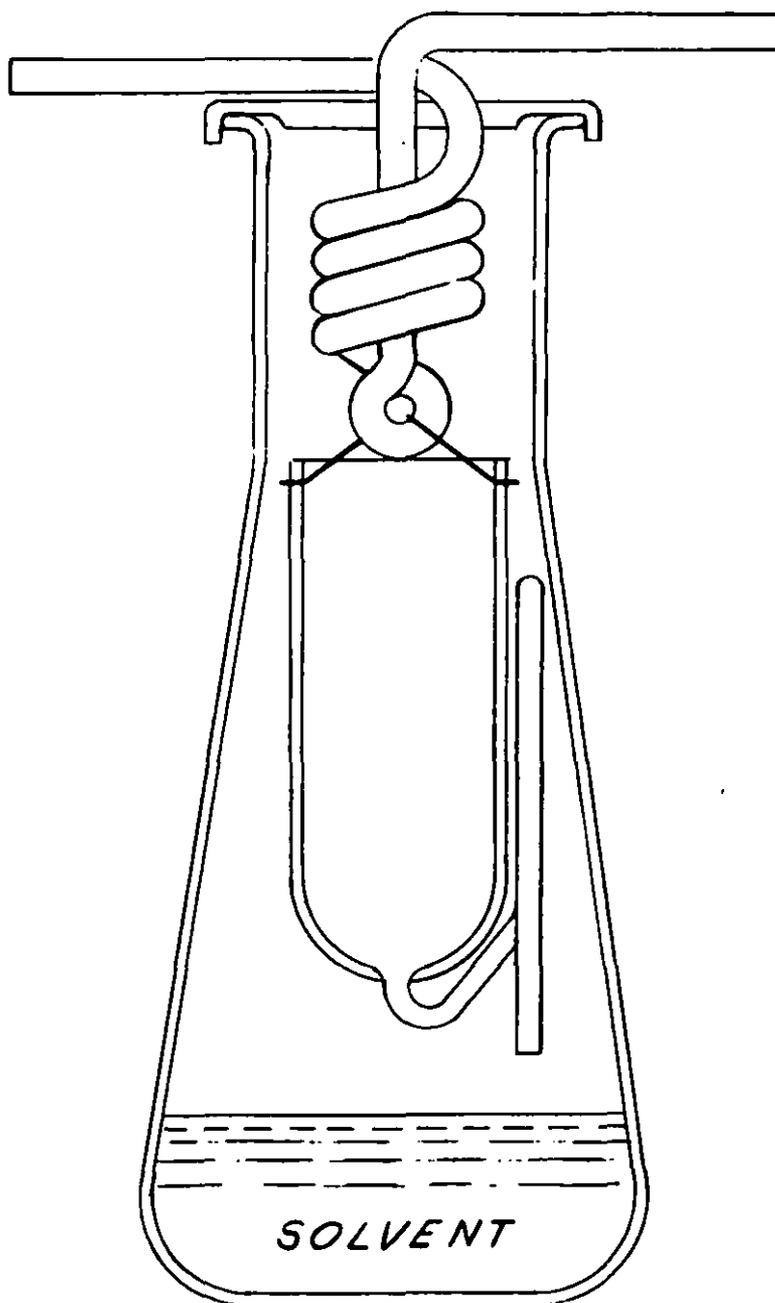


FIGURE 6311

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CHLOROFORM-SOLUBLE AND WATER-SOLUBLE MATERIALS,
AND INSOLUBLE INORGANIC MATERIALS

1. SCOPE

1.1 This method is intended for determining the amount of materials in leather, such as oils and greases, excess and loosely bound tannins, sugar and other non-tannin materials which will be removed by chloroform and/or water extraction or remain as insoluble inorganic materials after the leather has been exposed to high temperature.

2. TEST SPECIMEN

2.1 The specimen for test shall be approximately 10 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus

4.1.1 A forced circulating air oven capable of maintaining the specified temperature.

4.1.2 Soxhlet extraction apparatus and thimbles.

4.1.3 Extraction apparatus as shown in figure 6341.

4.1.3.1 Stirrer for circulating the water bath

4.1.3.2 Heating device capable of maintaining the water bath at the specified temperature.

4.1.3.3 Glass extraction tube of the Reed-Churchill type having an internal diameter of 45 ± 2 millimeters and a body height of 233 ± 10 millimeters.

4.1.3.4 One liter volumetric flask.

4.1.4 Standard tannin dish, 70 millimeters outside diameter by 50 millimeters high, weighing between 30 and 39 grams.

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4.1.5 Large porcelain crucible.

4.1.6 Muffle furnace.

4.2 Reagents.

4.2.1 Unless otherwise specified in the material specification, chloroform USP, that has not been used previously, shall be the solvent for the extraction in 5.2.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6221 - Moisture Content, Oven Method.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimens for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 Chloroform-soluble material. The specimen for test shall be weighed to the nearest 0.001 gram and the value recorded as "O". The weighed specimen shall be loosely packed in an appropriate size thimble and placed in a Soxhlet extraction apparatus. Prior to extraction, the extraction flask shall be dried in a circulating air oven for one hour at $100^{\circ} \pm 1^{\circ}\text{C}$, cooled in a desiccator and weighed to the nearest 0.001 gram. The weighed flask shall be approximately three-fourths filled with chloroform, the apparatus assembled and extraction of the specimen continued for a minimum of 20 complete extractions. At the end of the extraction period, the flask shall be removed and the chloroform driven off. This may be facilitated if the extraction tube is allowed to fill and the chloroform drained off before siphoning back to the extraction flask. When 10 to 20 milliliters of chloroform remain in the flask, the flask shall be removed and the remaining solvent driven off on a steam bath. Removal of the chloroform is facilitated by utilizing a vacuum or a gentle stream of filtered (oil and water free) air. After the chloroform has been removed, the flask containing the extract shall be dried at $100^{\circ} \pm 1^{\circ}\text{C}$ in a circulating air oven to a constant weight (± 0.005 gram) and cooled to room temperature in a desiccator. The weight of the chloroform-soluble material shall be recorded to the nearest 0.001g as "C".

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5.3 Water-soluble material. The extracted leather from 5.2 shall be dried at room temperature and transferred to the Reed-Churchill extraction tube having a glass wool plug or fritted glass disc placed so as to retain the leather material in a tube during extraction. Distilled water shall be added to cover the material in the tube to a depth of approximately 75 millimeters. The tube with the specimen shall be assembled in the apparatus as shown in figure 6341, the temperature of the bath shall be raised to $35^{\circ} \pm 0.5^{\circ}\text{C}$ and the extraction started. The temperature of the bath shall be maintained throughout the extraction. The specimen shall be extracted by adjusting the rate of flow of water through the pinch cock into a 1 liter volumetric flask at such a rate that 1 liter of extract will be collected in 3 hours \pm 10 minutes.

5.3.1 When 1 liter has been collected, the liter flask with contents shall be cooled to room temperature, readjusted up to 1 liter with distilled water and mixed thoroughly. A 100 milliliter aliquot shall be transferred to a tared tannin dish and evaporated to dryness on a steam bath. The residue shall be dried to a constant weight (± 0.005 gram) in a forced circulating air oven at a temperature of $105^{\circ} \pm 5^{\circ}\text{C}$, cooled in a desiccator at room temperature, and immediately weighed to the nearest 0.001 grams. This is the weight of the water-soluble material and shall be recorded as "W".

5.4 Insoluble ash. The extracted leather from 5.3 shall be air dried at room temperature or oven dried and transferred to a large tared porcelain crucible. The crucible and specimen shall be placed in a muffle furnace. The temperature of the oven shall be gradually raised to $600^{\circ} \pm 25^{\circ}\text{C}$ and shall be maintained at this temperature until the crucible and contents have reached a constant weight (± 0.005 gram). After ashing, the crucible and specimen shall be cooled to room temperature in a desiccator and then weighed immediately to the nearest 0.001 gram. This is the weight of the insoluble ash and shall be recorded as "A".

5.5 Calculation of results:

5.5.1 The results, based on the weight of the dry specimen, shall be calculated to the nearest 0.1 percent using the following formulas:

$$\text{Chloroform-soluble material, percent} = \frac{C}{0 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)} \times 100$$

$$\text{Chloroform and water-soluble material, percent} = \frac{(W \times 10) + C}{0 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)} \times 100$$

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$$\text{Water-soluble material, percent} = \frac{(W \times 10)}{O \times \left(\frac{100 - \% \text{ moisture}}{100} \right)} \times 100$$

$$\text{Insoluble ash, percent} = \frac{A}{O \times \left(\frac{100 - \% \text{ moisture}}{100} \right)} \times 100$$

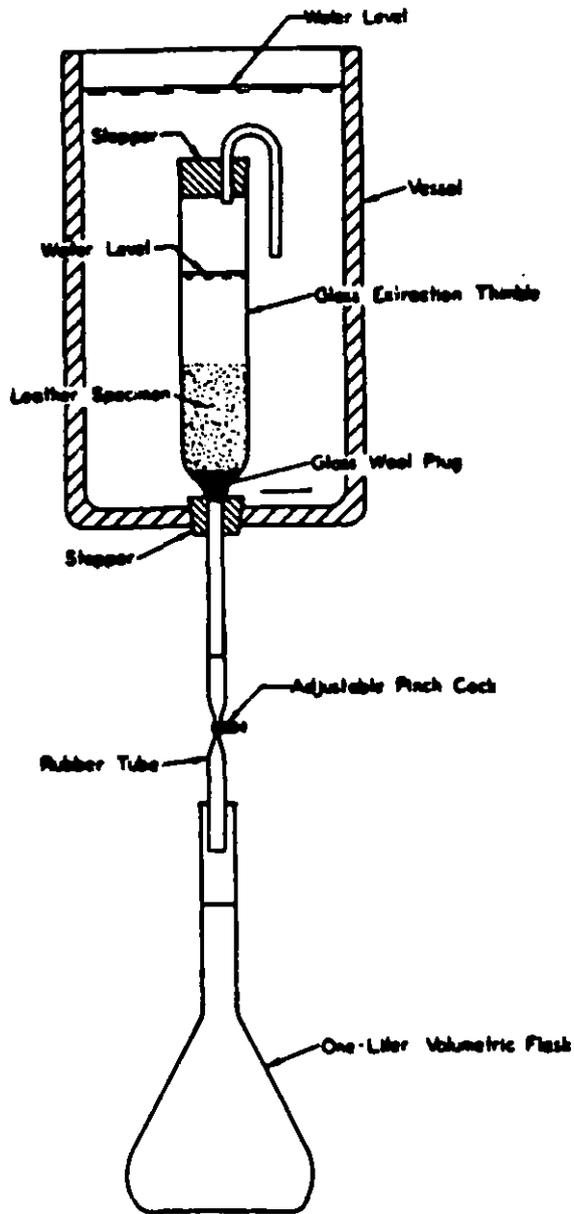
Where: W = weight of water-soluble material, grams
 O = weight of original dry specimen, grams
 C = weight of chloroform-soluble material, grams
 A = weight of insoluble ash, grams

6. REPORT

6.1 Unless otherwise specified in the material specification, the chloroform-soluble material, water-soluble material, and insoluble inorganic material in the specimen shall be the average of the results obtained and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

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Figure 6341

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CHLORIDES, SOLUBLE

1. SCOPE

1.1 This method is intended for quantitatively determining the water-soluble chlorides in leather.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED.

4.1 Apparatus.

4.1.1 Fine porosity filtering crucible or Gooch crucible with asbestos pad.

4.2 Reagents.

4.2.1 Silver nitrate (AgNO_3), 5 grams dissolved in recently boiled distilled water and made up to 100 milliliters.

4.2.2 Nitric acid (HNO_3) - specific gravity 1.42.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6221 - Moisture Content, Oven Method.

4.3.3 Method 6341 - Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 The specimen shall be weighed to the nearest 0.001 gram and the value recorded as W_2 . The specimen shall be extracted with chloroform as described in method 6341 and then dried at $99^\circ \pm 1^\circ\text{C}$ for 1 hour.

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5.2 The dried specimen shall be transferred to a 400 milliliter beaker and 200 milliliters of distilled water at $65^{\circ} \pm 5^{\circ}\text{C}$ added. The mixture shall be allowed to cool at room temperature and set aside, under cover, for 17 ± 1 hours with occasional stirring. The solution shall be filtered through a filter paper into a liter beaker. The residue on the paper shall be washed five times with 10 milliliter portions of distilled water at approximately 50°C adding the washings to the filtrate in the beaker. After washing, the residue shall be returned to the original beaker. The above treatment shall be repeated twice, except that each extraction shall stand for 2 hours instead of 17 hours.

5.3 All the filtrates and washings shall be combined in the liter beaker and acidified by adding 1 milliliter of nitric acid for every 100 milliliters of solution. Ten milliliters of silver nitrate solution shall be added slowly to the solution while stirring. The beaker shall be covered with a watch glass and shielded from direct sunlight. The solution shall be heated to boiling and kept at or near that temperature with occasional stirring until the precipitate settles leaving a clear supernatant liquid. The clear solution shall be tested for chlorides with a few drops of silver nitrate solution. If a precipitate or cloudiness appears, 1 milliliter of silver nitrate shall be added and the heating repeated. This shall be repeated until all the chloride has been precipitated from the solution.

5.4 The solution shall be decanted through a tared chloride free filtering crucible and the precipitate transferred to the crucible by means of hot distilled water. The precipitate shall be washed free from soluble silver salts with hot distilled water. The crucible and contents shall be dried to a constant weight (± 0.005 grams) in a forced circulating air oven at $105^{\circ} \pm 1^{\circ}\text{C}$, cooled to room temperature in a desiccator, weighed to the nearest 0.001 gram and the weight of the precipitate recorded as W_1 .

5.5 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.6 Calculation of results. The water-soluble chlorides in the specimen shall be calculated as sodium chloride as follows:

$$\text{Sodium chloride, percent (moisture-free basis)} = \frac{W_1 \times 40.78}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

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January 15, 1969

Sodium chloride, percent (moisture-free and chloroform extractable-free basis) =

$$\frac{W_1 \times 40.78}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

Where: W_1 = weight of the silver chloride, grams.

W_2 = weight of the original specimen, grams.

40.78 = gravimetric factor.

6. REPORT

6.1 The water soluble chloride as sodium chloride in the specimen shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The water-soluble chloride as sodium chloride in the specimen shall be reported on the moisture-free or moisture-free and chloroform extractable-free basis as specified in the material specification.

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METHOD 6361
January 15, 1969

SULFATES, SOLUBLE

1. SCOPE

1.1 This method is intended for quantitatively determining the soluble sulfates in leather.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 Fine porosity filtering crucible or Gooch crucible with asbestos pad.

4.2 Reagents.

4.2.1 Barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), 10 percent solution (10 grams dissolved in distilled water and diluted to 100 milliliters.)

4.2.2 Hydrochloric acid (HCl), specific gravity 1.19.

4.2.3 Silver nitrate (AgNO_3), 0.85 gram dissolved in distilled water and diluted to 100 milliliters.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6221 - Moisture Content, Oven method.

4.3.3 Method 6341 - Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 The specimen shall be weighed to the nearest 0.001 gram and the value recorded as W_2 . The specimen shall be extracted with chloroform as described in method 6341 and then dried at $100^\circ \pm 1^\circ$ for 1 hour.

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5.2 The dried specimen shall be transferred to a 400 milliliter beaker and 200 milliliters of distilled water at $65^{\circ} \pm 5^{\circ}\text{C}$ added. The mixture shall be covered, allowed to cool to room temperature and set aside for 17 ± 1 hours with occasional stirring. The solution shall then be filtered through filter paper into a liter beaker. The specimen and the paper shall be washed five times with 10 milliliters portions of distilled water at approximately 50°C . Add the washings to the filtrate in the beaker. The washed specimen shall be returned to the original beaker and the treatment shall be repeated twice, except that each extraction shall stand for 2 hours instead of 17 hours.

5.3 All the filtrates and washings shall be combined in the liter beaker, neutralized with hydrochloric acid and 5 milliliters added in excess. The solution shall be heated to boiling and 10 milliliters of barium chloride solution added slowly while stirring. The solution shall be covered with a watch glass, kept warm (38°C) for three hours and then set aside to cool to room temperature.

5.4 The cooled solution shall be decanted through a tared filtering crucible and the precipitate transferred to the crucible by means of hot distilled water. The precipitate shall be washed with hot distilled water until free from chloride as shown by the absence of precipitation when a dilute solution of silver nitrate is added to the filtrate. The crucible with contents shall be dried to a constant weight (± 0.005 gram) in a forced circulating air oven at $105^{\circ} \pm 1^{\circ}\text{C}$, cooled in a desiccator to room temperature, weighed to the nearest .001 gram and the weight of the precipitate recorded as W_1 .

5.5 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for the chemical tests.

5.6 Calculation of results. The soluble sulfates in the specimen shall be calculated as sulfuric acid as follows:

$$\text{Sulfuric acid (H}_2\text{SO}_4\text{), percent (moisture-free basis)} = \frac{W_1 \times 42.02}{W_2 \times \left(\frac{100 - \% \text{ moisture content}}{100} \right)}$$

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Where: W_1 = weight of $BaSO_4$, grams.

W_2 = weight of the original specimen.

42.02 = gravimetric factor.

Sulfuric acid (H_2SO_4), percent (moisture-free and chloroform extractable-free basis) =

$$\frac{W_1 \times 42.02}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

6. REPORT

6.1 The soluble sulfates in the specimen shall be the average of the results obtained from the specimens tested and shall be reported as sulfuric acid to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The soluble sulfates in the specimen shall be reported on the moisture-free or on the moisture-free and chloroform extractable-free basis as specified in the material specification.

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METHOD 6371
January 15, 1969

ACETONE INSOLUBLE MATERIAL

1. SCOPE

1.1 This test is intended for quantitatively determining the acetone-insoluble material in the chloroform extract of impregnated sole leather.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 15 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 Reflux condenser apparatus.

4.1.2 Circulating air oven.

4.2 Reagents.

4.2.1 Acetone, U.S.P.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6341 - Chloroform Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 The chloroform extract shall be determined in a tared flask as described in method 6341 and the weight of the extract recorded as W_1 . Fifty milliliters of acetone shall be added to the dried chloroform-soluble residue from above, the flask connected to a reflux condenser, and the mixture refluxed at the boil for at least 1 hour. After refluxing,

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the mixture shall be allowed to cool to $35^{\circ} \pm 5^{\circ}\text{C}$, and the clear solution decanted off, leaving the undissolved solid in the flask. Fifty milliliters of acetone shall be added and the procedure repeated. After refluxing, the acetone shall be decanted off, and the flask containing the remaining solids shall be placed in a circulating air oven at $99^{\circ} \pm 1^{\circ}\text{C}$ and dried to a constant weight (± 0.005 gram). The residue shall be weighed to the nearest 0.001 gram and recorded as W_2 .

5.2 Calculation. The acetone-insoluble material shall be calculated as follows:

$$\text{Acetone insoluble material, percent} = \frac{W_2}{W_1} \times 100$$

Where: W_1 = weight of the chloroform extract, grams.

W_2 = weight of the residue after extraction with acetone, grams.

6. REPORT

6.1 The acetone-insoluble material in the chloroform extract shall be the average of the results obtained, and shall be reported to the nearest 0.1 percent. Individual results used in obtaining the average shall be reported.

METHOD 6421
January 15, 1969

ASH, TOTAL

1. SCOPE

1.1 This method is intended for determining the total amount of nonvolatile inorganic material in leather.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 2 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS AND METHODS CITED

4.1 Apparatus.

4.1.1 Porcelain crucible, large

4.1.2 Muffle furnace.

4.2 Methods cited.

4.2.1 Method 6002 - Preparation of Composite Sample.

4.2.2 Method 6221 - Moisture Content, Oven method.

4.2.3 Method 6341 - Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

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January 15, 1969

5.2 When calculation on a chloroform extractable-free basis is specified, the specimen shall be extracted with chloroform as described in method 6341.

5.3 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram, and the value recorded as W_2 . The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^\circ + 25^\circ\text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^\circ + 25^\circ\text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained ($+ 0.01$ gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight ($+ 0.01$ gram) has been obtained. The weight of the ash shall be recorded as W_1 .

5.4 Calculation of Results. The total ash in the specimen shall be calculated as follows:

$$\text{Total ash, percent (moisture-free basis)} = \frac{W_1 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture content}}{100} \right)}$$

Where: W_1 = weight of the ash.

W_2 = weight of the original specimen.

Total ash, percent (moisture-free and chloroform extractable free basis) =

$$\frac{W_1 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

6. REPORT

6.1 The total ash in the specimen shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The total ash in the specimen shall be reported on the moisture-free basis or moisture-free and chloroform extractable-free basis as specified in the material specification.

METHOD 6431
January 15, 1969

ALKALI-SOLUBILITY OF LEATHER-CELLULOSE MIXTURES

1. SCOPE

1.1 This method is intended for quantitatively determining the alkali-soluble content of leather-cellulosic combinations, such as shank board, counter and heel-base composition materials. The alkali-soluble materials include leather, glue, and other non-cellulosic organic materials and inorganic compounds.

2. TEST SPECIMENS

2.1 The test specimen shall consist of 4.0 ± 0.3 grams of material prepared from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 Gooch crucibl. with asbestos pad.

4.2 Reagents.

4.2.1 Sodium hydroxide solution (10 grams sodium hydroxide in 1000 milliliters of distilled water).

4.2.2 Acetic acid, 2 percent solution (2 milliliters 56 percent acetic acid in 54 milliliters distilled water).

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite Sample.

4.3.2 Method 6221 - Moisture Content, Oven Method.

4.3.3 Method 6341 - Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

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5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 The weighed specimen shall be extracted with chloroform as described in method 6341, transferred to a tared weighing bottle and dried in a circulating air oven at $100^{\circ} + 1^{\circ}\text{C}$ for 2 hours. The weight of the dried specimen shall be recorded to the nearest 0.1 gram as W_1 .

5.3 The dried sample shall be removed from the weighing bottle and placed in a 600 milliliter beaker containing 200 milliliters of sodium hydroxide solution. The beaker shall be covered with a watch glass and the solution heated at a mild boil for 1 hour.

5.4 The solution shall then be filtered through a weighed Gooch crucible and asbestos pad. The residue shall be washed 4 times (twice with 50 milliliters of hot water, once with 50 milliliters of 2 percent acetic acid, and once with 50 milliliters of hot water) and then dried in a circulating air oven at $100^{\circ} + 1^{\circ}\text{C}$ to a constant weight. The specimen shall be cooled to room temperature in a desiccator and weighed to the nearest 0.1 gram. The value shall be recorded as W_2 .

5.5 Calculation of results. The alkali-solubility of the sample shall be calculated as follows:

$$\text{Alkali-solubility, percent} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 = weight of the chloroform extracted and dried cellulosic and leather specimen.

W_2 = weight of the specimen after alkali digestion.

6. REPORT

6.1 The alkali-soluble material of the sample for test shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 1.0 percent. Individual results utilized in obtaining the average shall be reported.

METHOD 6441
January 15, 1969

NITROGEN, COLLAGENOUS AND HIDE SUBSTANCE

1. SCOPE

1.1 This method is intended for quantitatively determining the collagenous nitrogen and hide substance in leather. Leather is occasionally treated with nitrogenous substances that will interfere with the accurate determination of collagenous nitrogen as described in this method. Nitrogen may be added to the leather through the use of certain resins, polymers, finishes, loading materials, and tanning materials.

2. TEST SPECIMEN

2.1 The specimen for test shall be approximately 0.5 to 2.0 grams of leather (see table I) from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Kjeldahl flask, 500 or 800 milliliter capacity.

4.1.2 Glass beads or other anti-bumping agents.

4.1.3 Condenser equipped with delivery tube and bulb trap (fitted with rubber stopper). The condenser tube may be made of pyrex glass, block tin, or other material which has been shown not to react with hot dilute ammonia solution. The lower end of the delivery tube shall have an opening of about 3 millimeters. To insure against any carry-over of nonvolatile alkali with the vapors, the trap shall be properly baffled. A trap employing a metal screen in the lower tube has been found to be most efficient in scrubbing the distilled vapors (Journal of the Association of Official Agricultural Chemist 31, 432 (1948)).

4.2 Reagents.

4.2.1 Digestion catalyst, mixture consisting of 7 grams of yellow-powdered mercuric oxide and 100 grams of anhydrous sodium or potassium sulfate.

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January 15, 1969

4.2.2 Sulfuric acid, specific gravity 1.83.

4.2.3 Methylene blue indicator, reagent grade, 0.1 percent solution in 95 percent ethyl alcohol.

4.2.4 Sodium hydroxide, approximately 0.1N solution, standardized (4.5 grams of 98 percent sodium hydroxide pellets dissolved in water to make 1 liter).

4.2.5 Sodium hydroxide, approximately 11 N solution (450 grams of 98 percent sodium hydroxide pellets dissolved in water to make 1 liter.)

4.2.6 Sodium thiosulfate, 8 percent solution (80 grams of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ per liter of distilled water).

4.2.7 Sulfuric acid solution standardized (see table I).

4.2.8 Boric acid indicator solution, 43 grams of H_2BO_3 (free from borax), 6 milliliters of methyl red indicator, and 4 milliliters of methylene blue indicator per liter of freshly distilled water.

4.3 Methods cited.

4.3.1 Method 6002 - Preparation of Composite sample.

4.3.2 Method 6221 - Moisture content, Oven method.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 The specimen for test shall be weighed to the nearest 0.0001 gram and the value recorded as W. The specimen shall be transferred to a Kjeldahl digestion flask. Digestion catalyst and sulfuric acid (specific gravity 1.83) shall be added to the flask in the amounts indicated in table I for the applicable specimen weight. The contents of the flask shall be shaken until all of the specimens are wet by the acid and then digested over a flame or electric heater until the organic matter is oxidized. The digestion temperature shall be maintained at low heat at first to prevent excess spattering and foaming (a piece of paraffin wax may be added to reduce foaming). After the foaming has ceased and the mixture cleared, it shall be digested at full heat for the minimum time indicated in table I

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and until digestion is complete. After the digestion is complete, the flask shall be removed from the heat, cooled to room temperature and diluted to approximately 300 milliliters with distilled water. Twenty-five milliliters of 8 percent sodium thiosulfate solution shall be added and the flask set aside for 5 to 10 minutes with occasional stirring.

TABLE I

Specimen weight (grams)	Digestion catalyst (grams)	H ₂ SO ₄ sp.gr. 1.83 (milliliters)	Std. H ₂ SO ₄ (normality)	NaOH 11 Normal (milliliters)	Boric Acid indicator solution (milliliters)	Time of digestion, minimum (hours)
0.5	8	20	0.12	80	50	1
1.5	10	25	0.25	100	100	1.5
2.0	12	30	0.36	120	125	2.0

5.3 A measured volume of boric acid indicator solution, as indicated in table I, shall be transferred to a 500 milliliter receiving flask. The flask shall be placed under the condenser tube so that the lower end of the delivery tube dips just below the surface of the boric acid indicator solution.

5.4 Glass beads or other anti-bumping agent shall be added to the Kjeldahl flask containing the digested specimen to prevent bumping during distillation. An amount of 11 N sodium hydroxide solution as specified in table I, shall be added by slowly pouring the solution down the side of the flask so that it will not mix immediately with the acid. The flask shall be connected immediately with the bulb trap attached to the condenser. The rubber stopper on the bulb shall be wetted with water and forced tightly into the neck of the flask so as to prevent, as far as possible, any gaps between the stopper and the flask wall. The flask shall be shaken to mix the contents thoroughly, placed over a gas flame or heater, and distilled until all the ammonia has passed over into the receiving flask. This usually requires about 150 milliliters of distillate. During the last few minutes of distillation, the receiving flask shall be lowered so that the tip of the delivery tube is above the surface of the liquid in order to permit any solution adhering to the tube to be washed into the flask. At the end of the distillation, the outside of the lower end of the delivery tube shall be washed into the flask with distilled water. During the distillation period, the temperature of the boric acid solution shall be kept below 40°C by shielding the receiving flask if necessary.

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5.5 The solution in the receiving flask shall be made to approximately 250 milliliters for a 0.5 gram specimen, 300 milliliters for a 1.5 gram specimen, and 350 milliliters for a 2.0 gram specimen, with distilled water. The ammonia in the solution shall then be titrated with standard sulfuric acid solution of the normality specified in table I. During the titration, the color changes from a green to a red-violet end point, the intensity of the color increasing with further addition of acid.

5.6 A blank determination shall be made using all the reagents, adding a small amount of sugar to reduce any nitrates present and following the procedure for the size specimen used. If the blank solution is alkaline, it shall be titrated to the red-violet end point and the volume of standard acid required subtracted from that required to titrate the ammonia in the specimen. If the blank solution is acid (red-violet), sufficient standard sodium hydroxide solution shall be added to turn the color green and then the solution back titrated with standard acid solution. In this case, the milliliters of the standard acid required to titrate the blank (the difference between the milliliters of standard acid and the milliliters of standard sodium hydroxide corrected for normality) shall be added to that required to titrate the ammonia in the specimen.

5.7 Calculation of results.

5.7.1 The collagenous nitrogen in the specimen shall be calculated as follows:

Collagenous nitrogen (moisture-free basis), percent =

$$\frac{A \times N \times 0.014 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Where: A = milliliters of standard acid (corrected for blank) required to titrate the specimen.

N = normality of the standard acid.

W = the weight of the specimen in grams.

5.7.2 The hide substance in the specimen shall be calculated as follows:

Hide substance (moisture-free basis), percent =

$$\text{Percent collagenous nitrogen} \times 5.62$$

200

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6. REPORT

6.1 The collagenous nitrogen in the specimen shall be the average of the test results obtained from the specimens tested, and shall be reported to the nearest 0.01 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The hide substance in the specimen for test shall be reported to the nearest 0.1 percent.

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METHOD 6452
January 15, 1969

NITROGEN, WATER EXTRACTABLE

1. SCOPE

1.1 This method is intended for quantitatively determining the water extractable nitrogen in leather.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be approximately 15 grams of the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite specimen shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 Kjeldahl flask, 800 milliliters capacity.

4.1.2 Glass beads or other anti-bumping agents.

4.1.3 Condenser equipped with delivery tube and bulb trap (fitted with rubber stopper). The condenser tube shall be made of pyrex glass, block tin, or other material which has been shown not to react with hot dilute ammonia solution. The lower end of the delivery tube shall have an opening of about 3 millimeters. To insure against any carry-over of non-volatile alkali with the vapors, the trap shall be properly baffled. A trap employing a metal screen in the lower tube has been found to be most efficient in scrubbing the distilled vapors (Journal of the Association of Official Agricultural Chemist, 31 432 (1948)).

4.2 Reagents.

4.2.1 Digestion catalyst, mixture consisting of 7 grams of yellow-powdered mercuric oxide and 100 grams of anhydrous sodium or potassium sulfate.

4.2.2 Sulfuric acid, specific gravity 1.83.

4.2.3 Boric acid indicator solution, 43 grams H_3BO_3 (free from borax), 6 milliliters of methyl-red indicator, and 4 milliliters of methylene blue indicator per liter of freshly distilled water. The solution shall be preserved in a pyrex glass container.

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4.2.3.1 Methyl-red indicator, 0.1 percent solution in 95 percent ethyl alcohol.

4.2.3.2 Methylene blue indicator, 0.1 percent solution in 95 percent ethyl alcohol.

4.2.4 Sodium hydroxide, approximately 0.1N solution, standardized (4.5 grams of 98 percent sodium hydroxide pellets dissolved in water to make 1 liter).

4.2.5 Sodium hydroxide: Approximately 11N solution (450 grams of 98 percent sodium hydroxide pellets dissolved in water to make 1 liter).

4.2.6 Sodium thiosulfate, 8 percent solution (80 grams of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ per liter of distilled water).

4.2.7 Sulfuric acid solution, approximately 0.12N, standardized (6.39 grams sulfuric acid, specific gravity 1.83) diluted to 1 liter with distilled water.

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221.

5.2 The specimen shall be weighed to the nearest 0.005 gram and extracted with chloroform and water as described in method 6341.

5.3 The extract from 5.2 shall be cooled to room temperature, made up to a 1000 milliliters volume with distilled water, thoroughly mixed, and filtered. Five-hundred milliliters of the solution shall be transferred to an 800 milliliter Kjeldahl flask. Eight grams of digestion catalyst and 20 milliliters of sulfuric acid (specific gravity 1.83) shall be added to the solution and the contents of the flask mixed thoroughly.

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5.4 The mixture shall be digested over a low heat source until all organic matter is oxidized as indicated by the end of foaming and clearing of the solution. When foaming has ceased and the solution has cleared, full heat shall be applied to the mixture and digestion continued for a minimum of 1 hour. After digestion is complete, the flask shall be removed from the heat, and allowed to cool to room temperature. Twenty-five milliliters of 8 percent sodium thiosulfate shall then be added and the flask set aside for 5 to 10 minutes with occasional stirring.

5.5 Fifty milliliters of boric acid indicator shall be placed in a 500 milliliter receiving flask and the flask placed under the condenser tube, so that the lower end of the delivery tube dips just below the boric acid indicator solution.

5.6 Glass beads or other anti-bumping agent shall be added to the Kjeldahl flask containing the digested specimen to prevent bumping during distillation. Eighty milliliters of 11N sodium hydroxide solution shall be added by slowly pouring the solution down the side of the flask so that it will not mix immediately with the acid. The flask shall be connected immediately with the bulb trap attached to the condenser. The rubber stopper on the bulb shall be wetted with water and forced tightly into the neck of the flask in order to prevent, as far as possible, any gaps between the stopper and flask wall. The flask shall be shaken to mix the contents thoroughly, placed over a gas flame or heater, and distilled until all the ammonia has passed over into the receiving flask. This usually requires about 150 milliliters of distillate. During the last few minutes of distillation, the receiving flask shall be lowered so that the tip of the delivery tube is above the surface of the liquid in order to permit any solution adhering to the tube to be washed into the flask. At the end of the distillation, the outside of the lower end of the delivery tube shall be washed into the flask with distilled water. During the distillation period, the temperature of the boric acid solution shall be kept below 40°C, by shielding the receiving flask if necessary.

5.7 The solution in the receiving flask shall be made to approximately 250 milliliters with distilled water. The ammonia in the solution shall then be titrated with 0.12N sulfuric acid. During the titration, the color changes from green to a red-violet end point.

5.8 A blank determination shall be made using all the reagents, adding a small amount of sugar to reduce any nitrates present. If the blank solution is alkaline, it shall be titrated to the red-violet end-point and the volume of standard acid required subtracted from that required to

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titrate ammonia in the specimen. If the blank solution is acid (red-violet), sufficient standard sodium hydroxide solution shall be added to return the color to green, and the solution back titrated with standard acid solution. In this case, the milliliters of standard acid required to titrate the blank (the difference between the milliliters of standard acid and the milliliters of standard sodium hydroxide corrected for normality) shall be added to that required to titrate the ammonia in the specimen.

5.9 Calculation of results.

5.9.1 The water extractable nitrogen in the specimen shall be calculated as follows:

Water extractable nitrogen (moisture-free basis), percent =

$$\frac{A \times N \times 0.014 \times 2 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Where: A = milliliters of standard acid (corrected for blank) required to titrate the specimen.

N = normality of the standard acid.

W = weight of the specimen, grams.

6. REPORT

6.1 The water extractable nitrogen in the specimen shall be the average of the results obtained from the specimens tested, and shall be reported to the nearest 0.01 percent. Individual results utilized in obtaining the average shall be reported.

METHOD 6511
January 15, 1969

CHROMIC OXIDE, FUSION METHOD

1. SCOPE

1.1 This method is intended for quantitatively determining the chromic oxide in leather. This method is desirable for use where a total ash content is required.

2. TEST SPECIMEN

2.1 The specimen shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Platinum crucible.

4.1.2 Iodine flasks.

4.1.3 Muffle furnace.

4.2 Reagents.

4.2.1 Fused powdered borax.

4.2.2 Potassium carbonate, anhydrous.

4.2.3 Potassium iodide solution (10 grams potassium iodide dissolved in distilled water and diluted to 100 milliliters), iodate free.

4.2.4 Sodium carbonate.

4.2.5 Sodium thiosulfate, 0.1N solution, standardized (dissolve 25 grams $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water, add 1 gram of Na_2CO_3 and dilute to 1 liter).

4.2.6 Starch indicator solution (make a paste of 2 grams of soluble starch in about 10 milliliters of distilled water, add 90 milliliters of distilled water and boil for one minute with stirring, cool, and add one drop of chloroform.)

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4.2.7 Hydrochloric acid, specific gravity 1.19.

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

4.3.4 Method 6441. Nitrogen, Collagenous and Hide Substance.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimens for moisture content shall be determined at the same time under the same ambient conditions as the specimens weighed for chemical test.

5.2 When calculation on a chloroform-extractable-free basis is specified, the specimen shall be extracted with chloroform as described in method 6341.

5.3 When calculation on a hide substance basis is specified, the hide substance shall be determined as described in method 6441.

5.4 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram, and the value recorded as W. The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^{\circ} + 25^{\circ}\text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^{\circ} + 25^{\circ}\text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained. (+ 0.01 gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight (\pm 0.01 gram) has been obtained.

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5.5 Six grams of a fusion mixture consisting of 2 grams of sodium carbonate, 2 grams of potassium carbonate, and 2 grams of fused powdered borax shall be added to the ash in the crucible. The ash and fusion compound shall be mixed well with a platinum wire and then fused for at least 30 minutes over an oxidizing flame. The fused mass shall be cooled, transferred to a 250 milliliter beaker and dissolved in hot distilled water. The solution shall be filtered into a 400 milliliter beaker and the paper and residue washed with hot distilled water adding washings to the filtrate. If any chrome residue remains on the paper, it shall be ashed and the fusion repeated with about 1 gram of the fusion compound. The fused mass shall be dissolved in hot distilled water as previously described and the filtrate and washings added to the first solution.

5.6 The solution shall be cooled to room temperature, transferred to a 500 milliliter volumetric flask, made up to a volume of 500 milliliters with distilled water and mixed well. Two hundred milliliters of the solution shall be transferred from the volumetric flask to a 500 milliliter iodine flask, 10 milliliters of hydrochloric acid (specific gravity 1.19) added and the solution cooled again. Ten milliliters of 10 percent potassium iodide solution shall be added, the flask stoppered and set aside in darkness for about 2 minutes. The solution shall then be titrated immediately with 0.1N sodium thiosulfate solution, using starch indicator near the end of the titration.

5.7 Calculation of results. The chromic oxide in the specimen shall be calculated as follows:

Chromic Oxide (Cr_2O_3), percent (moisture-free basis) =

$$\frac{A \times N \times 0.02533 \times 2.5 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Chromic Oxide (Cr_2O_3), percent (moisture-free and chloroform-extractable-free basis) =

$$\frac{A \times N \times 0.02533 \times 2.5 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

Where: A = number of milliliters of standard thiosulfate required to titrate the specimen.

N = normality of the thiosulfate solution.

W = weight of the specimen, grams:

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Chromic Oxide (Cr_2O_3), percent (hide-substance basis) = $\frac{B}{C} \times 100$

Where: B = percent of chromic oxide on the moisture-free basis.

C = percent of hide substance on the moisture-free basis.

6. REPORT

6.1 The chromic oxide in the specimens shall be the average of the results obtained and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The chromic oxide in the specimen shall be reported on the moisture-free basis, on the moisture-free and chloroform extractable-free basis, or on the hide-substance basis as specified in the material specification.

METHOD 6515
January 15, 1969

CHROMIC OXIDE, PERCHLORIC ACID METHOD

1. SCOPE

1.1 This method is intended for quantitatively determining the chromic oxide in leather. This method is more rapid than method 6511, and is desirable for use when a total ash content is required.

2. TEST SPECIMEN

2.1 The specimen shall consist of approximately 2 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Porcelain crucible.

4.1.2 Iodine flasks.

4.1.3 Muffle furnace.

4.2. Reagents.

4.2.1 Perchloric acid, 60 to 72 percent.

4.2.2 Potassium iodide solution (10 grams potassium iodide dissolved in distilled water and diluted to 100 milliliters).

4.2.3 Sodium thiosulfate, 0.1N solution (dissolve 25 grams of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water, add 0.1 gram of Na_2CO_3 and dilute to 1 liter).

4.2.4 Starch indicator solution (make a paste of 1 gram of soluble starch in about 10 milliliters of water, add 90 milliliters of water and boil for one minute with stirring, cool and add one drop of chloroform).

4.2.5 Phosphoric acid, 40 percent (dilute 45 milliliters of 85 percent phosphoric acid with distilled water to 100 milliliters).

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4.2.6 Sulphuric acid, specific gravity 1.83.

4.2.7 Nitric acid, specific gravity 1.42.

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

4.3.4 Method 6441. Nitrogen, Collagenous and Hide Substance.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be in accordance with method 6221. The weight of the composite specimens for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 When calculation on a chloroform-extractable-free basis is specified, the specimen shall be extracted with chloroform as described in method 6341.

5.3 When calculation on a hide substance is specified, hide substance shall be determined on a moisture-free basis as described in method 6441.

5.4 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram, and the value recorded as W. The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^{\circ} \pm 25^{\circ} \text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^{\circ} \pm 25^{\circ} \text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained (± 0.01 gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight (± 0.01 gram) has been obtained.

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5.5 The crucible containing the ash shall be placed sidewise in a 400 milliliter beaker and 15 milliliters of sulfuric acid (specific gravity 1.83), 4 milliliters of nitric acid (specific gravity 1.42), and 10 milliliters of 60 to 72 percent perchloric acid added in order. (Caution: Perchloric acid should never be used without the accompanying use of sulfuric and nitric acids. Improper use of concentrated perchloric acid can lead to violent and serious explosions!) The crucible shall be tipped so that the reagents are in contact with the ash. The beaker shall be covered with a watch glass, the contents heated to white fumes (approximately 190°C) and the heating continued until the solution turns a clear red-orange color. Heat an additional two minutes to insure complete oxidation of chromium. During the heating, any particles adhering to the beaker that are not washed down with condensed acid shall be washed down with additional sulfuric acid. The solution shall be cooled to room temperature and transferred to a 500 milliliter Erlenmeyer flask.

5.6 Distilled water shall be added to make a volume of about 100 milliliters and the solution boiled until all chlorine is removed, as indicated by a negative test when starch-iodide paper is held in the vapor. Do not let the volume go below 75 milliliters. The solution shall be cooled and made up to 100 milliliters with distilled water at room temperature. The solution shall then be transferred to an iodine flask and thirty milliliters of 40 percent phosphoric acid (to complex iron) and ten milliliters of 10 percent potassium iodide solution shall be added. The flask shall be stoppered, shaken by hand and allowed to stand in the dark for 2 minutes with about 10 milliliters of distilled water in the funnel section to retain escaping iodine fumes. After two minutes, the stopper shall be removed allowing the distilled water to enter the flask. The solution shall then be titrated with 0.1N sodium thiosulfate solution, using starch indicator near the end of the titration. The number of milliliters of standard thiosulfate solution required shall be recorded as A.

5.7 Calculation of results. The chromic oxide in the specimen shall be calculated as follows:

Chromic Oxide (Cr_2O_3), percent (moisture-free basis) =

$$\frac{A \times N \times 0.02533 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

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Where: A = number of milliliters of standard thiosulfate solution required to titrate the specimen.

N = normality of the thiosulfate solution.

W = weight of the specimen, grams.

Chromic Oxide (Cr_2O_3), percent (moisture-free and chloroform-extractable-free basis) =

$$\frac{A \times N \times 0.02533 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ extractable}}{100} \right)}$$

Chromic Oxide (Cr_2O_3), percent (hide substance basis) = $\frac{B}{C} \times 100$

Where: B = percent chromic oxide on the moisture-free basis.

C = percent of hide substance on the moisture-free basis.

6. REPORT

6.1 The chromic oxide in the specimen shall be the average of the results obtained and shall be reported to the nearest 0.1 percent. Individual test results utilized in obtaining the average shall be reported.

6.2 The chromic oxide in the specimen shall be reported on the moisture-free basis, moisture-free and chloroform extractable-free basis, or hide substance basis as specified in the material specification.

METHOD 6518
January 15, 1969

CHROMIC OXIDE, WET OXIDATION

1. SCOPE

1.1 This method is intended for quantitatively determining the chromic oxide in leather by oxidation of the unwashed specimen. This method offers a rapid procedure for the determination of chromic oxide in leather and is desirable for use where a total ash content is not required.

2. TEST SPECIMEN

2.1 The specimen for test shall be approximately 2 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. REAGENTS AND METHODS CITED

4.1 Reagents.

4.1.1 Perchloric acid, 60 to 72 percent.

4.1.2 Potassium iodide solution (10 grams potassium iodide dissolved in distilled water and diluted to 100 milliliters).

4.1.3 Sodium thiosulfate, 0.1N solution (dissolve 25 grams of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water, add 1 gram of Na_2CO_3 and dilute to 1 liter).

4.1.4 Starch indicator solution (make a paste of 2 grams of soluble starch in about 10 milliliters of water, add 90 milliliters of water and boil for 1 minute with stirring. Cool and add 1 drop of chloroform).

4.1.5 Phosphoric acid, 40 percent (dilute 45 milliliters of 85 percent phosphoric acid with distilled water to 100 milliliters).

4.1.6 Sulfuric acid, specific gravity 1.83.

4.1.7 Nitric acid, specific gravity 1.42.

4.2 Methods cited.

4.2.1 Method 6002. Preparation of Composite Sample.

4.2.2 Method 6221. Moisture Content, Oven Method.

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4.2.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

4.2.4 Method 6441. Nitrogen, Collagenous and Hide Substance.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the specimen is drawn shall be in accordance with method 6221. The weight of the composite sample for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 When calculation on a chloroform-extractable free basis is specified, the specimen shall be extracted with chloroform as described in method 6341.

5.3 When calculation on a hide substance basis is specified, hide substance shall be determined as described in method 6441.

5.4 The specimen shall be weighed to the nearest .001 gram and transferred to a 500 milliliter Erlenmeyer flask and 15 milliliters of sulfuric acid (specific gravity 1.83), 4 milliliters of nitric acid (specific gravity 1.42), and 10 milliliters of 60 to 72 percent perchloric acid added in order. (Caution: Perchloric acid should never be used without the accompanying use of sulfuric and nitric acids. Improper use of concentrated perchloric acid can lead to violent and serious explosions). The flask shall be covered with a small watch glass and placed with the contents on a hot plate and heated to white fumes (approximately 190°C). The heating shall be continued until the solution turns a deep orange color. Heat an additional 2 minutes to insure a complete oxidation of chromium. During the heating, any particles adhering to the flask that are not washed down with condensed acid shall be washed down with additional sulfuric acid. The solution shall then be cooled to room temperature.

5.5 Distilled water shall be added to make a volume of about 100 milliliters, and the solution boiled until all chlorine is removed, as indicated by a negative test when starchiodide paper is held in the vapor. Do not let the volume fall below 75 milliliters. The solution shall be cooled and made up to 100 milliliters with distilled water at room temperature. The solution shall then be transferred to an iodine flask and 30 milliliters of 40 percent phosphoric acid (to complex iron) added. Ten milliliters of 10 percent potassium iodide solution shall be added.

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The flask shall be stoppered, shaken by hand, and allowed to stand in the dark for 2 minutes with about 10 milliliters of distilled water in the funnel section to retain escaping iodine fumes. After 2 minutes the stopper shall be removed, allowing the distilled water to enter the flask, and the solution titrated with 0.1N sodium thiosulfate solution, using starch indicator solution near the end of the titration.

5.6 Calculation of results. The chromic oxide in the specimen shall be calculated as follows:

$$\begin{aligned} \text{Chromic oxide (Cr}_2\text{O}_3\text{), percent (moisture-free basis)} \\ = \frac{A \times N \times 0.02533 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right)} \end{aligned}$$

Where: A = number of milliliters of standard thiosulfate required to titrate the specimen.

N = normality of the thiosulfate solution.

W = weight of the specimen, grams.

Chromic oxide (Cr₂O₃), percent (moisture-free and chloroform extractable-free basis)

$$\frac{A \times N \times 0.02533 \times 100}{W \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

Chromic oxide (Cr₂O₃), percent (hide substance basis)

$$= \frac{B}{C} \times 100$$

Where: B = percent chromic oxide on a moisture-free basis.

C = percent chromic oxide on a hide substance basis.

6. REPORT

6.1 The chromic oxide in the specimen shall be the average of the results obtained and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The chromic oxide in the specimen shall be reported on the moisture-free basis, moisture-free and chloroform extractable-free basis, or hide substance basis as specified in the material specification.

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January 15, 1969

CHROMIC OXIDE, COLORIMETRIC METHOD

1. SCOPE

1.1 This method is intended for qualitatively determining small amounts of chromic oxide in leather. It is applicable to materials such as catgut ligatures and leather products for which a minimum amount of chromium is specified.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Color comparison tubes or colorimeter.

4.1.2 Platinum crucible.

4.1.3 Muffle furnace.

4.2 Reagents.

4.2.1 Fused powdered borax.

4.2.2 Potassium carbonate, anhydrous.

4.2.3 Potassium dichromate solution (0.06 gram of KCr_2O_3 , and 60 milliliters of a solution made up of 100 milliliters of sulphuric acid (specific gravity 1.83) and 200 milliliters of distilled water).

4.2.4 Sulphuric acid, 50 percent.

4.2.5 Sodium carbonate, anhydrous.

4.2.6 Litmus paper.

4.3 Methods cited.

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4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimens for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram. The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^{\circ} + 25^{\circ}\text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^{\circ} + 25^{\circ}\text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained. (± 0.01 gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight (± 0.01 gram) has been obtained.

5.3 Six grams of a fusion mixture consisting of 2 grams of sodium carbonate, 2 grams of potassium carbonate, and 2 grams of fused powdered borax shall be added to the ash in the crucible. The ash and fusion compound shall be mixed well with a platinum wire and then fused for at least 30 minutes over an oxidizing flame. The fused mass shall be cooled, transferred to a 400 milliliter beaker and dissolved in 200 milliliters of distilled water. The solution shall be boiled for about 5 minutes, cooled and filtered into a 250 milliliter volumetric flask. The solution shall be neutralized with 50 percent sulfuric acid, using litmus-paper indicator, and 5 milliliters excess acid added. The solution shall be cooled to room temperature, made up to 250 milliliters with distilled water at room temperature and mixed well.

5.4 Fifty milliliters of the solution shall be transferred to a colorimetric tube or other suitable tube of uniform dimensions. Fifty milliliters of the standard dichromate solution shall be transferred to

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another tube of the same dimensions. A comparison of the color intensity of the two solutions shall be made by placing the bottoms of the tubes against a white background and looking through the solutions lengthwise from the top. If available, the colorimeter may be used for making the color comparison. A 5 gram specimen containing the equivalent of 0.3 percent Cr_2O_3 will give a dichromate color equal in intensity to that of the standard dichromate solution. A lighter shade in the test solution indicates less than, and a darker shade indicates more than 0.3 percent chromic oxide.

6. REPORT

6.1 The chromic oxide in the specimen shall be reported as pass or fail.

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METHOD 6531
January 15, 1969

IRON AND ALUMINUM OXIDES

1. SCOPE

1.1 This method is intended for quantitatively determining the iron and aluminum oxides in leather. These compounds may be present as tanning agents or as impurities from other inorganic salt treatments in the preservation or tanning of leather. The presence of titanium or zirconium will cause erroneous results since they are interfering agents and will be carried down in the precipitation.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Platinum crucible.

4.1.2 Muffle furnace.

4.2 Reagents.

4.2.1 Ammonium chloride.

4.2.2 Methyl red indicator, 0.02 percent solution in 60 milliliters ethyl alcohol and 40 milliliters of distilled water.

4.2.3 Potassium carbonate, anhydrous.

4.2.4 Sodium carbonate.

4.2.5 Ammonium hydroxide (1:1 solution).

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

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4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 When calculation on a chloroform extractable-free basis is specified, the specimen shall be extracted with chloroform as described in method 6341.

5.3 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram, and the value recorded as W_2 . The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^{\circ} + 25^{\circ} \text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^{\circ} + 25^{\circ} \text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained (± 0.01 gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight (± 0.01 gram) has been obtained.

5.4 Four grams of a fusion mixture, containing 2 grams of potassium carbonate and 2 grams of sodium carbonate shall be added to the ash in the crucible. The ash and fusion compound shall be mixed well with a platinum wire and then fused over an oxidizing flame for at least 30 minutes. The fused mass shall be transferred to a 400 milliliter beaker, dissolved in hot distilled water and made acid with hydrochloric acid. If the solution is not clear, it shall be filtered into a 400 milliliter beaker and the residue washed with distilled water adding the washings to the filtrate. If any residue remains on the paper, it shall be ashed and the fusion repeated, using only 1 gram of the fusion mixture. The fused mass shall be dissolved, filtered and washed as previously described, and the filtrate and washings added to the first solution.

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5.5 The solution shall be made up to 300 milliliters with distilled water, and 8 grams of ammonium chloride and a few drops of methyl red indicator shall be added. The solution shall be heated to boiling and ammonium hydroxide (1:1) added dropwise until the solution is distinctly yellow. This solution shall be boiled for about two minutes and filtered immediately. The precipitate shall be washed thoroughly with hot 2 percent ammonium chloride, dissolved in hot hydrochloric acid, reprecipitated, filtered and washed as previously described.

5.6 The filter paper and precipitate shall be transferred to a weighed platinum crucible and placed in a cool muffle furnace. The temperature of the furnace shall be raised to 925 C where heating shall be continued until the crucible and contents reach a constant temperature. The crucible and residue shall be cooled in a desiccator to room temperature, immediately weighed and the residue weight recorded as W_1 .

5.7 Calculation of results. The iron and aluminum oxides (Fe_2O_3 and Al_2O_3) in the specimen shall be calculated as follows:

Iron and aluminum oxides (Fe_2O_3 and Al_2O_3), percent (moisture-free basis)

$$= \frac{W_1 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Iron and aluminum oxides (Fe_2O_3 and Al_2O_3), percent (moisture-free and chloroform extractable-free basis)

$$= \frac{W_1 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right) \times \left(\frac{100 - \% \text{ chloroform extractable}}{100} \right)}$$

Where: W_1 = weight of the residue, grams.

W_2 = weight of the specimen, grams.

6. REPORT

6.1 The iron and aluminum oxides in the specimen shall be the average of the results obtained and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

6.2 The iron and aluminum oxides in the specimen shall be reported on the moisture-free basis or moisture free and chloroform-extractable free basis as specified.

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 225/226

METHOD 6541
January 15, 1969

MAGNESIUM SULFATE (EPSOM SALT)

1. SCOPE

1.1 This method is intended for quantitatively determining the magnesium sulfate (Epsom Salt) in leather.

2. TEST SPECIMEN

2.1 The specimen for test shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Platinum crucible.

4.1.2 Filtering crucible.

4.1.3 Suction flask.

4.1.4 Muffle furnace.

4.2 Reagents.

4.2.1 Hydrochloric acid, specific gravity 1.19.

4.2.2 Nitric acid, specific gravity 1.42.

4.2.3 Ammonium chloride.

4.2.4 Ammonium hydroxide, 1 to 1 and 1 to 9 parts by volume with distilled water.

4.2.5 Ammonium nitrate solution (10 grams dissolved in distilled water and made up to 100 milliliters).

4.2.6 Ammonium oxalate (saturated solution).

4.2.7 Ferric chloride solution (10 grams dissolved in distilled water and made up to 100 milliliters).

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- 4.2.8 Methyl orange indicator (0.05 percent).
- 4.2.9 Sodium ammonium hydrogen phosphate (saturated solution).
- 4.3 Methods cited.
 - 4.3.1 Method 6002. Preparation of Composite Sample.
 - 4.3.2 Method 6221. Moisture Content, Oven Method.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn, shall be determined in accordance with method 6221. The weight of the composite specimens for moisture content shall be determined at the same time and under the same ambient conditions as the specimens weighed for chemical tests.

5.2 The specimen shall be placed in a tared porcelain crucible, weighed to the nearest 0.001 gram, and the value recorded as W_2 . The weighed specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^\circ + 25^\circ\text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^\circ + 25^\circ\text{C}$ for 30 minutes and repeat the cooling and weighing procedure until a constant weight is obtained ($+ 0.01$ gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight ($+ 0.01$ gram) has been obtained.

5.3 The ash shall be moistened with distilled water and 15 milliliters of hydrochloric acid (specific gravity 1.19) shall be added to the crucible. The contents of the crucible shall be transferred to a 400 milliliter beaker and diluted to about 60 milliliters with distilled water. Two drops of nitric acid (specific gravity 1.42) shall be added and the solution heated on a steam bath for about 15 minutes. Ammonium hydroxide (1:1) shall be added dropwise, while stirring, until the solution is slightly alkaline to methyl orange or a faint odor of ammonia appears at this point. If the precipitate does not have the characteristic reddish-brown color of ferric hydroxide, the precipitate shall be dissolved in hydrochloric acid, a few drops of ferric chloride solution added, and the addition of a slight excess of ammonium hydroxide repeated. The contents of the beaker shall be boiled for a few minutes, filtered and washed free from chlorides with hot water, catching the filtrate and washings in a 400 milliliter beaker.

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5.4 Three grams of ammonium chloride shall be added and the solution evaporated to about 175 milliliters; one milliliter of ammonium hydroxide (1:1) shall be added and the solution heated to boiling. Ten milliliters of saturated ammonium oxalate solution shall be added slowly while stirring, the beaker shall then be covered and set aside over a steam bath for at least 2 hours. The solution and precipitate shall be transferred to a 250 milliliter volumetric flask, cooled to room temperature, diluted to the mark and mixed well. The solution shall be filtered through a dry quantitative filter paper.

5.5 An aliquot of 50 milliliters shall be transferred to a beaker, diluted to about 150 milliliters with distilled water, a few drops of methyl orange indicator added, and hydrochloric acid added until slightly acid. A slight excess of saturated sodium ammonium hydrogen phosphate over that necessary to precipitate the magnesium shall be added (usually 5 milliliters is sufficient). While stirring vigorously, the solution shall be made slightly ammoniacal by the dropwise addition of ammonium hydroxide (1:1) and set aside for 15 minutes. Five milliliters of ammonium hydroxide (1:1) shall be added while stirring and the solution set aside overnight at room temperature.

5.6 The solution shall be filtered through a filtering crucible, the precipitate transferred to the platinum crucible and washed free from the chlorides with ammonium hydroxide (1:9) solution. The precipitate shall be moistened with a few drops of 10 percent ammonium nitrate solution, dried cautiously, ignited to a constant weight (± 0.001 gram) at 1000° to $1100^{\circ}\text{C}.$, cooled in a desiccator to room temperature, immediately weighed and the value recorded as W_1 .

5.7 Calculation of results. The magnesium sulfate (Epsom Salt, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in the specimen shall be calculated as follows:

Magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), percent (moisture-free basis)

$$= \frac{W_1 \times 2.2147 \times 5 \times 100}{W_2 \times \left(\frac{100 - \% \text{ moisture}}{100} \right)}$$

Where: W_1 = weight of the ignited precipitate ($\text{Mg}_2\text{P}_2\text{O}_7$), grams.

W_2 = weight of the specimen, grams.

6. REPORT

6.1 The magnesium sulfate in the specimen shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

052/622

METHOD 6551
January 15, 1969

LEAD SALTS, PRESENCE OF

1. SCOPE

1.1 This method is intended for qualitatively determining the presence of lead salts in leather.

2. TEST SPECIMEN

2.1 The specimen shall consist of approximately 5 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from the composite sample shall be tested.

4. APPARATUS, REAGENTS AND METHODS CITED

4.1 Apparatus.

4.1.1 Muffle furnace.

4.1.2 Porcelain crucible.

4.2 Reagents.

4.2.1 Nitric acid, 1:1 dilution (equal volumes of nitric acid and distilled water).

4.2.2 Hydrochloric acid (specific gravity 1.19).

4.2.3 Ammonium acetate 3N (231 grams of ammonium acetate dissolved in distilled water to make one liter).

4.2.4 Potassium chromate 5 percent solution (50 grams potassium chromate dissolved in distilled water to make 1 liter).

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

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5. PROCEDURE

5.1 The specimen shall be placed in a tared porcelain crucible. The specimen shall be placed in a cold muffle furnace or precarbonized over a burner prior to placing in a hot furnace. The temperature of the furnace shall gradually be raised to $600^{\circ} + 25^{\circ}\text{C}$ and maintained at this temperature for 60 minutes. Remove the crucible and contents, cool in a desiccator and weigh. Replace in the furnace at $600^{\circ} + 25^{\circ}\text{C}$ for 30 minutes and repeat the weighing procedure until a constant weight is obtained (± 0.01 gram). If it is difficult to obtain a constant weight, the residue shall be leached with hot distilled water and filtered through an ashless filter paper. The filter paper shall be placed in the crucible and ashed. The filtrate shall be added to the crucible and evaporated. The crucible shall then be put back in the muffle furnace and heated, cooled and weighed as above until a constant weight (± 0.01 gram) has been obtained.

5.2 After the crucible containing the ash has cooled, 5 milliliters of nitric acid (1:1) shall be added and the mixture carefully evaporated to dryness. One milliliter of concentrated hydrochloric acid shall then be added and evaporated to dryness. Five milliliters of distilled water shall be added and evaporated to dryness. Add 10 milliliters of hot distilled water, filter and wash the residue twice with 5 milliliter portions of hot 3N ammonium acetate collecting all the washings. The filtrate shall be heated slightly and a few drops of concentrated hydrochloric acid followed by a few drops of 5 percent potassium chromate shall be added. The formation of a yellow precipitate of lead chromate shall indicate the presence of lead.

6. REPORT

6.1 The presence or absence of lead shall be reported.

METHOD 6621

January 15, 1969

pH VALUE OF LEATHER

1. SCOPE

1.1 This method is intended for determining the pH value of a distilled water extract of all types of leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be 4.00 ± 0.05 grams of leather from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 Potentiometric pH apparatus.

4.1.1.1 The pH meter shall include a glass electrode, a calomel reference electrode, and a potentiometer that will measure electromotive force and indicate pH directly with a precision of 0.1 pH units and a reproducibility of 0.05 pH units.

4.1.2 Glass stoppered Erlenmeyer flasks, 1000 milliliters and 250 milliliters capacities.

4.2 Reagents.

4.2.1 Water. Distilled water shall be boiled for 30 minutes to remove carbon dioxide. The flask shall be cooled to room temperature and stoppered. The pH of the carbon-dioxide-free distilled water shall be between 6.2 and 7.0 at 25°C. In cases of dispute, the water shall be prepared as indicated in Note 1.

4.2.2 Standard buffer solutions (see Note 2).

4.2.2.1 Hydrochloric acid buffer (pH 1.10 at 25°C). Add 2 grams of hydrochloric acid to 450 grams of distilled water. Standardize and dilute to 0.1N.

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4.2.2.2 Potassium hydrogen tartrate buffer (pH 3.56 at 25°C). Add 1 gram of $\text{KHC}_4\text{H}_4\text{O}_6$ to 100 milliliters of distilled water at approximately 30°C; shake vigorously for several minutes; cool to 25°C; decant and filter to remove any suspended salt. Add a crystal (about 0.1 gram) of thymol as a preservative.

4.2.2.3 Potassium hydrogen phthalate buffer (pH 4.01 at 25°C). Dissolve 10.21 grams of $\text{KHC}_8\text{H}_4\text{O}_4$ (ACS primary standard) in distilled water and dilute to 1000 milliliters.

4.2.2.4 Phosphate buffer (pH 6.86 at 25°C). Dissolve 3.40 grams of monobasic potassium phosphate KH_2PO_4 (ACS primary standard) and 3.55 grams of anhydrous disodium hydrogen phosphate Na_2HPO_4 in distilled water and dilute to 1000 milliliters.

4.2.2.5 Borax buffer (pH 9.18 at 25°C). Dissolve 3.81 grams of sodium tetraborate decahydrate $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$ in distilled water and dilute to 1000 milliliters.

4.2.2.6 Alkaline phosphate buffer (pH 11.72 at 25°C). Dissolve 1.42 grams of anhydrous disodium hydrogen phosphate Na_2HPO_4 in 100 milliliters of a 0.1N carbonate-free solution of sodium hydroxide NaOH and dilute to a liter with distilled water.

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 The specimen, weighed to the nearest 0.01 gram, shall be transferred to a 250 milliliter Erlenmeyer flask and twenty times its weight of freshly boiled and cooled distilled water added. The flask shall be tightly stoppered, the mixture agitated thoroughly and set aside with occasional agitation for a period of not less than 4 nor more than 18 hours. If the mixture is difficult to wet, apply vacuum until the leather sinks. The mixture shall then be transferred to a clean beaker and the temperature of the solution adjusted to $23^\circ \pm 1^\circ\text{C}$.

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5.2 Before the pH of the solution is measured, the instrument shall be turned on, allowed to warm up thoroughly, and brought to electrical balance in accordance with the manufacturer's instructions. The glass and calomel electrodes and the beaker shall be washed 3 times with distilled water and dried gently with clean absorbent tissue. A fresh liquid junction shall be formed. The temperature dial of the meter shall be adjusted to the proper setting.

5.3 Two standard buffer solutions that range above and below the anticipated pH of the test solution shall be selected. The electrodes shall be immersed in the first buffer solution (at the same temperature as the test solution), and the meter adjusted to the balance point. The pH shall be read directly from the dial. Successive portions of the buffer shall be taken until the instrument remains in balance within 0.02 pH units.

5.4 The electrodes shall be washed 3 times with distilled water and a fresh liquid junction formed. The electrodes shall be immersed in the second buffer, the instrument adjusted to the new balance point, and the pH read from the dial. The setting of the asymmetry potential knob shall not be changed. Additional portions of the second buffer shall be used until successive readings are in close agreement. The assembly shall be judged to be operating satisfactorily if the readings obtained for the second buffer solution agree with the assigned pH of that buffer within a 0.1 pH unit.

5.5 After the meter has been standardized with the 2 buffer solutions, the electrodes shall be washed 3 times with distilled water, and a new liquid junction formed. The electrodes shall be immersed in the test solution and the pH value obtained directly from the dial.

6. REPORT

6.1 The pH of the specimen shall be the average of the results obtained from the specimens tested, and shall be reported to the nearest 0.1 pH unit. Individual results utilized in obtaining the average shall be reported.

NOTE 1. In case of dispute, distilled water having a pH of not less than 6.2 nor more than 7.0 and a maximum residue of 0.0005 grams per 100 milliliters when evaporated to dryness shall be used. Test for the presence of alkaline impurities by boiling 2 specimens of approximately 100 milliliters for 15 minutes.

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Cover each with a watch glass and cool to room temperature. Wash the electrodes well with distilled water, then with previously boiled and cooled distilled (carbon dioxide-free) water. Measure the pH of both specimens. If these are not between 6.2 and 7.3, redistill the water from a solution containing 1 gram of KMnO_4 and 4 grams of NaOH per liter to remove acid or organic material, or from a dilute solution of H_2SO_4 , to remove any volatile alkalinity. The redistilled water shall have a pH of 6.7 to 7.2.

NOTE 2. Buffer salts prepared in accordance with National Bureau of Standards recommendations are sold by reputable laboratory supply houses and may be used.

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METHOD 6631
January 15, 1969

COMBINED TANNIN AND DEGREE OF TANNAGE.

1. SCOPE

1.1 This method is intended for determining the combined tannin and the degree of tannage of all types of vegetable tanned leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be as specified in the applicable test method.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, the number of specimens tested shall be as designated in the applicable test method.

4. APPARATUS, REAGENTS, AND METHODS CITED

4.1 Apparatus.

4.1.1 The apparatus shall be as specified in methods 6341 and 6441.

4.2 Reagents.

4.2.1 The reagents shall be as specified in methods 6341 and 6441.

4.3 Methods cited.

4.3.1 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

4.3.2 Method 6441. Nitrogen, Collagenous and Hide Substance.

5. PROCEDURE

5.1 The chloroform and water extractable materials and insoluble ash in the specimen for test shall be determined in accordance with method 6341.

5.2 The hide substance in the specimen for test shall be determined in accordance with method 6441.

5.3 Calculation of results.

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5.3.1 The percentage of combined tannin in the specimen shall be calculated as follows:

$$\text{Combined tannin, percent} = 100 - (A + B + C + D)$$

Where: A = percent of chloroform-soluble material.

B = percent of water-soluble material.

C = percent of insoluble ash.

D = percent of hide substance.

5.3.2 The degree of tannage shall be calculated as follows:

$$\text{Degree of tannage} = \frac{T}{D} \times 100$$

Where: T = percent of combined tannin.

D = percent of hide substance.

6. REPORT

6.1 The combined tannin in the specimen shall be reported to the nearest 0.1 percent.

6.2 The degree of tannage in the specimen shall be reported to the nearest 0.5 degree.

METHOD 6711

January 15, 1969

PARANITROPHENOL, COLORIMETRIC METHOD

1. SCOPE

1.1 This method is intended for quantitatively determining the amount of paranitrophenol used as a fungicide in leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be approximately 10.00 grams of leather weighed to the nearest 0.01 gram, from the composite sample, method 6002.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens from the composite sample shall be tested.

4. APPARATUS, REAGENTS, AND METHODS CITED.

4.1 Apparatus. Colorimetric equipment of either type listed below shall be used.

4.1.1 A spectrophotometer which can be set at a wave length of 630 millimicrons.

4.1.2 An electrophotometer with appropriate filters to permit reading at approximately 630 millimicrons.

4.2 Reagents.

4.2.1 Paranitrophenol. Dissolve 1 gram of paranitrophenol and 2 grams of sodium hydroxide in 1 liter of distilled water. Dilute 50 milliliters of this solution to 500 milliliters with distilled water.

4.2.2 Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$), 10 percent solution. Dissolve 20 grams sodium citrate in 180 milliliters of distilled water.

4.2.3 Titanous chloride, 20 percent solution. This reagent should be stored under refrigeration. If the solution becomes cloudy, it is no longer usable.

4.2.4 Sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), 3 percent solution adjusted to pH 9.6 with a 10 percent solution of sodium hydroxide.

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4.2.5 Phenol (C_6H_5OH), 5 percent solution.

4.2.6 Sodium hydroxide (NaOH).

4.3 Methods cited.

4.3.1 Method 6002. Preparation of Composite Sample.

4.3.2 Method 6221. Moisture Content, Oven Method.

4.3.3 Method 6341. Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the moisture content of the composite sample from which the test specimen is drawn shall be determined in accordance with method 6221. The weight of the composite specimen for moisture content shall be determined at the same time and under the same conditions as the specimen weighed for chemical tests.

5.2 The weighed specimen shall be transferred to a fat-free extraction thimble of suitable size to hold the specimen in a loosened state. Five milliliters of distilled water shall be added from a pipet to the specimen in the thimble. The specimen shall then be extracted with chloroform as described in method 6341. At the end of the extraction period, the flask shall be removed and the chloroform distilled off until the volume is less than 100 milliliters. The extract shall be transferred quantitatively to a 100 milliliter volumetric flask and made up to volume with chloroform. Twenty-five milliliters of the solution shall be placed in a 100 milliliter volumetric flask and made up to volume with chloroform.

5.3 To each of eight 100 milliliter volumetric flasks, add 20 milliliters of 10 percent sodium citrate solution.

5.4 Seven of the flasks containing sodium citrate as described in 5.3 shall be used for preparing standards containing the following increments of paranitrophenol: 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, and 0.40 milligrams. These concentrations shall be prepared by adding 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and 4.0 milliliters of the solution of paranitrophenol containing 0.0001 gram in 1 milliliter, described in 4.2.1, to each of the flasks respectively.

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5.5 One milliliter of the chloroform solution described in 5.2 shall be added to the eighth flask containing sodium citrate described in 5.3. The 8 flasks representing standards and unknown shall be treated alike in the subsequent procedure.

5.5.1 The paranitrophenol standards and the unknown shall be reduced to paraminophenol by slowly adding by pipet a solution of titanous chloride (prepared by diluting 1 milliliter of 20 percent solution to 100 milliliters with previously boiled and cooled distilled water) until the color changes from yellow to slight purple (2.5 to 10 milliliters will be required). The flask containing the mixture should be shaken vigorously.

5.5.2 Thirty milliliters of borate buffer solution and 10 milliliters of phenol solution shall be added to each flask and the flasks shaken vigorously. The flasks shall be made to volume with the borate buffer solution and thoroughly mixed. The flasks shall stand from 1 to 3 hours in subdued light to develop color (indophenol blue). If a cloudy color develops in the unknown, it shall be filtered through a No. 42 Whatman filter paper, discarding the first portion of the filtrate, before the color is determined.

5.6 The paranitrophenol in the unknown shall be determined colorimetrically by one of the following methods, depending on the type of equipment available.

5.6.1 With a spectrophotometer, read the transmission of the color standards at 630 millimicrons, and plot the transmission as the ordinate against milligrams of paranitrophenol as the abscissa. Read the transmission of the unknown solution, and determine the equivalent milligrams of paranitrophenol by reference to the standard curve.

5.6.2 With an electrophotometer, read the color of standards and unknown using a filter showing maximum transmission of about 630 millimicrons (Fisher's electrophotometer with 650-A filter or equivalent is satisfactory). Plot standard curve and determine concentration of paranitrophenol of the unknown as above (5.6.1).

5.7 Calculation of results. The paranitrophenol in the specimen shall be calculated as follows:

Paranitrophenol, percent =

$$\frac{\text{Milligrams paranitrophenol in aliquot} \times 40}{\text{Weight of specimen in grams} - \text{Moisture content in grams}}$$

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6. REPORT

6.1 The paranitrophenol in the specimen shall be the average of the results obtained from the specimens tested, and shall be reported to the nearest 0.01 percent. Individual results utilized in obtaining the average shall be reported.

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GASOLINE-WATER SEPARATION

1. SCOPE

1.1 This test is intended for determining the separation of water from gasoline by chamois leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be a piece of leather 10 ± 2 centimeters square, cut from the sample unit.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Funnel having an inside diameter of 75 ± 5 millimeters and an angle of 59 ± 2 degrees.

4.1.2 Wide-mouth jar (500 milliliters) with gasoline-resistant screw cap.

4.1.3 Stopwatch or other timing device.

4.2 Reagents.

4.2.1 Gasoline, conforming to Federal Specification VV-G-109 Gasoline, Unleaded.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.2 The specimen shall be placed in the bottle containing 100 milliliters of unleaded gasoline and shaken for 5 minutes. The specimen shall then be removed, allowed to drain, and formed in the funnel. Twenty-five milliliters of gasoline, to which 5 milliliters of distilled water has been added, shall be shaken for a few seconds and poured into the funnel immediately. The filtrate shall be collected in a dry 50 milliliter graduated cylinder and, after standing for one minute under the funnel, it shall be visually examined for globules of water or cloudiness.

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5.3 When specified in the material specification, the time required for the gasoline to be filtered shall be recorded to the nearest second.

6. REPORT

6.1 The absence or presence of globules of water or cloudiness in the filtered gasoline shall be reported.

6.2 When specified, the time required for the gasoline to be filtered shall be reported to the nearest second.

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METHOD 7011
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SHRINKAGE TEMPERATURE

1. SCOPE

1.1 This method is intended for determining the shrinkage temperature of leather. For the purpose of this method, shrinkage temperature shall be defined as the temperature at which noticeable shrinkage occurs on gradually heating the leather in a liquid medium, specifically water or a glycerine-water solution.

2. TEST SPECIMEN

2.1 The specimen for test shall be a rectangle of leather 76.0 ± 0.5 millimeters in length by 12.5 ± 0.5 millimeters in width, die cut from the sample unit of leather with the long dimension perpendicular to the backbone.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus. The apparatus for testing shall be as shown in figure 7011, consisting of the following parts:

4.1.1 A iron stand for supporting the testing equipment.

4.1.2 Two clamps mounted vertically 63.5 millimeters apart. The bottom clamp shall be stationary and the upper clamp shall be movable.

4.1.3 A liter beaker for holding the heating medium.

4.1.4 A stirrer for mixing the heating medium.

4.1.5 A thermometer graduated in degrees C.

4.1.6 A heating device with controls for raising the temperature of the medium at a rate of 3° to 5° C per minute.

4.1.7 An indicating device attached to the upper (movable) clamp and equipped with an adjustable weight assembly mounted over a pulley. The assembly shall consist of a small bottle filled with lead shot and a lid which shall be attached to the string running over the pulley. The weight

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assembly shall be approximately 178 grams to counterbalance the weight of the clamp, to overcome any inertia or friction of the indicating device, and to maintain the specimen under a slight tension. The weight shall be adjusted accordingly, so that it will not cause an elongation (prior to shrinkage) of more than 10 percent. The device shall register a scale reading to shrinkage or swelling ratio of the specimen of a least 25:1, and shall accurately detect the point at which shrinkage begins.

4.1.8 A metal die for cutting the specimen to the required dimensions.

4.1.9 A 500 milliliter suction flask.

4.2 Reagents.

4.2.1 Glycerine-water medium. When glycerine-water medium is specified for use, it shall consist of 75 volumes of glycerine and 25 volumes of water. The specific gravity of the solution shall be adjusted to 1.19 at 23°C. The glycerine-water mixture shall not be heated over 112°C.

5. PROCEDURE

5.1 When shrinkage temperatures at or below 99°C are specified, the heating medium shall be water.

5.2 When shrinkage temperatures over 99°C are specified, the heating medium shall be as described in 4.2.1.

5.3 The heating medium shall be transferred to a liter beaker, the heating device inserted, and the temperature adjusted to $50^{\circ} \pm 5^{\circ}\text{C}$. The bottom clamp shall be mounted on the iron stand in a stationary position. The movable clamp shall be mounted 63.5 millimeters above the stationary clamp. Prior to mounting, the specimen shall be placed in a 500 milliliter suction flask containing approximately 150 milliliters of the specified heating medium at room temperature. The flask shall be stoppered and vacuum applied until such time that upon release of the vacuum, the specimen settles to the bottom of the flask. The specimen shall be removed from the suction flask, fastened in the 2 clamps, completely immersed in the heating medium, and allowed to remain for a period of 10 ± 1 minutes, to allow the temperature of the specimen to adjust to that of the liquid medium. The indicator shall be adjusted to a reference or zero point on the dial. While stirring, the heating medium shall be heated in such a way as to raise the temperature at a rate of 3° to 5°C per minute. Frequently, the specimen will lengthen before it begins to contract. When this occurs, it is important to maintain the specimen under slight tension. Absence of shrinking or the temperature of the heating medium at which the specimen begins to shrink shall be noted.

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6. REPORT

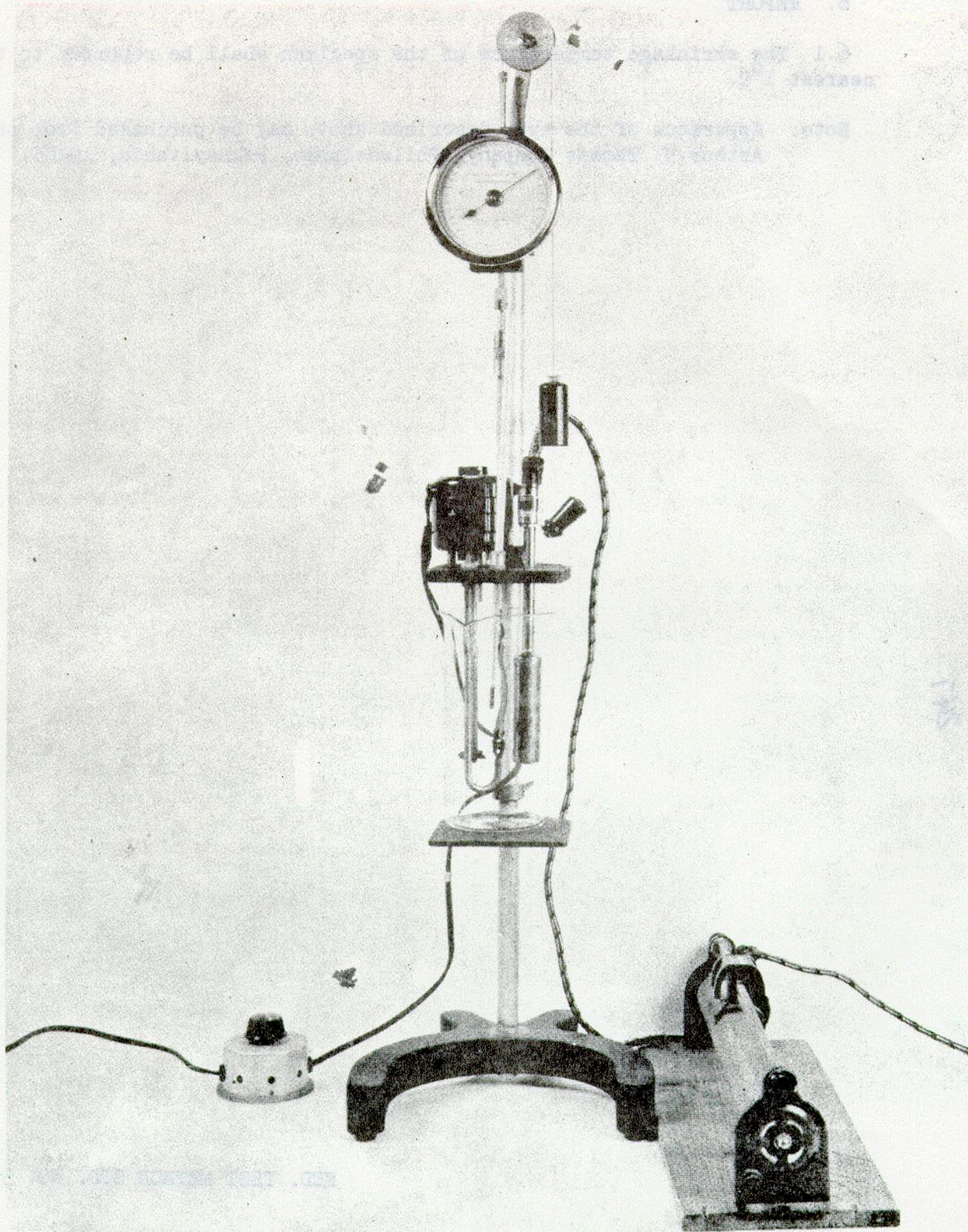
6.1 The shrinkage temperature of the specimen shall be reported to the nearest 1^oC.

Note: Apparatus of the type described above may be purchased from the Arthur H. Thomas Company, Philadelphia, Pennsylvania, 19105.

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Figure 7011. APPARATUS FOR TESTING SHRINKAGE TEMPERATURE.



FED. TEST METHOD STD. NO. 311

METHOD 7031
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AREA STABILITY OF LEATHER

1. SCOPE

1.1 This method is intended for determining the area dimensional change of leather after water immersion.

2. TEST SPECIMEN

2.1 The test specimen shall be a 51 ± 2 millimeter die cut square of the material undergoing test. If cut from a hide, it shall be selected from the test area specified. If prepared from insoles or other cut parts, it shall be selected not nearer than 13 millimeters from any previously cut or prepared edge.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Petri dishes.

4.2 Scale divided in millimeters.

4.3 Draft oven capable of maintaining a temperature between 20° and 25° C.

4.4 Distilled water.

4.5 Die, 51 by 51 millimeters, for cutting the specimen.

5. PROCEDURE

5.1 The required test specimen shall be cut from the sample unit. After the specimen has been cut, two measurements of each dimension shall be made and averaged. Each measurement shall be made 13 millimeters from a side of the specimen. The average measurements shall be used to determine the original area of the specimen and this area recorded as "A".

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5.2 Unless otherwise specified, each test specimen shall be placed separately in the bottom of a petri dish and covered with 50 milliliters of distilled water at a temperature of 20° to 25°C. The dish shall be covered and left standing for two hours. The test specimen shall then be removed and blotted with paper towels to remove excess water. The specimen shall then be laid flat on a wire screen and placed in a forced draft oven at a temperature of 20° to 25°C for four hours. At the end of the drying period, the specimen shall be removed from the oven and placed on a flat surface under standard conditions until equilibrium is reached.

5.3 A total of four measurements shall be taken on each test specimen. Each measurement shall be taken 13 millimeters from a side of the specimen. Two measurements shall be taken in one dimension, averaged, and the average recorded as "B". Two measurements shall be taken in the other dimension, averaged, and the average recorded as "C".

5.4 Calculation of results.

5.4.1 The percent area change shall be calculated to the nearest 0.1 percent as follows:

$$\text{Percent area change} = \frac{A - (B \times C)}{A} \times 100$$

Where: A = original area of the specimen

B = average of two measurements (millimeters)
in one dimension of test specimen.

C = average of two measurements (millimeters) in
other dimension of test specimen.

NOTE: In the event of increase in area of the test specimen the calculations shall be made as follows:

$$\text{Percent area change} = \frac{(B \times C) - A}{A} \times 100$$

6. REPORT

6. Unless otherwise specified, two specimens shall be tested from each sample unit and the results averaged and reported to the nearest 0.1 percent. Individual results utilized in obtaining the average shall be reported.

METHOD 7211
January 15, 1969

CRACKING, LOW TEMPERATURE

1. SCOPE

1.1 This method is intended for determining the resistance of leather to cracking on being bent at low temperatures.

2. TEST SPECIMEN

2.1 The specimen for test shall be free from mechanical damage and shall be a piece of leather 50 ± 2 millimeters square, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A low temperature chamber capable of maintaining the required temperature to within $\pm 1^{\circ}\text{C}$, and equipped to permit insertion of hands to hold the specimen.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed on material conditioned as specified in Section 5.

5.2 Unless otherwise specified, the conditioned material shall be exposed to a temperature of $0^{\circ} \pm 1^{\circ}\text{C}$ for a period of 2 hours ± 5 minutes and then tested at that temperature.

5.3 The specimen shall be placed in the chamber and maintained at the required temperature for the required period of time. While remaining in the cold chamber at the specified temperature, the specimen shall be folded crosswise sharply upon itself with the grain of the leather out. The folded specimen shall be examined for cracks in the area around the bend.

6. REPORT

6.1 The presence of cracks in the leather shall be reported.

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METHOD 8011

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PERMEABILITY, WATER VAPOR

1. SCOPE

1.1 This method is intended for determining the permeability of leather to water vapor by measuring the rate at which vapor passes through the material. The test is used as an indication of the relative comfort of shoe and garment leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be a disc of leather 69 to 76 millimeters in diameter, cut from the sample unit of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 The apparatus for permeability testing shall be a cup made of a non-corrosive material having the approximate design and dimensions as shown in figure 8011.

4.1.2 A metal die for stamping or cutting the specimen to the required dimensions.

4.1.3 Balance and weights.

4.2 Reagents.

4.2.1 A desiccant consisting of 8 mesh anhydrous calcium chloride dried at 200°C for 2 hours before use.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, the test shall be carried out under standard conditions specified in Section 5.

5.2 Unless otherwise specified in the material specification, the grain side of the specimen shall face the desiccant.

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5.3 The cup shall be filled with the desiccant to the level of the top of the rim. The specimen shall be immediately placed over the desiccant so that it will fit as nearly as possible over the center of the cup. A slight pressure shall be applied so that the specimen makes good contact around the entire rim of the cup. After the specimen has been placed on the rim of the cup, the top clamp shall be secured to the base so that the specimen will be held tightly in place. Plastic tape or other nonporous material shall then be placed over the edge of the cup and rim to prevent any passage of vapor through the edges of the specimen.

5.4 The completed assembly of cup and specimen shall be supported in such a manner that conditioned air may freely circulate over the exposed surface of the specimen. At the end of the first hour of exposure, the assembly shall be weighed to the nearest 0.01 gram. The weighing shall be repeated at intervals of 1 hour and the time recorded to the nearest minute, until the increase in weight has become constant to within ± 0.01 gram. The interval between weighings may be extended to 2 hours or more for specimens showing low permeability. The increase in weight, however, shall be calculated to a 1-hour exposure period, and shall be recorded as W.

5.5 Calculation of results. The water vapor permeability of the specimen in grams per square meter per day shall be calculated as follows:

$$\text{Permeability, grams per square meter per day} = \frac{W \times 240000}{A}$$

Where: W = average increase in weight of the assembly for a 1 hour exposure period, grams.

A = area in square centimeters of the specimen exposed to the desiccant.

6. REPORT

6.1 The water vapor permeability of the specimen shall be reported to the nearest 1 gram per square meter per day.

NOTE: Apparatus conforming to the requirements described in 4.1.1 may be purchased from Testing Machines Inc., Mineola, N. Y. 11501.

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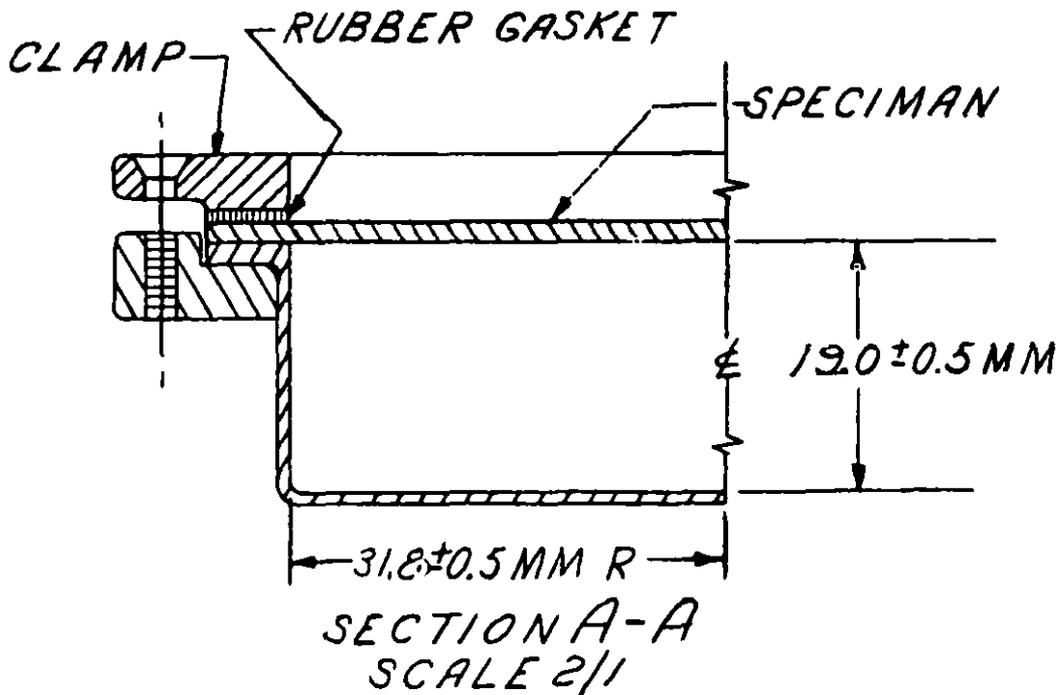
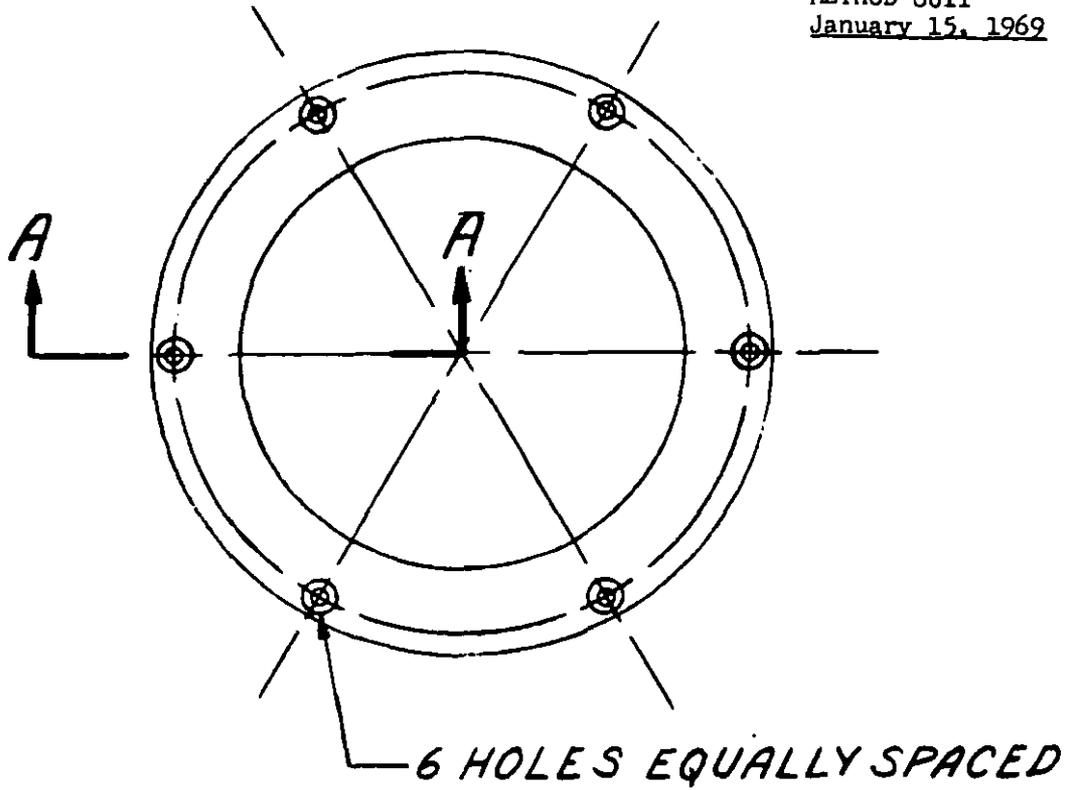


FIGURE 8011. PERMEABILITY TESTING APPARATUS.

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METHOD 8021
January 15, 1969

DYNAMIC WATER RESISTANCE OF LEATHER
BY THE MAESER WATER PENETRATION TESTER

1. SCOPE

1.1 This method is intended for determining the dynamic water resistance of leather by the Maeser water penetration tester. The flex imparted to the leather is similar to the flex given the vamp of the shoe in actual wear. Initial water penetration and water absorption can be measured simultaneously by this method. The method is not interchangeable with method 8131.

2. TEST SPECIMEN

2.1 The specimen for test shall be a square of leather approximately 4 by 4 inches, cut from the sample unit. The minimum size shall be not less than 3-7/8 by 4 inches; the maximum size shall be not greater than 4 by 4-1/2 inches.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Maeser Water Penetration Tester or its equivalent. The essential features of the machine are:

4.1.1.1 The machine shall have a specimen holder made of two V-shaped clamps with rubber wedges for holding the specimen. The clamps shall be 2.5 ± 0.1 inches apart, inside measurement, when their tops are in the same horizontal plane. One clamp shall be in a fixed position; the other clamp shall be pivoted and attached through a connecting link to a motor-driven eccentric which runs at 90 ± 5 rpm. In one rotation of the eccentric, the center of top of the moveable clamp shall move a distance of 1.00 ± 0.05 inches below the horizontal and return.

4.1.1.2 A water tank made of copper, stainless steel, or other noncorrosive material. The tank shall be of such a size that it can be placed around the clamps and of such a depth that, when in position for use, the top is 1.25 to 1.50 inches above the lowest part of the movable clamp.

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4.1.1.3 A base for the water tank. The base shall be removeable from between the machine frame and the water tank.

4.1.1.4 Two counting systems should be used to indicate the number of cycles through which the specimen is flexed. One shall be a mechanical reset counter connected to the moveable clamp. The other counter shall be electrical and shall consist of a high electrode and a common electrode. The resistance across the electrodes shall be between 5,000 and 10,000 ohms. When the resistance falls below this value, the relay shall be energized. The high electrode shall be placed inside the leather specimen in contact with the steel balls. The common electrode shall be in a salt solution which is in continuous contact with the specimen during flexing. The switch, when manually closed, shall start the machine and activate the testing circuit.

4.1.2 Monel or stainless steel balls, 1/8 inch diameter, 400 series.

4.1.3 Magnet (to facilitate removal of balls).

4.1.4 Balance sensitive to 0.01 gram.

4.2 Reagents.

4.2.1 Sodium chloride solution (1 gram per liter). Dissolve 1 gram of sodium chloride in distilled water and dilute to 1 liter.

5. PROCEDURE

5.1 The initial water penetration and water absorption may be determined on the same specimen as follows:

5.1.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.1.2 The specimen shall be weighed to the nearest 0.01 gram. The machine shall be set with the tops of the clamps in the same horizontal plane. The specimen shall then be folded in half with the grain side on the outside, so that it will be exposed to the salt solution. The specimen shall be fastened in the clamp by means of the V-shaped wedges, so that a trough is formed with the sides of the leather under slight tension.

5.1.3 The high electrode shall be adjusted so that it projects into the trough of the mounted specimen approximately 0.1 inch above the fold of the specimen when the distance between the clamps is at a minimum.

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5.1.4 Cleaned and dried monel or stainless steel balls weighing 135 ± 5 grams shall be placed into the trough formed by the folded specimen. (The balls may be cleaned with toluene or a chlorinated hydrocarbon and dried immediately at 100°C).

5.1.5 The water tank shall be put in place and filled with sodium chloride solution at $23^{\circ} \pm 1^{\circ}\text{C}$ to a level 0.75 ± 0.05 inches above the lowest point of the specimen adjacent to the fixed clamp. The other electrode shall be attached to the pan, the mechanical counter set to zero, and the electrode circuit activated.

5.1.6 The power to the mechanical drive shall be turned on. The number of flexes indicated on the counter when the machine stops shall be recorded as initial water penetration.

5.1.7 If water absorption is to be determined, the specimen shall be removed immediately from the apparatus, lightly blotted dry with absorbent paper to remove surface water, and weighed to the nearest 0.01 gram.

5.2 Calculation of results. The water absorption shall be calculated as follows:

$$\text{Water absorption, percent} = \frac{A-B}{B} \times 100$$

Where: A = weight of the specimen after flexing.

B = weight of the specimen before flexing.

6. REPORT

6.1 Initial water penetration shall be reported as the number of cycles recorded at the termination point.

6.2 Water absorption shall be reported to the nearest 0.1 percent.

NOTE: Apparatus of the type described in this method may be purchased from the Koehler Instrument Company, Inc., 168-56 Douglas Avenue, Jamaica, Long Island 11433.

The electrical counter may be purchased from Digit Controls, Marblehead, Mass. 01945.

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METHOD 8111
January 15, 1969

WATER ABSORPTION, STATIC METHOD

1. SCOPE

1.1 This method is intended for determining the amount of water absorbed by leather at room temperature when immersed under static conditions.

2. TEST SPECIMEN

2.1 The specimen for test shall be a rectangle of leather 76 ± 1 millimeters in length and 38 ± 1 millimeters in width, cut from the sample unit.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Blotting paper conforming to Federal Specification NNN-P-35, Paper, Blotting, (Laboratory).

4.2 Circulating air oven.

4.3 Glass dish or other container of suitable size for immersing the specimen in water in a horizontal position.

4.4 Weights for immersing and weighing the specimen.

4.5 Tannin dish.

4.6 Balance sensitive to 0.01 gram.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.2 The specimen shall be weighed and the weight recorded as W_1 . Distilled water at $23^{\circ} \pm 1^{\circ}\text{C}$ shall be transferred to the glass container and the specimen completely immersed in the water in a horizontal position with the grain side up. The specimen shall be suspended under the water in such a manner that no appreciable amount of surface area of the leather is left unexposed to the water. At the end of a 30 ± 1 minute immersion period, the specimen shall be removed and the surface water blotted off with blotting paper. The specimen shall then be weighed immediately and the weight recorded as W_2 .

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5.3 Water-soluble material. When specified in the material specification, a correction for water-soluble material shall be made as follows: The water in the glass container shall be stirred thoroughly and an aliquot of at least 25 milliliters of water shall be transferred from the immersion dish to a weighed tannin dish and evaporated to dryness in an oven at $105^{\circ} + 5^{\circ}\text{C}$. The dish with residue shall be weighed. The weight of the residue shall be used to calculate the total weight of water soluble material in the solution. The adjusted weight shall be recorded as W_3 .

5.4 Calculation of results.

5.4.1 The amount of water absorbed by the specimen shall be calculated as follows:

$$\text{Water absorbed, percent} = \frac{W_2 - W_1}{W_1} \times 100$$

Where: W_1 = weight of the original specimen, grams.

W_2 = weight of the specimen after immersion in water, grams.

5.4.2 The amount of water absorbed by the specimen, after correction for water-soluble material, shall be calculated as follows:

$$\text{Water absorbed, percent} = \frac{W_2 + W_3 - W_1}{W_1} \times 100$$

Where: W_3 = weight of the material dissolved from the specimen by water, grams.

6. REPORT

6.1 The amount of water absorbed by the specimen for test and whether it has been corrected for water solubles or not shall be reported to the nearest 0.1 percent.

METHOD 8112
January 15, 1969

WATER ABSORPTION, TUMBLE METHOD

1. SCOPE

1.1 This method is intended for determining the percent of water absorbed by leather.

2. TEST SPECIMEN

2.1 The specimen for test shall be a 2 inch die cut square of leather.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Balance sensitive to 0.01 gram and weights.

4.2 Two 32-ounce glass containers. Each container shall have a wide mouth and a screw cap. The diameter of each container shall be 3.5 ± 0.25 inches, and the total height (including screw cap) shall be 7 ± 0.25 inches.

4.3 Tumbling machine. The machine shall be of the rotating type, equipped to hold two 32-ounce containers, for end-over-end agitation. The speed of rotation shall be 55 to 60 revolutions per minute.

4.4 Blotting paper. The blotting paper shall conform to Federal Specification NNN-P-35, Paper, Blotting, (Laboratory).

4.5 A metal die having suitable measurements for cutting the specimen described in 2.1.

4.6 Non-absorbent flat-plate approximately 10 inches square.

5. PROCEDURE

5.1 Unless otherwise specified, this test shall be performed under the conditions and on material conditioned as specified in Section 5.

5.2 Each specimen shall be weighed to the nearest 0.01 gram and the weight recorded as W_1 .

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5.3 From 15 specimens of the sample lot being tested, 8 weighed samples shall be placed in 1 container, and the 7 other weighed specimens, plus 1 specimen for ballast, shall be placed in the other container.

5.4 Add to each container a volume of distilled water, at $23^{\circ} + 1^{\circ}\text{C}$, equal to 10 times the weight of the specimens therein. The containers shall be tightly closed and immediately placed in a tumbling machine. The containers shall be mounted vertically in an end-to-end position, so that the point of contact between the 2 containers rotates at the horizontal axis of rotation of the tumbling machine. The machine shall be started immediately after the containers are mounted and the tumbling continued for $30 + 1$ minutes. The water shall then be removed from both containers. One container shall be resealed. The specimens shall then be removed from the other container and evenly placed grain up between 2 sheets of blotting paper. No sheet of blotting paper shall be used more than once. A 10 by 10 inch, non-water absorbing, rigid flat plate and weight (if necessary) shall be placed on the cover blotter and a weight placed on top of the plate so as to subject the specimens to a total load of $860 + 60$ grams for 2 minutes. The plate and weight (if used) shall be removed and the specimens individually taken from between the blotters and weighed to the nearest 0.01 grams. This weight shall be recorded as W_2 . The procedure for weighing and blotting shall be repeated for the specimens in the other container. The total time required for blotting and weighing the specimens of both containers shall not exceed 20 minutes.

5.5 Calculation of results. The water absorbed by the specimen shall be calculated as follows:

$$\text{Water absorbed, percent} = \frac{W_2 - W_1}{W_1} \times 100$$

Where: W_1 = initial weight of the specimen.

W_2 = weight of the specimen after immersion in water.

6. REPORT

6.1 The amount of water absorbed by the specimen shall be reported to the nearest 0.1 percent.

METHOD 8121
January 15, 1969

WATER RESISTANCE, WITH TAP TESTER

1. SCOPE

1.1 This method is intended for determining the water resistance of shoe upper or glove leather. It simulates condition of leather being squeezed in contact with a moist object.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of leather, 1.25 ± 0.25 inches wide and of suitable length to be held firmly by the clips, cut from the sample unit.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 Suitable equipment as shown in figures 8121A and 8121B.

4.1.1 The apparatus shall consist of a rotating cylinder of brass or monel metal, 1.00 ± 0.05 inches wide and 2.80 ± 0.05 inches in diameter, with grooves 0.038 ± 0.008 inch wide and 0.038 ± 0.008 inch deep, spaced 18 degrees apart. This cylinder shall be the anvil for a brass or monel metal pressure foot 0.250 ± 0.005 inch in diameter, mounted on a lever arm of aluminum, stainless steel, or monel metal, 0.75 ± 0.10 by 0.75 ± 0.10 by 16.5 ± 0.5 inches. While held in a pivot, this arm shall be lifted to a height of 0.500 ± 0.015 inch above the anvil by means of a rotating cam. The shafts holding the cam and the anvil shall be counted on a common base, and shall be driven at a speed of 37.5 ± 3.0 rpm.

4.1.2 The arm shall be fitted with a platform or spindle or other means for holding a weight sufficient to exert a thrust at the pressure foot of 0.5, 2.0, or 3.5 pounds, as specified in the material specification, when the foot is released by a half-turn of the cam and falls upon the anvil. The force is measured by temporarily connecting the arm at the point where the pressure foot is attached to the rod of a spring scale having a range from 0 to 5 pounds and subdivided into 1 ounce divisions. A ± 1 ounce tolerance is permissible for each thrust force specified.

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4.1.3 Near the pivot of the arm, an arresting level shall be attached to the base holding the arm so as to lift the arm up when not in use and to facilitate the insertion of the specimen at the other end.

4.1.4 A suitable attaching device shall be used so that the sample may be held without flapping or interfering with the tapping action.

4.2 A trough for immersing the circular anvil into water.

4.3 A counting device to record the number of taps to which the specimen is subjected. This device shall be attached to either of the rotating shafts. A timer counting the number of seconds during which the test apparatus is operated may be used in place of the counter.

4.4 An electrical system for detecting the leak point of the test specimen. The system shall be capable of clearly indicating resistance values up to 1.2 megohms in electrical circuits.

NOTE: An electrical switch, equivalent to the Ripley Electronic Switch, Model 8282 - Type ES-15, 3 megohms, reduced in sensitivity to 1.0 ± 0.2 megohms, meets these requirements.

4.4.1 The test leads shall be attached in such a manner as to immediately detect water penetration through the specimen.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.2 Unless otherwise specified in the material specification, the specimens shall be mounted with the grain side of the leather exposed to the water.

5.3 Distilled water at a temperature of $23^{\circ} \pm 1^{\circ}\text{C}$ shall be added to the trough, and shall cover the periphery of the anvil to a depth not exceeding $3/8$ inch. Electronic switching devices shall be activated one minute prior to starting the test. The test shall be started by turning on the motor drive, releasing the arm from the holder, and placing the pressure foot with covering specimen on the anvil. The rotation counter or timing device shall be started. The tapping of the specimens shall be continued until the leak detector device shows the leather resistance value to be 1.0 ± 0.2 megohm, or until the test specimen has exceeded the number of taps or seconds of testing specified in the material specification.

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6. REPORT

6.1 The number of cycles or seconds to water penetration of the specimen shall be reported.

NOTE: Further details of construction of a single anvil machine are available from the U.S. Army Natick Laboratories, Natick, Massachusetts. A commercial source of the single and multi-anvil tap tester is the Kearny Engineering Co., 333 38th Street, Brooklyn, New York 11232.

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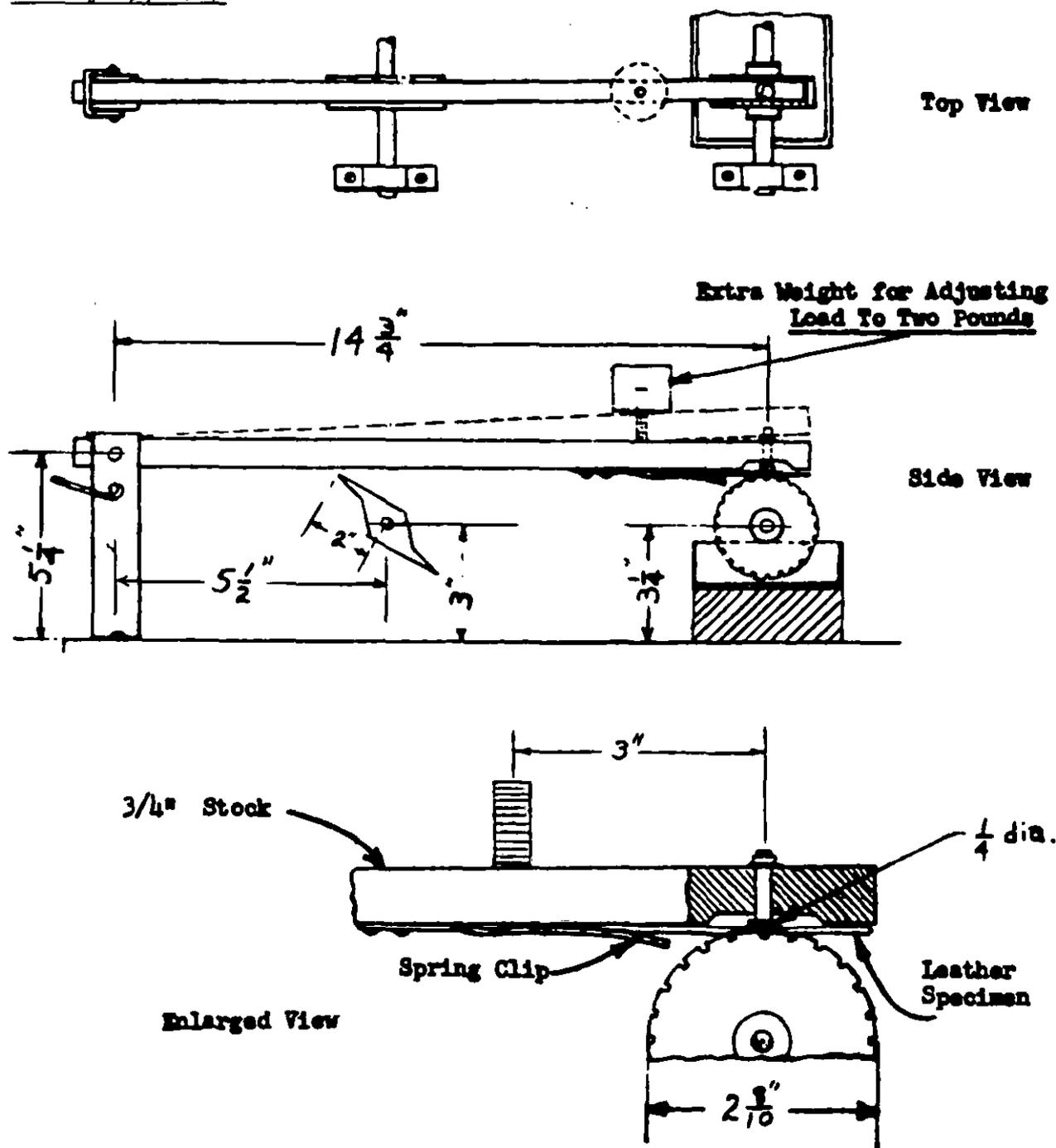


Fig. 8121 A

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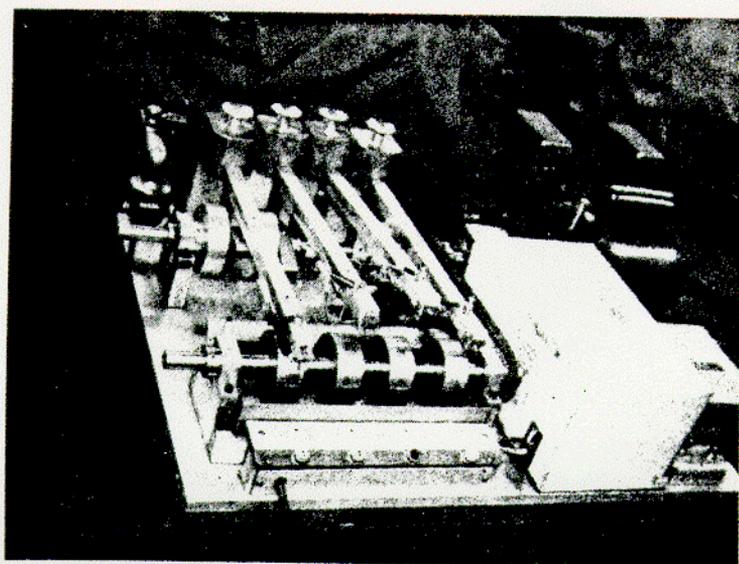


Fig. 8121 B

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Q12/692

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DYNAMIC WATER RESISTANCE OF SHOE UPPER
LEATHER BY THE DOW CORNING LEATHER TESTER

1. SCOPE

1.1 This method is intended for determining the dynamic water resistance of shoe upper leather. It measures initial water penetration and water absorption of the upper leather by flexing the leather while in contact with water. The flex imparted to the leather is a magnification of the flex given the vamp of the shoe in actual wear.

2. TEST SPECIMEN

2.1 The test specimen shall be a 10.0 ± 0.3 by 10.0 ± 0.3 centimeter square of leather cut from the sample unit.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS, REAGENTS AND MATERIALS

4.1 Apparatus.

4.1.1 Test machine. The test machine shall have the following essential features:

4.1.1.1 Specimen holder. The specimen holder shall consist of two vertical clamps. Each clamp shall have two arms constructed of non-corrosive material, one arm fixed, the other moveable. One clamp shall be mounted on a horizontal reciprocating shaft that is attached by a connecting link to a motor-driven eccentric which turns at 60 rpm. The bottom of the clamps shall be in the same horizontal plane. In one rotation of the eccentric, the minimum distance between clamps shall be 3.800 ± 0.025 centimeters (inside measurement) and the maximum distance between clamps shall be 6.350 ± 0.025 centimeters (inside measurement).

4.1.1.2 Water pan. The machine shall have a water pan made of stainless steel or other non-corrosive material. It shall be of such a size that it can be placed around the clamps, and of such a depth that when in position for use, the top of the pan is at least 6 centimeters above the bottom of the clamps, and the bottom of the pan is at least 1.5 centimeters below the bottom of the clamps.

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4.1.1.3 Cycle counters. In addition to having a mechanical reset counter attached to the moveable clamp, the test machine shall have an electrical counting system having a high electrode that is positioned so that it fits inside of the leather specimen and a common electrode which is immersed in a salt solution that is in continuous contact with the specimen during flexing. (caution - see Note) The electrical counter shall be arranged so that it may be set in operation by a manual switch and automatically stopped when initial water penetration is detected by a current leakage from the high electrode through the specimen to the common electrode in the conduction solution.

NOTE: Caution: Specimens should be put into position or removed only when the electrode circuit is not energized.

4.1.2 Balance accurate to 0.01 gram.

4.1.3 Monel or stainless steel balls, 0.318 centimeter diameter, 400 series.

4.1.4 Rubber gasket material 0.60 centimeters thick (red sponge corrugated floor material has been found satisfactory.)

4.1.5 Magnet (to facilitate removal of balls).

4.2 Reagents.

4.2.1 Sodium chloride solution (1 gram per liter). Dissolve 1.0 gram of sodium chloride in distilled water and dilute to 1 liter.

5. PROCEDURE

5.1 Water penetration and water absorption (optional).

5.1.1 The specimen shall be weighed to the nearest 0.01 gram if water absorption is to be determined.

5.1.2 The sodium chloride solution (at room temperature) shall be added to the pan until the level is 3.7 ± 0.2 centimeters above the bottom of the clamps when the pan is in operating position and there is no specimen in the clamps.

5.1.3 The drive shaft shall be turned until the clamps are at the maximum distance apart.

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5.1.4 The test specimen shall be folded in half with the surface to be exposed to the salt solution on the outside. A rubber gasket shall be folded in half and inserted into the folded specimen in a position flush with the edge of the specimen and in contact with the bottom of the fold. (When water absorption is not specified, a gasket cement may also be utilized).

5.1.5 The edge of the specimen with the inserted gasket shall be securely clamped between the jaws of the back clamp and shall be positioned with the bottom of the fold flush with the bottom of the clamp. The same procedure shall be followed for the opposite end of the specimen, except that it shall be securely clamped between the jaws of the front clamp.

5.1.6 The high electrode shall be adjusted so that it projects into the trough of the mounted specimen at a position of 0.30 ± 0.15 centimeters above the fold in the specimen when the distance between the clamps is at a minimum.

5.1.7 135 ± 5 grams of cleaned and dried monel or stainless steel balls shall be placed in the pocket formed by the folded specimen. (The balls may be cleaned with toluene or a chlorinated hydrocarbon and dried immediately at 100°C .)

5.1.8 The apparatus shall be started, after the pan containing the salt solution is in position, by engaging the drive link mechanism and actuating the electrode circuit.

5.1.9 The number of cycles indicated on the counter when the machine stops shall be recorded as initial water penetration.

5.1.10 If water absorption is to be determined, the specimen shall be removed from the apparatus, blotted dry with absorbent paper, and weighed to the nearest 0.01 gram.

5.2 Calculation of results.

5.2.1 The water absorption shall be calculated as follows:

$$\text{Water absorption, percent} = \frac{A-B}{B} \times 100$$

Where A = weight of specimen after flexing.

B = weight of specimen before flexing.

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6. REPORT

6.1 Initial water penetration shall be reported as the number of cycles recorded at the termination of the test.

6.2 Water absorption shall be reported to the nearest 0.1 percent.

NOTE: Apparatus of the type described in this method may be purchased from the Dow Corning Corp., Midland, Michigan 48640.

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METHOD 8141
January 15, 1969

WATER ABSORPTION, INSOLES

1. SCOPE

1.1 This method is intended for determining the amount of water absorbed by shoe insole leather with the grain side exposed.

2. TEST SPECIMEN

2.1 The specimen for test shall be a disc of leather 7.5 ± 0.2 centimeters in diameter, cut from the leather insole.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS AND MATERIALS

4.1 Apparatus.

4.1.1 Plexiglass disc, 7.5 ± 0.2 centimeters in diameter, weighing between 15 and 20 grams.

4.1.2 Weight, 500 grams.

4.1.3 Balance, accurate to the nearest 0.01 gram.

4.2 Materials.

4.2.1 Blotting paper conforming to Federal Specification NNN-P-35, Paper, Blotting, (Laboratory).

4.2.2 Water-resistant cement.

5. PROCEDURE

5.1 Unless otherwise specified in the material specification, this test shall be performed on material conditioned as specified in Section 5.

5.2 The 7.5 ± 0.2 centimeter disc of conditioned leather shall be weighed to the nearest 0.01 gram and the weight recorded as W_1 . After weighing, the disc shall be carefully cemented around the edge by the application of a uniform coating of water-resistant cement. The cemented specimen shall be allowed to remain under standard conditions for a minimum of 24 hours and reweighed to the nearest 0.01 gram. This weight shall be recorded as W_2 .

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5.3 Five sheets of blotting paper, each measuring 25.4 by 25.4 centimeters, shall be weighed and individually soaked in water at 23°C until their weight has been increased by 210 ± 10 percent. The five sheets of blotting paper shall then be stacked one upon the other on a smooth, rigid horizontal surface.

5.4 The weighed specimen from 5.2 shall be placed flesh side up on the wet blotting paper. A plexiglass disc having a diameter of 7.5 ± 0.2 centimeters and weighing between 15 and 20 grams shall be placed exactly on top of the specimen, and a 500 gram weight shall be placed on top of the disc. After 10 ± 1 minutes, the specimen shall be removed, blotted of excess surface water, if present, and then immediately weighed to the nearest 0.01 gram. The weight shall be recorded as W_3 . No more than four specimens per unit stack of blotting paper shall be permitted. Re-use of the blotting paper and retesting of the specimens shall not be permitted.

5.5 Calculation: The amount of water absorbed by the insole shall be calculated as follows:

$$\text{Water absorption, percent} = \frac{W_3 - W_2}{W_1} \times 100$$

Where: W_1 = original weight of the conditioned specimen.
 W_2 = weight of the specimen after cementing.
 W_3 = weight of the specimen after absorbing water.

6. REPORT

6.1 The amount of water absorbed by the insole shall be reported to the nearest 0.1 percent.

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METHOD 8231
January 15, 1969

WATER MARKS ON GRAIN LEATHER

1. SCOPE

1.1 This method is intended for determining whether or not water will produce marks on grain leather.

2. TEST SPECIMEN

2.1 Unless otherwise specified, the specimen shall consist of a piece of leather at least 2 by 4 inches in size.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, one specimen from each sample unit shall be tested.

4. APPARATUS

4.1 A block with a plane at least 5 inches square sloped at a 45° angle to the horizontal.

4.2 A 50 milliliter pipet mounted above the center of the block so that its tip is (10 inches) away from the center of the incline.

4.3 Mandrel, 0.25 inch diameter.

5. PROCEDURE

5.1 The specimen shall be placed grain up on a flat surface and one end wrapped over a $1/4$ inch mandrel. The specimen shall be rolled so that its full length is bent ten times over the mandrel. The specimen shall be unrolled and examined for cracking or parting of finish. If no cracking or parting is visible, the specimen shall be immediately mounted on a minimum 5 inch square plane sloped at a 45° angle to the horizontal. Fifty milliliters of distilled water at $80^{\circ} + 5^{\circ}F$ shall be drawn into a pipet and after mounting the pipet so that its tip is 10 inches away from the center of the specimen, the water shall be allowed to flow out and run off the grain side of the specimen. The specimen shall then be placed on a horizontal surface. After two hours, the specimen shall be hand buffed with a soft cloth and examined at a distance of two feet. The absence or presence of spotting, discoloration or blistering shall be reported.

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6. REPORT

6.1 If water marks are visible at a distance of 2 feet, the specimen shall be reported as failing the test.

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SECTION 9

SUPERSESION DATA, SOURCE INFORMATION, AND INTERESTED AGENCIES

9.1 This Standard incorporates and supersedes all of the provisions of Federal Specification KK-L-311A, Leather; Methods of Sampling and Testing, in addition to instituting revisions in the sampling and testing procedures as required to provide a more technically adequate and updated laboratory tool.

9.2 This Standard contains the following changes with regard to Federal Specification KK-L-311A. The changes are effective on date of issue of this standard:

<u>Methods combined</u>	<u>Consolidated method</u>
1211 - Width, Unit 1221 - Width, Specimen	1211 - Width and Length, Unit
2011 - Tensile Strength 2021 - Elongation (Medium and Heavy Materials)	2021 - Breaking Force or Tensile Strength, and Elongation; Dumbell strip Method
2031 - Breaking Strength 2041 - Elongation (Light Materials)	2031 - Breaking Force and Elongation, Grab Method
6321 - Organic Solvent Extract (Grease) 6341 - Water-Soluble Materials 6411 - Ash, Insoluble	6341 - Chloroform-Soluble and Water-Soluble Materials, and Insoluble Inorganic Materials
6441 - Nitrogen, Total 6461 - Hide Substance	6441 - Nitrogen, Collagenous and Hide Substance
6631 - Tannin, Combined 6641 - Tannage, Degree of	6631 - Combined Tannin and Degree of Tannage

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9.3 The following methods have been deleted as a result of combination with other procedures as outlined above or as being obsolete:

Method 1221 - Width, Specimen
 Method 2011 - Tensile Strength
 Method 2041 - Elongation, (Light Materials)
 Method 3311 - Aging, Accelerated
 Method 4311 - Abrasion
 Method 5001 - Microbiological Tests, General
 Method 5011 - Mildew Resistance, Mixed Spores
 Method 5031 - Mildew Resistance, Myrothecium Verrucaria and
 Aspergillus Ustus
 Method 6321 - Organic Solvent Extract (Grease)
 Method 6331 - Unsaponifiable Material
 Method 6411 - Ash, Insoluble
 Method 6451 - Nitrogen, Total, Referee Method
 Method 6461 - Hide Substance
 Method 6611 - Glucose
 Method 6641 - Tannage, Degree of
 Method 7021 - Curling Temperature
 Method 7111 - Stiffness, Low Temperature
 Method 8031 - Water Penetration, Amount

9.4 The following new methods have been included:

Method 1231 - Apparent Density of Leather
 Method 2161 - Tearing Strength, Slot Tear
 Method 3031 - Colorfastness (Resistance to Rubbing), Precision
 Crockmeter
 Method 3121 - Blocking
 Method 3202 - Finish Stability
 Method 3203 - Corrosive Effects of Leather
 Method 3221 - Resistance to Perspiration (White Chrome Leather)
 Method 5041 - Mildew Resistance, Tropical Chamber Method
 Method 6371 - Acetone Insoluble Material
 Method 6452 - Nitrogen, Water Extractable
 Method 6518 - Chromic Oxide, Wet Oxidation
 Method 6551 - Lead Salts, Presence of
 Method 6811 - Gasoline-Water Separation
 Method 7031 - Area Stability of Leather
 Method 8112 - Water Absorption, Tumble Method
 Method 8131 - Dynamic Water Resistance of Shoe Upper Leather by
 Dow Corning Leather Tester
 Method 8141 - Water Absorption, Insoles
 Method 8231 - Water Marks on Grain Leather

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