TANNIC ACID

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SYNONYMS

Tannins (food grade), gallotannic acid, INS No. 181

DEFINITION

Consists of gallotannins obtained by solvent extraction from certain natural sources; the substance is not an acid in the chemical sense. The common name "Tannic acid" has been adopted to distinguish the commercial substance from other tannins, such as condensed tannins. These specifications relate only to hydrolysable gallotannins, i.e., those which yield gallic acid on hydrolysis. Hydrolysable gallotannins may be obtained from nutgalls, the excrescences which form on young twigs of various *Quercus* species, e.g., *Q. infectoria*; these include Chinese and Aleppo tannins. They may also be obtained from various Sumac species, e.g. *Rhus corieria*, *R. galabra*, *R. thypia*; these include Sicilian and American sumacs. All of these consist essentially of polydigalloyl esters of glucose. A further source of hydrolysable gallotannins is the seed pods of Tara (*Caesalpinia spinosa*); these tannins consist essentially of the polydigalloyl esters of quinic acid. These specifications do not apply to many other kinds of tannins which occur in nature, including condensed (non-hydrolysable) tannins and hydrolysable ellagitannins.

Assay

Not less than 96% on dried basis

DESCRIPTION

Amorphous powder, glistening scales or spongy mass, varying in colour from yellowish white to light brown; odourless or with a faint, characteristic odour

FUNCTIONAL USES

Clarifying agent, flavouring agent, flavour adjunct

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Soluble in water, acetone and ethyl alcohol; insoluble in ether; 1 g dissolves in about 1 ml of warm glycerine.

Colour reaction

To a 1 in 10 solution add a small quantity of ferric chloride TS. A bluish black colour or precipitate forms

Precipitate formation

A solution of the sample when added to a solution of either albumin or gelatin produces a precipitate

Test for gallic acid

Passes test after hydrolysis
See description under TESTS

PURITY

Loss on drying (Vol. 4)	Not more than 7% (105°, 2 h)
Sulfated ash (Vol. 4)	Not more than 1% Test 2.0 g of the sample (Method I)
Gums or dextrin	Dissolve 1 g in 5 ml of water, filter, and to the filtrate add 10 ml of ethanol; no turbidity is produced within 15 min
Resinous substances	Dissolve 1 g in 5 ml of water, filter and dilute the filtrate to 15 ml; no turbidity is produced
Condensed tannins	Not more than 0.5% See description under TESTS
Residual solvent (Vol. 4)	Not more than 25 mg/kg acetone or ethyl acetate, singly or in combination See description under TESTS
Lead (Vol. 4)	Not more than 2 mg/kg Determine using an AAS/ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4 (under "General Methods, Metallic Impurities").

TESTS

IDENTIFICATION TESTS

Test for gallic acid

Proceed as directed under *Chromatography (thin-layer chromatography)* (see Volume 4).

Sample preparation
Add 20 mg of tannase to 100 ml of 1 in 1000 solution of the sample. Incubate at 37° for 2 h.

Standard preparation
1 in 1000 solution of gallic acid.

Developing solvent
A mixture of 5 volumes of chloroform, 4 volumes of ethyl formate and 1 volume of formic acid.

Spraying reagent
Solution A: Prepare a 1% solution of ferric chloride ($FeCl_3$) in 10% ethanol
Solution B: Prepare a 1% solution of potassium ferricyanate ($K_3Fe(CN)_6$) in 50% ethanol
Mix 1 volume of solution A and 1 volume of solution B.

Adsorbent
Silica gel

Procedure

Apply 10 µl of the sample preparation and 10 µl of the standard preparation. Stop the development when the solvent front has advanced about 15 cm, dry in air, and spray the reagent. The Rf value of the sample and that of the reference standard are identical.

PURITY TESTS

Condensed tannins

The following HPLC method can be used for the determination of condensed tannins in the sample.

Sample preparation

Dissolve 200 mg of the sample in 100 ml of a mixture of 100 volumes of 10% methanol and 0.1 volume of phosphoric acid.

Standard preparation

Dissolve 10 mg of rutin in 100 ml of methanol.

Conditions

Column: Merck Hibar, LiChrosorb RP 18, 250 mm x 4.6 mm id, 5 µ (Art. 50333), or equivalent.

Eluent

A: 0.2% H_3PO_4 in water; B: 0.2% H_3PO_4 in methanol

Gradient elution:

Time (min)	% A	%B
0	90	10
65	25	75
66	0	100
75	0	100
76	90	10
95	90	10

Flow rate: 1 ml/min.
Detection wavelength: 350 nm
Injection volume: 10 µl

Calculate the content of the condensed tannins as follows:

$$\% \text{ condensed tannins (as rutin)} = \frac{A \times W_o}{A_o \times W} \times 100$$

where

A is peak area of the sample (condensed tannins);
A_o is peak area of the reference standard (rutin);
W is weight in mg of the sample taken; and
W_o is weight in mg of the reference standard taken.

Residual solvents

Reagents

- Acetone
- Ethyl acetate

Standard solutions

Place 1 g of acetone and 1 g of ethyl acetate in a volumetric flask and add water to total volume of 100 ml, and prepare 0.02 - 0.4

g/100 ml solutions by dilution of this solution.

Procedure

Place 1 g (1.0±0.1 g) of powdered sample in a sample vial. Add 5 µl of water to the sample vial and seal it quickly with a septum. Set the sample vial in a pre-conditioned gas chromatograph and start the analysis under the below-mentioned conditions.

Standard

Take 1 g of tannic acid free of solvent or with known residual solvent content in a sample vial, add 5 µl of the standard solution and seal it quickly with a septum. Set the sample vial in a pre-conditioned gas chromatograph and start the analysis under the following conditions and obtain standard curves for each solvent.

Determine by *Gas chromatography*, using a head space sampler under the following conditions:

- Column: 100% methyl polysiloxane 30 m x 0.53 mm id, 1 µm film thickness
- Column conditioning: Heat to 60° for 2-3 h with approximately 10 ml/min of nitrogen
- Carrier gas: Nitrogen
- Flow rate: 5 ml/min
- Detector: Flame ionization
- Temperatures
- injection port: 110°
- column: 40°
- detector: 110°

Head space sampler

- Sample heating temp.: 80°
- Sample heating time: 40 min
- Syringe temperature: 85°
- Sample gas injection: 0.4 ml

Calculation

$$C_i\ (\text{mg/kg}) = \left(\frac{A_i \times f_i}{W}\right)$$

where

C_i is the concentration of solvent (mg/kg);
A_i is peak are of solvent (Area units);
f_i is the slope of the standard curve (µg/Area units); and
W is weight of sample in g.

METHOD OF ASSAY

Sample test

Accurately weigh about 2.0 g of the sample (W), transfer to a 500-ml volumetric flask, add water to dissolve and make up to the volume with water. Transfer 100 ml of this solution into a 300 ml Erlenmeyer flask and add 7.2 g of Hide Powder (a suitable grade is available from L.H. Lincoln & Son, Inc., Tanning Materials, Coudersport, Pennsylvania, 16915 USA). Shake the flask for 20 min. Let stand for 10 min. and filter through a G_4-filter. The filtrate shall be clear. Pipette 50 ml of the filtrate into a tared crystallizing dish. Evaporate to dryness on a steam bath and heat in an oven at

105° for 1 h. Cool in a desiccator and weigh (a).

Blank test:
On each lot of Hide Powder (a suitable grade of Hide Powder may be obtained from L.H. Lincoln & Son, Inc., Tanning Materials, Coudersport, Pennsylvania, 16915 USA) a blank test has to be carried out. Weigh 7.2 g of Hide Powder EFT into a 300 ml Erlenmeyer containing 100 ml water. Proceed as directed for the Sample test, beginning with "Shake the flask for 20 min...". Cool in a desiccator and weigh (b).

Calculation

$$\%\,\text{Tannic Acid} = \left(\frac{(W-(a-b))\times 1000}{W}\right)$$

where

a is the weight of dry matter found in the sample test (g);
b is the weight of dry matter found in the blank test (g); and
W is weight of sample, on dry basis (g).

NOTE: the multiplication factor 1000 = 10 (500 ml/50 ml) x 100 (percent).