This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D6404 – 99 (Reapproved 2020)

Standard Practice for Sampling Vegetable Materials Containing Tannin[1]

This standard is issued under the fixed designation D6404; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers obtaining representative samples from shipment lots of botanical materials containing tannin.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1517 Terminology Relating to Leather
2.2 *ALCA Method:*
J10 Sampling Vegetable Materials Containing Tannin[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of general leather and tanning terms used in this practice refer to Terminology D1517.

3.1.2 *quartering*—the term applied to a method described in this practice of reducing the size of samples without impairing their representative quality.

3.1.3 *tannin*—an astringent substance found in the various parts of plants such as bark, wood, leaves, nuts, fruits, roots, etc.

3.1.4 *vegetable tannins*—mixtures of substances (natural products) obtained from plant tissues by water extraction which have the chemical and physical properties necessary to convert animal hides and skins into leather.

4. Summary of Practice

4.1 This practice describes methods for obtaining representative samples for analysis from shipments of vegetable tanning materials and tannin extracts.

5. Significance and Use

5.1 This practice provides standard procedures for obtaining representative samples of various materials used as a source of tannins for the tanning industry.

5.2 Procedures are described for obtaining representative samples of economical and convenient quantities from a lot, or sections of a lot, of material for examination and analysis so that agreement may be reached with regard to the extent of variation of quality in different portions of a lot and the average quality of the entire lot of material.

5.3 No directions for sampling, however explicit, can take the place of judgment, skill, and previous experience on the part of persons actually engaged in the sampling or the supervision of the sampling. These directions are intended to supplement that experience and, particularly, to serve as a guide in the selection of the method which is to be used, in common, by each of two or more contracting parties.

5.4 Tanning materials contain moisture in varying amounts, depending both on the nature of the material and on the climatic conditions, so that sampling must be carried out as quickly as is consistent with thoroughness in order to avoid changes in moisture content.

6. Apparatus

6.1 *Balance,* analytical balance which will weigh the quantity of material specified in this practice to an accuracy of ±10 g (± 0.35 oz).

6.2 *Canvas,* 11-oz weight and of sufficient size (as determined by the discretion of the sampler or analyst) for use in mixing and quartering samples.

[1] This practice is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.01 on Vegetable Leather. This test method has been adapted from and is a replacement for Method J10 of the Official Methods of the American Leather Chemists Association.

Current edition approved Dec. 1, 2020. Published December 2020. Originally approved in 1999. Last previous edition approved in 2014 as D6404 – 99 (2014). DOI: 10.1520/D6404-99R20.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Official Methods of the American Leather Chemists Association. Available from the American Leather Chemists Association, University of Cincinnati, P.O. Box 210014, Cincinnati, OH 45221-0014.

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6.3 *Sieve,* with round openings 1.27 cm (0.50 in.) in diameter.

6.4 *Sampling Tool,* this tool shall be made of brass or other corrosion-resistant metal tubing, with solid handle attached, similar to that described below and illustrated in Fig. 1.

6.4.1 Thin brass or other corrosion-resistant metal tubing, 37 cm long and 17.5 mm diameter, is used. A solid steel handle, 6 cm long, is welded to one end of the tube. A section 11 mm wide and 30 cm long is cut out of the side of the tube and the edges of this opening sharply beveled. The working end is cut to a sharp point for piercing sacks, or other tough materials, like plantation gambier.

7. Quartering Samples

7.1 "Quartering" is a term applied to the following method of reducing the size of samples without impairing their representative quality.

7.1.1 All of the material, taken from the various containers or parts of the shipment of solid material being sampled, shall be immediately and thoroughly mixed on a square piece of 11 oz canvas. In the case of solid extracts, large pieces shall have been broken previously so as to pass a screen having round openings 1.27 cm (0.50 in.) in diameter. The pile shall be divided into four equal portions and quarters A and B shall be discarded as illustrated in diagram X of Fig. 2. The remaining two quarters shall then be re-mixed thoroughly and again divided into four equal portions and quarters C and D shall be discarded as illustrated in diagram Y of Fig. 2. This process shall be repeated until the mixture of any two remaining quarters of the sample be of sufficient size to secure the individual samples as specified below. The individual samples shall be obtained by thoroughly mixing the two remaining quarters and dividing the mixture into as many uniform sections as necessary, such that each section will be large enough to fill one of the required number of containers. All fine, powdery material in each section shall be completely removed with its respective section, whether the section is discarded or placed in a sample container.

8. Filling Containers

8.1 Immediately after the final samples are obtained, they shall be put into clean, dry containers, closed airtight, sealed, and labeled as described in the procedure (11.11).

8.2 Liquid and powdered extracts and tannery liquor samples shall be put into stoppered glass bottles of the required size.

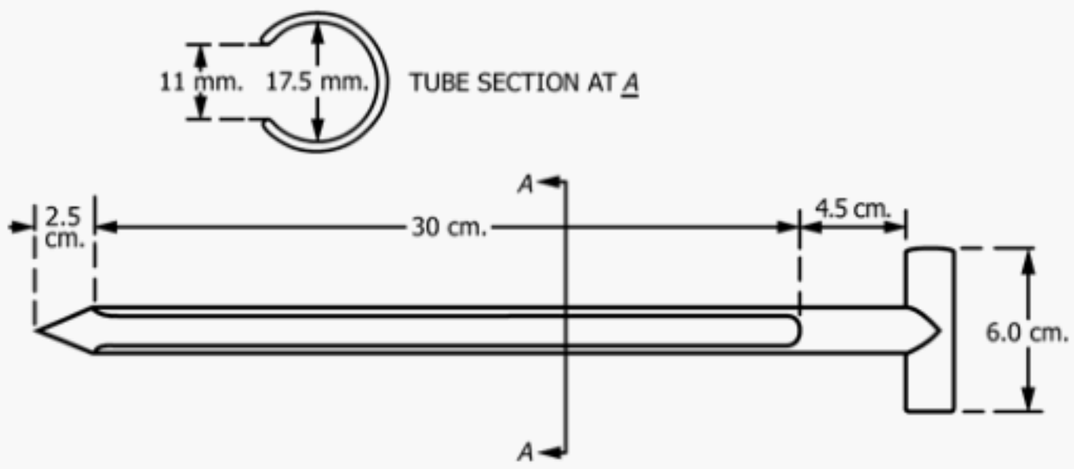

FIG. 1 Sampling Tool

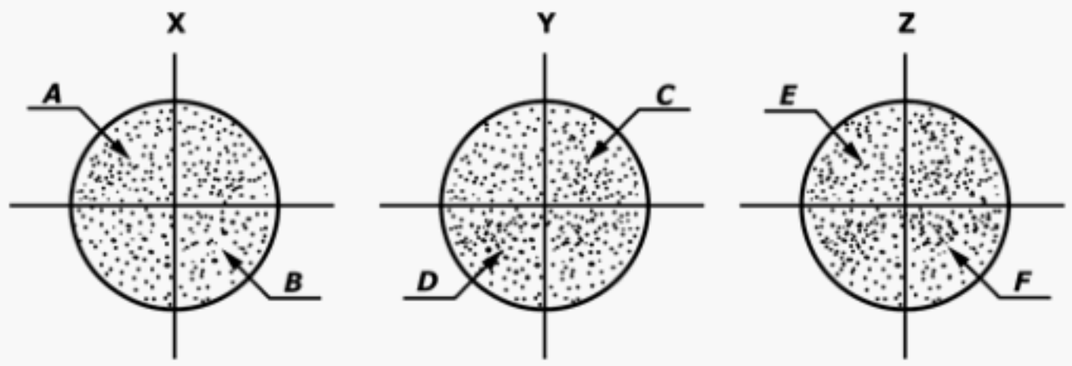

FIG. 2 Quartering Samples

8.3 Solid and pasty extracts shall be wrapped in aluminum foil and put into Mason-type jars or tin cans of the required size.

8.4 Cut bark, fruits, roots, galls, nuts, leaves, etc., and spent materials, shall be placed in lacquered, pressed-top tins of the required size, which close airtight. Boxes or bags must not be used as containers.

8.5 Sample containers shall be of no greater capacity than will conveniently be filled by the sample taken.

9. Number of Packages to be Sampled

9.1 The number of packages to be sampled from any given lot of tanning material composed of solid, powdered or pasty extract, crude or manufactured tanning materials, in bales, boxes, bags, barrels, or similar packages, shall be as shown in Table 1. The samples shall be selected from as uniformly distributed parts of the lot as is possible at the time of weighing.

9.1.1 Where the shipment exceeds 10 000 packages, it shall be divided into as few, equal, sections (of not more than 10 000 packages each) as possible and each section shall be sampled and analyzed as if it were an individual shipment. In such case, the mean of the analyses of the sections shall constitute the analysis of the shipment.

10. Number of Samples

10.1 In addition to the representative samples for submission to the contracting parties, at least one extra sample shall be taken and held in reserve by the sampler, in case one of the regular samples is lost or damaged.

11. Procedure

11.1 *Solid Extracts*—Cut a suitable triangular opening in the middle of the exposed side of the package to be sampled. Then cut a V-shaped wedge, 10 to 12.5 cm (4 to 5 in.) in width on the surface of the exposed side of the package, as near to the middle as possible and to the depth shown in the diagram illustrated in Fig. 3.

11.1.1 Remove this piece, including any dry or crumbly exterior portion of it, place on the quartering canvas and covered to prevent loss of moisture. Promptly break the V-shaped samples from all the selected packages to a size which will pass the sieve (6.3). Quarter the mixture and sample as described in Section 7. Each final sample shall weigh approximately 225 g (8 oz) and shall be packaged as described in 8.3.

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TABLE 1 Sampling Quantities

Number of Packages in Lot	Number of Packages to Be Sampled	Number of Packages in Lot	Number of Packages to Be Sampled	Number of Packages in Lot	Number of Packages to Be Sampled
1 to 3	each	1201 to 1300	25	4551 to 4700	48
3 to 24	3	1301 to 1400	26	4701 to 4900	49
25 to 40	4	1401 to 1510	27	4901 to 5100	50
41 to 60	5	1511 to 1640	28	5101 to 5300	51
61 to 84	6	1641 to 1730	29	5301 to 5520	52
85 to 112	7	1731 to 1860	30	5521 to 5720	53
113 to 144	8	1861 to 1980	31	5721 to 5940	54
145 to 180	9	1861 to 1980	32	5941 to 6160	55
181 to 220	10	2101 to 2240	33	6161 to 6380	56
221 to 264	11	2241 to 2380	34	6381 to 6600	57
265 to 312	12	2381 to 2520	35	6601 to 6840	58
313 to 364	13	2521 to 2660	36	6841 to 7080	59
365 to 420	14	2661 to 2810	37	7081 to 7320	60
421 to 480	15	2811 to 2960	38	7321 to 7560	61
481 to 544	16	2961 to 3120	39	7561 to 7800	62
545 to 612	17	3121 to 3280	40	7801 to 8060	63
613 to 684	18	3281 to 3440	41	8061 to 8320	64
685 to 760	19	3441 to 3610	42	8321 to 8580	65
761 to 840	20	3611 to 3780	43	8581 to 8840	66
841 to 920	21	3781 to 3960	44	8841 to 9120	67
921 to 1010	22	3961 to 4140	45	9121 to 9400	68
1011 to 1100	23	4141 to 4320	46	9401 to 9660	69
1101 to 1200	24	4321 to 4550	47	9661 to 10 000	70

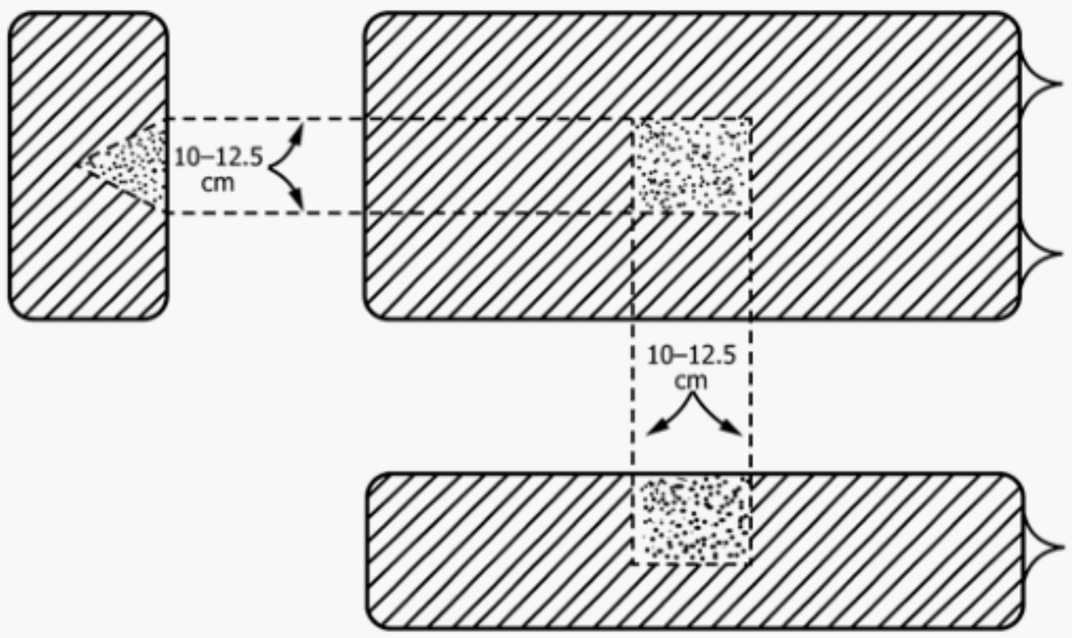

FIG. 3 Package Cutting

11.2 *Powdered Extracts*—Draw a portion from each of the specified number of bags, using the sampling tool (6.4). Lay the bags horizontally, with the narrow side exposed, and insert the sampling tool into the middle of the exposed side and to the center of the bag. The portions removed shall be of equal quantity, thoroughly mixed, and the mixture quartered and sampled as described in Section 7. Each sample shall weigh approximately 170 g (6 oz.) and be packaged as described in 8.2.

11.3 *Barks, Roots, Galls, Nuts, etc., in Bags*—Draw samples as under "Powdered Extracts" (11.2), using a scoop, when necessary, instead of the sampling tool. Thoroughly mix the portions and quarter and sample the mixture as described in Section 7. Each sample shall consist of enough material to fill a 3.8 L (1 gal) container, at least, and be packaged as described in 8.4.

11.3.1 In sampling Valonia cups and beards, take three portions from each of the specified number of bags, one from the middle, one midway between the middle and one end, and the third between the middle and the other end. Thoroughly mix the portions and quarter and sample the mixture as described in 11.3.

11.3.1.1 In case of disagreement, or when agreed upon between buyer and seller, the following method may be used: the specified number of bags shall be screened through the sieve (6.3) in their entirety and the percentage of cups and beards determined by weighing. Draw samples of each by mixing and quartering, etc., as described in 11.3. State the relative proportions of cups and beards, found by weighing, on the label and the same proportions taken for analysis.

11.4 *Bark and Wood in Bales*—Sample chopped bark or similar material in bales by cutting a 15 cm (6 in.) slit in the approximate middle of the covering of the bale. Using a meat hook about 25 cm (10 in.) long, dig out the material through the slit as deeply as possible. Thoroughly mix and sample these portions as described in Section 7. Each final sample shall consist of enough material to fill a 3.8 L (1 gal) container, at least, and be packaged as described in 8.4.

11.5 *Bark and Wood in Sticks or Logs, Bulk Shipment*—Select the required number of pieces and saw or break into smaller pieces, each representing in relative size, as nearly as is practicable, the piece from which it was taken. The sawing must be done so as to avoid iron contamination of the sample from the saw or other cutting instrument. Overheating of the sample because of friction from the cutting tool must be avoided. Thoroughly mix and sample the sample portions obtained as described in Section 7. Each final sample shall consist of enough material to fill a 3.8 L (1 gal) container, at least, and be packaged as described in 8.4.

11.6 *Wood as Chips (from Chipper)*—Take chips from the chipping machine at regular intervals during the run being examined and immediately place in a suitable, closed container. Thoroughly mix and sample these portions as described in Section 7. Each final sample shall consist of enough material

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to fill a 3.8 L (1 gal) container, at least, and be packaged as described in 8.4. Samples are not representative when water is used to lay the dust in chipping.

11.7 *Spent Materials*—Mix a portion of material from the inner and outer areas of the top, middle, and bottom of the leach for sampling, or take five portions from the pitch-hole as the spent material leaves the hole. Take the first portion 5 min after starting to pitch and the last portion 5 min before pitching is completed. Take the intermediate portions at equal time intervals between the first and the last portions. Each portion shall be of approximately equal size. Sample autoclaves continuously from the spent material conveyor. Thoroughly mix and sample these portions as described in Section 7. Each final sample shall consist of enough material to fill a 3.8 L (1 gal) container, at least, and be packaged as described in 8.4.

11.8 *Pasty Extracts*—Select the number of packages required for sampling. Insert a sampling tool (6.4) to the middle of each selected package three times, side by side, and empty the samples obtained into a suitable container. If the sampling tool fails, use a scoop or hatchet. Cut all portions into cubes of approximately 1.27 cm (0.50 in.) sides and thoroughly mix and sample as described in Section 7. Each final sample shall be of sufficient size to fill a 1.9 L (2 qt) Mason jar and packaged as described in 8.3.

11.9 *Liquid Extracts:*

11.9.1 Samples of liquid extracts are not representative when they have been frozen, or when steam has been passed directly into the extract. Add a suitable preservative to samples of liquid extract which are prone to ferment. Seal all samples immediately to prevent loss of moisture.

11.9.2 *Liquid Extracts in Barrels*—Determine the number of barrels to be sampled from Table 1 and take from evenly distributed parts of the total consignment. Before sampling, thoroughly mix the contents of the selected barrels by rolling. To assist in this mixing, withdraw two copper, or wooden, pailfuls of the extract before rolling, the barrels rolled, the extract in the pails returned, and the barrels again rolled. Take portions of at least 450 g (1 lb) each from each barrel through the bung-hole, avoiding contamination from the outside of the barrel. All such portions shall be of approximately equal size and collected in a suitable, covered container. Thoroughly mix the sample so obtained and take samples of at least 115 g (4 oz) and package as described in 8.2.

11.9.3 *Liquid Extracts in Tank Cars*—Sample the extract as follows, while the tank car is being loaded or unloaded. Take a 0.95 L (1 qt) portion from the intake or discharge 3 min after the extract has begun to flow, and take another 0.95 L (1 qt) portion at approximately 3 min before the flow ceases. Take three other 0.95 L (1 qt) portions at equal time intervals between these two. Transfer these five portions to a suitable container as soon as taken, cover to prevent evaporation or contamination, thoroughly mixed, and take and package samples of at least 115 g (4 oz) as described in 8.2.

11.10 *Tannery Liquors*—Mix the liquors by plunging, or other effective means, before sampling. If routine samples are taken daily for compositing, they shall be of equal size, and be mixed, and placed in covered containers. Samples of liquor for analysis shall be not less than 475 mL (1 pt) and be preserved with an anti-ferment. Recommended anti-ferments for this purpose are 0.5 g of an oil of mustard, oil of cloves, or beta-naphthol (powdered) per 0.95 L (1 qt).

11.11 *Sampler's Certificate:*

11.11.1 A sample drawn by an experienced sampler, adhering strictly to the methods of this practice shall be considered official. The following certificate shall accompany official samples:

> I (We), the undersigned sampler(s) of tanning material herein described:
>
> (see 11.11.2)
>
> do affirm that the samples were taken in strict accordance with the methods of this practice for the sampling of tanning materials.
>
> Signed (sampler)

11.11.2 The description shall have the following data, whenever possible: date sampled; kind of material; number of packages in the lot; distinguishing marks; number of packages sampled; weigher's lot number; any other marks descriptive of the lot sampled, such as car number, name of ship, order number, etc.

11.12 *Allowable Difference in Analysis*—The average of the tannin contents, found by the different laboratories on any sample taken by these methods of sampling shall be calculated. For extracts, the allowable difference between laboratories shall be 1.5 % of this average tannin content. For raw materials, the allowable difference between laboratories shall be 3 % of this average tannin content. Where the actual tannin contents, found by any two laboratories, differ from each other by an amount greater than the allowable difference calculated above, the analysis shall be repeated, if possible, and new analyses run until agreement is reached.

12. Keywords

12.1 sampling; tannin; tannin samples; tanning materials; vegetable tannin

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