Subpart B—TOBACCO

26 CFR 270.11: MEANING OF TERMS

Methods used in making chemical and smoking tests of reconstituted tobacco and products wrapped in such material. See ATF Procedure 73-5, below.

26 CFR 275.11: MEANING OF TERMS

Methods used in making chemical and smoking tests of reconstituted tobacco and products wrapped in such material. See ATF Procedure 73-5, below.

26 CFR 601.301: IMPOSITION OF TAXES, QUALIFICATION REQUIREMENTS, AND REGULATIONS (Also 26 CFR 270.11, 275.11)

Information is furnished about tests used by the Bureau in determining whether smoking products wrapped in reconstituted tobacco are cigars or cigarettes.

ATF Proc. 73–5

SECTION 1. PURPOSE.

This ATF Procedure describes some basic analytical methods used by the Bureau of Alcohol, Tobacco and Firearms in developing information to determine if specific reconstituted tobacco material is acceptable as a cigar wrapper, and to determine if a product wrapped in such material is a cigar or a cigarette for tax purposes.

SEC. 2. BACKGROUND.

.01 Before 1959 cigars produced in the United States were traditionally made with a wrapper of natural tobacco leaf. However, using ground or pulverized tobacco as the basic constituency industry researchers had by the late 1950's produced several forms of reconstituted tobacco sheet. Many of these sheets were submitted to the Bureau (then a part of the Internal Revenue Service) with a request they be accepted as wrappers for some cigars instead of natural tobacco leaf wrappers.

.02 The Bureau found that several of these reconstituted or "homogenized" tobacco materials did possess most of the essential characteristics of natural tobacco leaf then in use as cigar wrappers. It was therefore administratively held that those materials so found could be used as a wrapper for products taxable as cigars, provided the filler tobaccos were like those traditionally used in cigars and distinctly unlike cigarette filler tobaccos. Thereafter, as the technology of reconstituted tobacco material production and the technology of its use as both a binder and wrapper for cigars developed, many manufacturers began to produce cigars, both large and small, using this reconstituted tobacco material.

.03 In 1965 Congress specifically recognized and accepted reconstituted tobacco as a proper wrapper for cigars when it enacted Public Law 89-44 (79 Stat. 136), amending 26 U.S.C. 5702(a) and (b). In so doing Congress continued the principles of administration that acceptable wrapper materials for

cigars must possess the basic characteristics of tobacco and the filler tobaccos must not be like those used in cigarettes.

SEC. 3. GENERAL NATURE OF EXAMINATIONS AND TESTS

.01 In fulfilling its responsibilities under the taxing statutes the Bureau has developed analytical examinations and tests to provide objective information for use in ruling on individual wrapper materials and smoking products. This information is considered together with other relevant factors in (a) making distinctions between those materials which are reconstituted tobacco and those which do not possess the necessary tobacco character to be so categorized, (b) differentiating between the filler tobaccos typically used in cigars as opposed to those generally used in cigarettes, and (c) determining if a smoking product wrapped in reconstituted tobacco is taxable as a cigar or a cigarette under 26 U.S.C. 5701(a) and (b).

.02 The tests presently performed by the Bureau on both wrapper and filler materials consist of physical examinations, a six-solvent sequential extraction, determinations of ash, nicotine, various elements, and the pH (acidity-alkalinity). For the finished product, tests are also made to determine the pH of the smoke and to obtain the opinions of cigarette smokers as to whether they consider the product to be a cigar or a cigarette.

.03 While the following sections detail the examinations and tests generally performed by the Bureau at the time of publication of this procedure, it is expected techniques will continue to be refined and improved. Any significant changes in procedures will be announced as soon as practical after development and verification.

SEC. 4. WRAPPER EXAMINATIONS AND TESTS

Color	Visual examination with general description and statement as to whether it is in the range of natural leaf tobaccos traditionally used as a wrapper for American cigars.
Composition	Microscopic examination for tobacco particles, vegetable fibers, mineral particles, adhesives, other substances.
Cellulose Fibers	Microscopic examination may be reported as none, few, numerous; or chemical tests may be used.
Texture	Microscropic examination described as rough, smooth, etc.
Tensile Strength	Instrumentation can be used, but usually only a physical manipulation of stretching, tearing, etc. Generally reported as degree of dry and wet tensile strength.
Paper-like Qualities	Subjective evaluation based on appearance, tensile strength, number of fibers, general visual character, and evaluation of chemical composition as shown later.
Taste	Degree of tobacco-like character.

Burning ——— Described as more characteristic of tobacco or of paper.

Fragments of Tobacco ——— Microscopic examination, described as large, small, pulverized, none, etc.

Percent moisture
Percent total ash
Percent acid insoluable ash
Percent sodium
Percent potassium
Percent calcium
Other elements may be determined
pH of water extract
Percent nicotine
Sequential Differential Solvent Extractions as described in Section 7.08

Sec. 5 Filler Examinations and Tests

Composition ——— Microscopically observed character of filler, expressed as relative proportions of basic tobacco types.

Taste ——— Tobacco character, described as heavily fermented, type, fire-cured type, etc., and other descriptives such as added menthol, etc.

Odor before smoking ——— Expressed as to kind of tobacco product character, such as mild cigar-like, etc.

Odor when smoking ——— Expressed as to kind of tobacco product character such as mentholated cigarette-like, etc.

Percent moisture
Percent total ash
Percent acid insoluble ash
Percent sodium
Percent potassium
Percent calcium
Other elements may be determined
pH of water extract
Percent nicotine
Sequential Differential Solvent Extractions as described in Section 7.08

Sec. 6. Product Examinations and Tests

Diameter ——— In millimeters to the nearest tenth.

Length (Including filter) ——— In millimeters to the nearest tenth.

Weight of 1,000 (Including filters) ——— In pounds to nearest hundredth.

Added distinctive flavorings not otherwise reported, such as any included in the filter
pH of smoke
Opinions of cigarette smokers

.01 *Moisture Content (Method 1)*

Accurately weigh duplicate samples into previously dried and weighed platinum dishes. Spread out the sample over the bottom of the dish and dry for three days in an oven at 103 ± 2°C. Cool to room temperature in a dessicator containing silica gel and reweigh. Calculate the loss in weight as a percent of moisture.

$$\% \text{ Moisture} = \frac{\text{loss in weight} \times 100}{\text{weight of sample}}$$

.02 *Total Ash (Method 2)*

Carefully heat the samples from the moisture determination (Method 1) over a low flame until they stop smoking, taking care that the tobacco does not flame. Complete the ashing in an electric muffle furnace at 600°C for two hours. Lixiviate the ash with water if necessary and reheat in the furnace. Cool to room temperature in a dessicator containing silica gel and weigh the dish and contents. Calculate the percent total ash on a moisture-free basis.

$$\% \text{ Total ash} = \frac{\text{weight of total ash} \times 100}{\text{weight of moisture free sample}}$$

.03 *Acid Insoluble Ash (Method 3)*

Add 10 ml of 3 N HCl (1 + 3) to the ash (Method 2) in the platinum dish and evaporate just to dryness on a steam bath. Add another 10 ml of the 3 N HCl to the dish and warm on the steam bath. Decant the warm solution through Whatman #42 filter paper into a 100 ml volumetric flask. Repeat this operation with a second 10 ml portion of HCl. Transfer the residue to the filter paper and wash the dish and residue with hot distilled water until the volume approaches 100 ml. When cool, dilute to 100 ml and save for the determination of elements (Method 4).

Transfer the filter paper to the platinum dish and dry on a steam bath. Char the paper over a low flame and ignite in the muffle furnace at 600°C to constant weight. Cool to room temperature in a dessicator containing silica gel and weigh.

Calculate the percent acid insoluble ash on a moisture-free basis.

$$\% \text{ Ash (acid insoluble)} = \frac{\text{weight of acid insoluble ash} \times 100}{\text{weight of moisture free sample}}$$

.04 *Sodium, Potassium, and Calcium (Method 4)*

Apparatus: Atomic Absorption Spectrophotometer; Flame Spectrophotometer

Reagents: Standard Atomic Absorption Solutions

Method:

Use filtrate from the determination of the acid insoluble ash (Method 3). Dilutions will depend on the elemental concentrations under consideration. Unknowns and standards should have approximately the same concentration. If a flame spectrophotometer is used in calcium determination, it is necessary to simulate a standard with phosphate to minimize the phosphate effect. (Not necessary with sodium and potassium and not necessary for any element with atomic absorption spectrophotometer.)

.05 *pH Water Extract (Method 5)*

Apparatus: Digicord Photovolt pH meter with glass electrodes

Method:

Accurately weight 1 g of ground filler tobacco or wrapper material

(not dried) into a 250 ml Erlenmeyer flask. Using a pipette, add 50 ml of distilled water and swirl the flask to thoroughly wet the tobacco. Then add another 50 ml of water, washing down the sides of the flask. Stopper, and allow to stand for 24 hours with occasional shaking. Determine the pH of the slurry.

.06 *pH of Smoke (Method 6)*

Apparatus: Phipps and Bird 4-port smoking machine; Digicord Photovolt pH meter with glass electrodes

Method:

Cut 10 of the sample products to 60 mm (eliminating filter, if any) and smoke to a 5-mm butt length on a smoking machine collecting the particulate matter on a Cambridge filter. Remove the impregnated Cambridge filter and insert into a 25 ml syringe with a cotton plug in the base and fitted with a filter holder containing a 5.0 micron Metrocel filter. Muddle the Cambridge filter with 5 or 6 ml 95% ethanol and filter into a 25 ml volumetric flask. Repeat the extraction and elution process 3 more times with similar quantities of ethanol. (Extraction of soluble material from filter should be complete.) Make the solution to volume in the 25 ml flask with 95% ethanol. Remove 12½ ml of solution to a 50 ml volumetric flask and add 37½ ml of distilled water. Determine the pH of the ethanol-water solution.

.07 *Total Alkaloids (as Nicotine) (Method 7)*

Apparatus: Volatile Acid Still; Beckman D. U. Spectrophotometer

Reagents:

Alkali-Salt Solution—Dissolve 300 g of NaOH in 700 ml of water and saturate this solution with NaCl.

Hydrochloric Acid Solution.—Mix 1 volume of conc. HCl with 4 volumes of distilled water.

Standard Nicotine Solution.—Dissolve 1.0000 g. of Nicotine alkaloid in 1L of 0.05 N HCl.

Method:

Accurately weigh 0.1 g. of filler tobacco sample—or 0.4 g of wrapper material sample—and transfer to the inner chamber of the volatile acid still. Add 5 ml of the alkali salt solution, washing down the sides of the container with distilled water. Add 500 ml of water to the steam jacket and connect the still to a water cooled condenser.

Pipette 10 ml of the HCl solution into a 200 ml volumetric flask and place the flask so the condenser tube dips into the HCl solution. Collect about 190 ml of the distillate and dilute to volume with distilled water. (Add additional boiling water to the steam jacket when necessary.)

Dilute aliquots of the distillate, if necessary, with 0.05 N HCl so the absorbance at 259 mu is between 0.5 and 0.8 and read the absorbance at 236, 259, and 282 mu.

Calculate the percent total alkaloids on a moisture-free basis.

1. Correct the absorbance at 259 mu for background:

$$A_{259}\ (\text{corrected}) = 1{,}059\left[A_{259} - \tfrac{1}{2}(A_{236} + A_{282})\right]$$

2. % Total Alkaloids (as Nicotine) equals—

$$\frac{(A_{259}\ \text{cor.}) \times (\text{ppm std}) \times (0.02) \times (\text{dil. factor})}{(A_{259}\ \text{cor. std}) \times (\text{weight of moisture free sample})}$$

.08 *Sequential Differential Solvent Extractions (Method 8)*

1. The method is based on a series of extractions with solvents of

increasing extractability in the following order:

Reagents:

Petroleum ether, reagent grade (38° - 53°C)
Tetrahydrofuran, reagent grade
Acetonitrile, reagent grade
Ethyl alcohol, 95% USP
Dioxane-20% water (reagent grade and distilled respectively)
Water, distilled—One drop #820 anti-foam (Scientific Industries, Inc. 15 Park Street, Springfield, Mass. 01103) added per 100 ml of water. This water also used in dioxane-20% water solution.

Apparatus:

Standard Soxhlet extraction apparatus with a 125 ml flat bottom receiving flask; 25 mm by 85 mm extraction thimble with coarse porosity fritted glass; Precision Scientific six-unit electric extraction heater unit; Buchlet rotary evaporator; Mettler type H15 semi-micro analytical balance; standard convection oven; Wiley intermediate cutting mill.

2. Preparation of Sample:

The filler or wrapper samples are pulverized in a Wiley mill to pass a 20 mesh screen. The pulverized sample is made moisture-free by allowing the samples to remain in the oven for three days at 103 ± 2°C. The extraction thimble and 250 ml round bottom flask are heated in the oven at 103 ± 2°C. overnight and then allowed to come to room temperature inside a dessicator. One gram of 20 mesh moisture-free sample is transferred to a tared glass thimble and weighed. A small plug of cotton is then inserted into the thimble on top of the sample and the thimble and its contents are again weighed. The thimble is then placed into the thimble receptacle portion of the Soxhlet apparatus, and 100 ml of solvent and a chemically inert boiling chip are added to the 125 ml receiving flask.

3. Extraction Procedure:

The first solvent (100 ml petroleum ether) in the Soxhlet apparatus containing the sample is brought to boil and the extraction is is allowed to proceed for 24 hours. The heating is terminated and after cooling to room temperature the extraction solvent in thimble chamber and receiving flask is transferred to a tared 250 ml round bottom flask. The solvent is removed from the extract with a rotary evaporator. The tared flask containing the dried extract is placed in an oven overnight at 103 ± 2°C. The flask should then be removed and placed in a dessicator containing silica gel and allowed to come to room temperature. The tared flask containing the dried extract is then weighed on a semi-micro analytical balance. The same sample is then used sequentially for each of the five succeeding extraction solvents. In the case of petroleum ether only, the residual solvent in the thimble is removed from the sample by vacuum before proceeding to the next solvent (not necessary with solvents other than petroleum ether).

Calculate the percentage of each extract.

$$\% \text{ Extract} = \frac{\text{Weight of extract} \times 100}{\text{Weight of sample}}$$

Sec. 8 Evaluation of Data

.01 The meanings of the results of most of the physical examinations are self evident. The results of most of the chemical determinations of components are compared with data showing the proportions of the components normally found in tobaccos as reported in various technical pub-

lications and papers, such as those issued by the Department of Agriculture and those referenced in "Chemical Abstracts."

.02 The tobacco character of wrapper materials and the cigar (non-cigarette) character of filler tobaccos are calculated from the sequential differential solvent extraction data. In addition, the patterns of the different extractives are compared with extractives of the filler tobaccos of typical American cigars and cigarettes used in establishing the norms for the differential equations as shown in Tables I and II. The cigars and cigarettes used in developing the data in Tables I and II are various brands and styles from the domestic market and include at least one popular product of most major U.S. producers.

.03 The percentage of tobacco character of wrapper material is determined by comparing the total dry weight extractives from the sequential solvent extraction procedure with the comparable value (47.92%) for typical cigars as shown in Table I. (The cigar value is used for this calculation because most wrappers tested are produced from cigar type tobaccos.) The formula for this computation is:

$$\% \text{ Tobacco character} = \frac{\text{Total extract from sample wrapper} \times 100}{47.92}$$

.04 Tobacco character of filler tobaccos is computed the same as for wrapper material using the preceding formula. The cigar (non-cigarette) character of filler tobaccos can be computed from combinations of the six dry-weight extractives. While several combinations of comparisons are made, two are described below. These are the total of all six extractives, and the total of the extractives from tetrahydrofuran, acetonitrile, and ethyl alcohol. The computation for cigar (non-cigarette) character is based on the differences in the extractives between typical cigars and cigarettes, using in the formula the average values for cigar and cigarette extractives for the particular combination of solvents being compared, as shown in Tables I and II. The formula is expressed as:

$$\% \text{ Cigar character} = \frac{(\text{Average cigarette extract}) - (\text{Extract of unknown}) \times 100}{(\text{Average cigarette extract}) - (\text{Average cigar extract})}$$

The formula for all six extracts would therefore be:

$$\% \text{ Cigar character} = \frac{58.66 - (\text{Extract of unknown}) \times 100}{58.66 - 47.92}$$

The formula for the extracts from the three solvents tetrahydrofuran, acetonitrile, and ethyl alcohol, would be:

$$\% \text{ Cigar character} = \frac{(2.38 + 5.77 + 18.71) - (\text{same 3 extracts of unknown}) \times 100}{(2.38 + 5.77 + 18.71) - (2.47 + 3.68 + 9.10)}$$

$$\text{or } \frac{26.86 - (\text{extracts of unknown}) \times 100}{11.61}$$

Sec. 9 Smoking Test

Cigarette smokers, usually 100 or more, are asked to smoke a sample of the tobacco product and indicate on a questionnaire whether they consider the product a cigar or a cigarette. To eliminate any possible bias because of the order of items on the questionnaire, two versions are used in a 1 to 1 ratio for each test. The two versions of the

questionnaire are printed here as Exhibits A and B.

SEC. 10 INQUIRIES OR COMMENTS

Any inquiries or comments concerning this procedure should be addressed to the Director, Bureau of Alcohol, Tobacco and Firearms, Attention: Regulatory Enforcement, Washington, D.C. 20226.