Thesis.

The Estimation of Nicotine in Virginia Tobaccos and Tobacco Products.

Work on the Insecticidal Value of Nicotine and Nicotine Decoctions.

Respectfully submitted to Prof. R. J. Davidson, for the degree of Master of Science.

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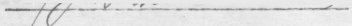

Statement of Problem.

Outline.

The determination of the nicotine content of tobaccos grown in Virginia and devising a practical method of extracting their nicotine.

1 Analyses of tobacco

2 Laboratory work showing results of different methods of extraction.

Tables

3 Field extraction

Conclusions

4 Results on Insects

Conclusions

5 Recommendations for making tobacco extracts

6 Bibliography.

For sometime numerous inquiries have been coming to the Experiment Station and the Crop Pest Commission from farmers and fruit growers in regard to some substance that they can grow or buy cheaply which will exterminate apple aphis. Some farmers ask the direct question in regard to the value of extracts made from tobacco stems and sweepings as a spray for trees infected with this insect. One fruit grower claimed that an extract of the stems killed all the lice. The object of this paper is to test out these suggestions and to be able to answer the questions asked by the farmer.

The first question was to determine the nicotine content of the tobaccos grown in Virginia and to devise a method which the farmer can utilize in extracting this nicotine for spraying purposes.

Tobacco, scientifically known as Nicotiana Tobaccum, is a plant of American origin. It was given the generic name, "Nicotiana" after Jean Nicot, a French ambassador to Portugal.

Nicotine, the native alkaloid of tobacco, is one of the most powerful and rapidly-acting poisons known to science. For this reason, various commercial extracts of tobacco, like "Black Leaf 40","Nikoteen", "Nikotiana", etc., have been put on the market in concentrated solutions which are to be used in varying dilutions, depending upon the use to which they are to be put.

Decoctions of tobacco have been used for a long time for exterminating certain insects from plants and animals. Because of its poisonous property nicotine is being used more and more each year as an insecticide. Hence, the farmers and the fruit growers are being confronted with the problem of finding some convenient, as well as cheap, form of nicotine not only to be used as a spray for trees and plants, but also as a dip for cattle and sheep.

The tobacco plant is successfully grown in Virginia and the by-products of the manufacture of tobacco are very important, not only for their nicotine but also for their fertilizing constituents. Among the types of tobacco grown in Virginia are the Narrow Leaf Orinocco, Olive, Blue Pryor, Sun-Cured, and White Stem. The Narrow Leaf Orinocco has the highest nicotine content (5.3 - 5.6%), found in any of the varieties analyzed in this laboratory and the flue cured tobaccos the lowest (2.3% - 3.7%). The cutters contain about 3% to 3.5% nicotine.

According to Bulletin No. 79, Bureau of Plant Industry, the nicotine content of stems varies from .12% in leaves cured on the stalk to .82% in leaves cured by the priming method. In the former, the nicotine content of the leaves was 1.46%, while in the latter it was 3.43%. Thus we see that stems from very light tobaccos should not be used in making nicotine solutions because of their very low nicotine content.

The nicotine[a] content of tobacco leaves varies from 1.07% to 5.21% in different tobaccos, depending upon the variety and other [illegible] conditions. Very rich soils high in nitrogenous materials tend to produce tobacco of high nicotine content. The maximum[b] nicotine content is reached just when the plant reaches maturity and decreases from this time, whether it is allowed to stand in the field or is harvested and cured. As the curing process goes on, there is a constant loss of nicotine from the tobacco plant and this loss continues even after the tobacco is cured, fermented and aged.

Chemical Methods of Analysis of Tobacco and Tobacco Products.

Two methods, the Kissling, adopted by the Association of Official Agricultural Chemists, and the Silicotungstate[c], suggested by Bertrand and Javillier, were used in analysing the samples. The Kissling method gave good results with the powdered tobacco and the

a. Bureau of Plant Industry. Bulletin 79. Pages 14-21.
b. Bureau of Plant Industry. Bulletin 141. Page 6.
c. R. M. Chapin, Bureau of Animal Industry. Bulletin 133.

concentrated extracts, such as "Black Leaf 40", etc., but could not be used in case of dilute aqueous extracts with any degree of accuracy. The per cent of nicotine in aqueous extracts by the Kissling method was higher than by the Silicotungstate method, due to decomposition products forming ammonia and other bases. To obviate this difficulty, the Silicotungstate Method was used in making analyses of all of the aqueous extracts from stems or leaves.

A Comparison of Results Obtained by Analyzing Aqueous Extracts by the Two Methods.

The Kissling method consists of two series of operations, (1) extraction of the material with ether and (2) distillation with steam and titration.

[a]The Kissling Method Adopted by the Acccciation of Official Agricultural Chemists.

Weigh from 5 to 6 grams of tobacco extract, or 20 grams of finely powdered tobacco, which has been previously dried at 60° C., so as to allow it to be powdered, into a small beaker. Add 10 c.c. of the alcohol-soda solution (6 grams NaOH, 40 c.c. water, 60 c.c. of 90% alcohol) and follow, in the case of the tobacco extract, with enough chemically pure powdered calcium carbonate to form a moist but not lumpy mass. Mix the whole thoroughly. Transfer this

a. Association of Official Agricultural Chemists. Bulletin 107. page 32.

to a Soxhlet extractor and exhaust for about five hours with ether. Evaporate the ether at a low temperature by holding over the steam bath, and take up the residue with 50 c.c. of the dilute sodium hydroxid solution (4 grams NaOH, 1000 c.c. Water). Transfer this residue by means of water to a Kjeldahl flask, capable of holding about 500 c.c., and distil in a current of steam, using a condenser through which water is flowing rapidly. Use a three-bend outflow tube, a few pieces of pumice, and a small piece of paraffin, to prevent bumping and frothing. Continue the distillation until all the nicotin has passed over, the distillate usually varying from 400 to 500 c.c. When the distillation is complete, only about 15 c.c. of the liquid should remain in the distillation flask. Titrate the distillate with standard sulphuric acid, using phenacetolin or cochineal as indicator. One molecule of sulphuric acid is equivalent to two molecules of nicotin.

This method is subject to one grave drawback, i.e., tobacco and tobacco extracts nearly always contain some ammonia, pyridin basis and amins, all of which would count as nicotine if not removed before titration.

It was found with one sample of tobacco that five hours were not sufficient time to extract with ether. A second extraction gave .1% nicotine.

The Kissling method gave an abnormally high result for the aqueous extracts. The sum of the apparent amounts of nicotine in the solutions was more than that which the stems contained to

start with. This was undoubtedly due to the ammonia which was formed in the extracts and was not volatilized during the extraction with ether. In the Bertrand and Javillier method, Silicotungstic acid is used but is expensive. In order to obviate the use of large amounts of this substance, aliquot portions containing small amounts of nicotine should be used in making determinations. This method can be used with tobacco extracts containing only small amounts of nicotine giving very close duplicates where the Kissling method would not be applicable, owing to errors which would arise because of the use of small amounts handled. Aside from this, it is a gravimetric method giving close duplicates and is easy of manipulation.

Method Used.[a]

This method naturally divides itself into three parts: (1) distillation, (2) precipitation, and (3) ignition.

Silicotungstate Method.

Weigh out such an amount of the preparation as will contain preferably between 1 and 2 grams of nicotin, except in the case of extracts high in extraneous matter, where not in excess of 30 grams should be employed; wash with water into a 500 c.c. round-bottomed flask; add 1 to 1½ grams paraffin, a few small pieces of pumice, and caustic soda solution to strong alkalinity (5 to 10 c.c of NaOH solution, 1 to 2). Distill in a rapid current of steam

a. Bureau of Animal Industry. Bulletin 133. P. 21-22.

through a three-bend connecting tube, a condenser, and adapter into 10 c.c. of dilute hydrochloric acid (1 to 4) in a capacious flask. When distillation is well started, apply heat to the distillation flask (a Babo funnel is most convenient) to reduce the volume of liquid as far as practicable without bumping or undue separation of insoluble matter. Continue distillation until a few cubic centimeters of the distillate collected from the condenser after removal of the adapter show no cloud or opalescence when treated with a drop of silicotungstic acid solution followed by a drop of dilute hydrochloric acid (1 to 4). Prove alkalinity of the residue in the distillation flask with phenophthalein solution or litmus paper.

Make the distillate to convenient volume, mix well and pass through a large dry filter, discarding a liberal first portion of the filtrate, and test a portion with methyl orange to assure its acidity. Pipett into a beaker an aliquot containing about 0.1 gram nicotin, add for each 100 c.c. of liquid 3 c.c. of dilute hydrochloric acid (1 to 4) - or more, if indicated necessary by the test with methyl orange - and add 1 c.c. of a 12 per cent solution of silicotungstic acid for each 0.01 gram nicotine supposed to be present. Stir thoroughly and let stand for eighteen hours. Then stir up the precipitate, making sure that it settles quickly and completely in crystalline form; filter and wash with cold water containing 1 c.c. of concentrated hydrochloric

acid per liter. Test the first portion of the filtrate with a few drops of nicotin distillate to prove excess of silicotungstic acid.

Continue washing for two or three fillings of the filter after no more opalescence appears when a few cubic centimeters of fresh filtrate is tested with a few drops of nicotin distillate. Bring the wet paper and precipitate into a weighed platinum crucible, using a scrap of moistened filter paper to transfer any precipitate which may have crept up the sides of the funnel; dry carefully, carbonize, and finally burn off the carbon at as low a temperature as possible. Gradually increase the heat and occasionally rotate the crucible to expose all parts of the residue. At last ignite the inclined crucible thoroughly over the full heat of a Bunsen burner, finishing with five to ten minutes - not longer - over a powerful Teclu burner, or five minutes over a moderately powerful blast lamp. Cool in a disiccator. The weight of the residue multiplied by 0.114 afford the weight of nicotine in the aliquot taken for precipitation. If the highest possible degree of accuracy is desired, filter the precipitate on a weighed Gooch crucible, dry at 125° C., and weigh the anhydrous nicotine silicotungstate.

We found that it was not necessary to let stand more than one hour after precipitation. For [illegible], by precipitating, stirring and filtering as soon as the precipitate settles clear, the results were the same.

A combination of the two methods gave good results in making analyses of tobacco stems or leaves. The material was extracted in a Saxohlet apparatus with ether and distilled as in the Kissling method. The distillate was made up to a definite volume, an aliquot portion measured out, precipitated, allowed to stand over night, filtered, washed, and ignited to constant weight. Since ammonia and amids are not precipitated by silicotungstic acid, the removal of ammonia during the extraction is not necessary and any ammonia or amids which may be formed during the distillation from amins does not interfere with the work of analysis.

Table No. I gives the results of analyses which were made in this laboratory of a number of Virginia tobaccos, stems, and sweepings. From our analyses, we found that the nicotine content of stems did not fall below .48% and was not above .609%. Analyses of stems by Johnson[a] showed a variation of .52% - .69% of nicotine. Our analyses of leaves of Virginia tobaccos gave a variation of 2.47% - 5.62% of nicotine.

a. CONN. Agr. Exp. Sta. 16th An. Report. 1892. P. 31-34. 1893.

Table I.

Nicotine Content of Virginia Tobaccos.

Kind.	Where from.	How Cured.	%Nicotine.
1. Stems	Richmond, Va.		.481
2. Stems	Danville, Va.		.609
3. Sweepings	Danville, Va.		.884
4. N. L. Orinocco	Appomattox, Va.	Open Fire.	5.335
5. Olive	Powhatan, Va.	Open Fire.	3.357
6. Light	Danville, Va.	Flue	2.984
7. Sweepings	Ky.Tob.Prod.Co. Louisville, Ky.		.735
8. Smoker	Chatham, Va.	Flue	2.3066
9. Wrapper	Chatham, Va.	Flue	3.0505
10. Cutter	Chatham, Va.	Flue	3.466
11. Dark	Appomattox, Va.	Sun	2.835
12. N.L.Orinocco	Bowling Green, Va.	Sun	5.629
13. Medium Smoker	Chatham, Va.	Sun	3.766
14. Common Smoker	Chatham, Va.	Sun	2.4705

TABLE II. Nicotine Preparations.

Name	Name and Address of manufacturer	Guaranteed Analysis	Found
Nikotiana	Alphine Mfg. Co., Madison, N. J.	12%	11.42%
Alphine	Alphine Mfg. Co., Madison, N. J.	.9%	1.27%
Hammonds Tobacco Extract	Hammond's Slug S. Wks, Fishkill-On-Hudson, N.Y.	4%	3.986%
Hammonds Trip Juice No. 2.	"	1%	1.067%
Nikoteen	Nicotine Mfg. Co., St. Louis, Mo.	30%	30.07%
"Black Leaf 40"	Ky. Tob. Prod. Co., Louisville, Ky.	40%	41.66%
"Nico-Fume"	"	40%	38.71%

Table No. II gives the Nicotine content of the nicotine preparations which are sold for use in exterminating plant lice, etc.

Laboratory Work Showing Results of Different Methods of Extraction.

In preparing tobacco extracts in the laboratory the vessels in which they were made were left open. With such a method of cooking there will always be a loss of water by evaporization as well as of nicotine if the water is allowed to boil. This loss can be reduced to a minimum by not letting the solution boil. The diminished amount of juice which is obtained after cooking is due not only to water which is taken up by the leaves or stems, but also to that which is lost by volatilization during cooking.

Notes on the Preparation of Tobacco Extracts.

Extract No. 1. Stems containing .609% nicotine were soaked 24 hours in water in the proportion of 62½ lbs. to 50 gallons, heated to boiling, cooled and strained. Yield of juice, 27 gallons. The juice contained .0702% nicotine or 41.42% nicotine of the stems was extracted.

Extract No. 2. Stems containing .609% nicotine were soaked 24 hours in water in the proportion of 62½ pounds to 50 gallons and strained. Yield of juice, 36¾ gallons .0 727% nicotine content, 57.87% total nicotine of the stems was extracted.

Extract No. 3. 62 1/2 pounds of stems containing .609% nicotine were placed in 50 gallons of water, heated to boiling in 30 minutes, kept just below boiling 30 minutes, cooled and strained.

Yield of juice 25¾ gallons of .0744% nicotine, 42.27% of the total nicotine being extracted.

Extract No. 4. Stems containing .609% nicotine were soaked 24 hours in water in the proportion of 31¼ pounds per 50 gallons and strained. Yield of juice 42½ gallons of .0394% nicotine content, 73.44% total nicotine being extracted.

Extract No. 5. Stems of .609% nicotine content were heated to boiling in water in the proportion of 31¼ pounds per 50 gallons 25 minutes being required to bring to boiling point. It was kept just below boiling 30 minutes, cooled and strained. Yield, 31¼ gallons of juice of .0442% nicotine content, 60.15% total nicotine being extracted.

Extract No. 6. Sweepings containing .884% of nicotine were soaked 24 hours in water in the proportion of 62½ pounds per 50 gallons and strained. Yield, 44½ gallons juice of .1052% nicotine, 70.73% total nicotine being extracted.

Extract No. 7. Sweepings of .884% nicotine content were soaked 24 hours in water in the proportion of 62½ pounds per 50 gallons. At the end of this time it was heated to boiling in 25 minutes, kept just below the boiling point 30 minutes, cooled and strained. The yield of juice was 39¾ gallons .1303% nicotine content, 78,21% total nicotine being extracted.

Extract No. 8. Sweepings of .884% nicotine content, and water in the proportion of 62½ pounds sweepings per 50 gallons were brought to boiling in 30 minutes, kept just below boiling for 30 minutes, cooled and strained. The yield of juice was 39 gallons

of .116% nicotine content, 68.43% total nicotine being extracted.

Extract No. 9. Sweepings containing .884% nicotine and water in the proportion of 31¼ pounds sweepings per 50 gallons of water were soaked 24 hours and strained. The yield of juice was 46¾ gallons of .0548% nicotine content, 77.79% total nicotine being extracted.

Extract No. 10. Sweepings containing .884% nicotine in water in the proportion of 31¼ pounds sweepings per 50 gallons water, were brought to boiling in 25 minutes and kept just below the boiling point 30 minutes, cooled and strained. The yield of juice was 45¼ gallons of .0628% nicotine content, 84.86% total nicotine being extracted.

Extract No. 11. Stems containing .609% of nicotine were heated to boiling in water in the proportion of 59 pounds per 50 gallons. The mixture was heated to boiling in 45 minutes, kept just below boiling for 30 minutes, cooled and strained. The yield of juice was 19 gallons of .0841% nicotine content, 37.16% total nicotine being extracted.

Extract No. 12. Stems containing .609% nicotine. The stems were ground before using, and heated to boiling with water in the proportion of 59 pounds per 50 gallons, 35 minutes being required to heat to this point. It was then kept just below boiling 30 minutes cooled and strained. The yield of juice was 30¼ gallons of .0894% nicotine content, 62.28% total nicotine being extracted.

Extract No. 13. Stems of .609% nicotine content and water in the proportion of 29½ pounds per 50 gallons were heated to boiling in 30 minutes and kept just below this point for 30 minutes, cooled and strained. The yield was 35¼ gallons of .0476% nicotine content, 77.91% total nicotine being extracted.

Extract No. 14. Stems containing .609% nicotine and water at the rate of 39¼ pounds per 50 gallons. The stems were soaked 24 hours in 2/3 of the water and then the remainder of the water was added, brought to boiling, cooled and strained. The yield of juice was 21¾ gallons of .0638% nicotine content, 48.52% being extracted.

Extract No. 15. Stems of .609% nicotine content and water in the proportion of 39¼ pounds per 50 gallons. The stems were soaked 24 hours in 2/3 of the water and strained. The residue was soaked 2 hours in the remainder of the water and strained. The two solutions were mixed. The yield of juice was 34½ gallons of .0642% nicotine content; 76.47% total nicotine being extracted.

Extract No. 16. Flue cured leaves of 3.05% nicotine content were heated to boiling in 35 minutes in water in the proportion of 29½ pounds per 50 gallons, cooled and strained. The yield of juice was 36 gallons of .2576% nicotine content, 85.76% total nicotine being extracted.

Extract No. 17. Sun cured leaves of 2.835% nicotine content were ground and mixed with water in the proportion of 29½ pounds

per 50 gallons, brought to boiling in 25 minutes, cooled and strained. The yield of juice was 38½ gallons of .2369% nicotine content, 91.37% of the total nicotine being extracted.

Extract No. 18. Sun cured leaves of 2.835% nicotine content were heated to boiling in water in the proportion of 29½ pounds per 50 gallons, cooled and strained. The yield of juice was 36 gallons of .1998% nicotine content, 72.11% total nicotine being extracted.

Extract No. 19. Sun cured leaves of 2.835% nicotine content were soaked 24 hours in water in the proportion of 29½ pounds per 50 gallons, brought to boiling in 30 minutes, cooled and strained. The yield of juice was 31 gallons of .2524% nicotine content, 78.54% total nicotine of the leaves being extracted.

Extract No. 20. Sun cured leaves of 2.835% nicotine content were heated to boiling in 20 minutes in distilled water in the proportion of 10½ pounds per 50 gallons, cooled and strained. The yield of juice was 38½ gallons of .0774% nicotine content, 83.19% total nicotine being extracted.

Extract No. 21. The same as Extract No. 20, with one exception. Tap water was used instead of distilled water. The yield of juice was 39½ gallons of .0824% nicotine content, 90.89% total nicotine being extracted.

Extract No. 22. Tobacco sweepings of .884% nicotine content and distilled water in the proportion of 33 pounds per 50 gallons were heated to boiling in 30 minutes and kept just below this poin 30 minutes, cooled and strained. The yield of juice was 47¾ gallons of .0666% nicotine content, 90.65% total nicotine

being extracted.

Extract No. 23. The same as Extract No. 22, except that tap water was used. The results were the same.

Table III gives a tabulated summary of the work done on the various methods of extraction.

Table III.

	Kind of Tobacco.	%of Nicotine in Tobacco.	Lbs. per 50 gals. of water.	Volume of Extract in Gallons.	%of Nicotine in Extract.	Total % of Nicotine extracted.
1	Stems	.609	62½	27	.0702	41.42
2	"	.609	62½	36¾	.0727	57.87
3	"	.609	62½	25¾	.0744	42.27
4	"	.609	31¼	42½	.0394	73.44
5	"	.609	31¼	31¼	.0442	60.15
6	Sweepings	.884	62½	44⅛	.1052	70.73
7	"	.884	62½	39¾	.1303	78.21
8	"	.884	62½	39	.116	68.43
9	"	.884	31¼	46¾	.0548	77.79
10	"	.884	31¼	45¼	.0628	84.86
11	Stems	.609	59	19	.0841	37.16
12	"	.609	59	30¼	.0894	62.28
13	"	.609	29½	35¼	.0476	77.91
14	"	.609	39¼	21⅝	.0638	48.52
15	"	.609	39¼	34¼	.0642	76.47
16	Leaves	3. 05	29½	36	.2576	85.76
17	"	2.835	29½	38½	.2369	91.37
18	"	2.835	29½	36	.1998	72.11
19	"	2.835	29½	31	.2524	78.54
20	"	2.835	10½	38½	.0774	83.19
21	"	2.835	10½	39½	.0824	90.89
22	Sweepings	.884	33	47¾	.0661	90.65
23	"	.884	33	47¾	.067	90.65

From the above tabulated experiments it was found that by soaking the material over night 70% of the nicotine of sweepings was extracted while, by soaking over night and bringing to boiling, 78% was available. However, the yield of juice was diminished by cooking. By heating to boiling, cooling and straining, only 68% of the nicotine was available. We found that practically the same results were obtained with stems as with sweepings. The stems took up about 25% of the water added to make the extract and diminished the yield of solution accordingly. The water which the stems take up increases their bulk so that it is not practical to press them out. This was tried but was not successful.

This work was duplicated with larger amounts of stems in a stock feed boiler. It was found that by soaking 24 hours, 78% of the total nicotine wwas extracted and that from 50 gallons of water used 38 3/4 gallons of juice were obtained.

A Comparison of the Effect of Distilled Water and of Tap Water in Making Tobacco Extracts.

Water from limestone regions contains salts of lime which in many instances is the bicarbonate. On being boiled, this breaks down into the normal carbonate which imparts an alkaline reaction to the water and CO_2 is driven off.

Since nicotine, ammonia, and pyradine bases are volatilized by heating with weak alkalies it was thought probable that water contain-

ing such basic material, when used in making tobacco extracts, would drive off some of the nicotine during the process of cooking. In order to compare the effect of distilled water and of water containing lime salts in extracting nicotine, two parts of sweepings of 300 grams each were weighed out. One was treated with one gallon of tap water and the other with one gallon of distilled water. The two were brought to boiling in 30 minutes and kept just below boiling for 30 minutes, cooled and strained. Yield in each case was 122 ounces. The amount of nicotine in each was .066%.

The result shows no difference. The tap water was a hard limestone.

Preparation of Tobacco Extracts for Spraying.

Tobacco extracts were made by three methods at the plant for making spray solutions for the Experiment Station. One extract was made by cooking with live steam, another by cooking in an open kettle, and the third by soaking the stems in cold water.

The equipment for cooking with live steam consists of an upright boiler so arranged that live steam is passed through the tobacco in barrels by means of steam pipes. These pipes are perforated and the live steam passes out through these perforations and into the solution which is to be cooked. By the use of a boiler of this type, a series of six or eight barrels may be cooked at one time.

In making tobacco solutions by this method it was found that when starting with 33 gallons of water and cooking two hours, 5¾ gallons were added, due to the condensation of steam. The amount of steam condensed is directly proportional to the time in which the cooking is done and is indirectly proportional to the steam pressure of the boiler.

The iron kettle cooker consists of an ordinary iron kettle surrounded by a jacket which serves to prevent any great loss of heat by radiation. In cooking the material in a kettle over an open fire, there will be a loss of water by evaporation; also, since it is hard to control the temperature, a part of the nicotine may be lost by volatilization because nicotine is volatile.

Solution No. 1. The solution was made from sun cured leaves of 2.835% nicotine content. 12½ pounds of leaves were put in a barrel with 25 gallons of water, heated to boiling, and kept just below this point two hours. It was then let stand 18 hours and drained off. The yield of juice was 24 gallons of .1282% nicotine content; 72.44% total nicotine being extracted. 4½ gallons of water were absorbed by the leaves and 3½ gallons were added to the condensation of steam during the cooking.

Solution No. 2. This solution was made from stems containing .481% nicotine. 33 pounds of stems and 33 gallons of water were put in a barrel and cooked just below boiling for two hours and strained. The yield of juice was 29½ gallons of .055% nicotine content; 85.29% total nicotine being extracted. 9¼ gallons of water were taken up by the stems and 5¾ gallons were added, due to condensation of steam.

Solution No. 3. Prepared by using an iron kettle which is used for making lime-sulphur solution. 30 pounds of stems of .609% nicotine content were placed in an iron kettle with 30 gallons of water, brought to boiling and kept just below this point for two hours. After 24 hours the juice was drained off. The yield was 14½ gallons of .074%

nicotine conte;t 48.46% total nicotine being extracted. 15 gallons of water were taken up by the stems.

Solution No. 4. This experiment was made with the object of finding out if it is practical to make nicotine extracts for spraying by merely letting stems soak; and also, if the nicotine of tobacco stems can be extracted as well by letting them soak in water for 24 hours as to cook them for two hours. 30 pounds of stems, of .481% nicotine content were soaked 24 hours in 30 gallons of water. The juice was drained off. The yield was 23¼ gallons of .0579% nicotine content; 78% being extracted.

Table IV gives a brief summary of the field work.

Table IV.

	Kind.	%Nicotine in TOB.	How Prepared.	Lbs. of tobacco taken.	Gallons of water used.	Volume of extract. Gals.	%Nicotine in Ext.	Total% of Nicotine extd.
1	Leaves	2.835	Steam Cooker	12½	25	24	.1282	72.44
2	Stems	.481	"	33	33	29½	.055	85.29
3	"	.609	Open Kettle	30	30	14½	.074	48.46
4	"	.481	Soaked	30	30	23¼	.0579	78.00

As a result of the field work it was found in making tobacco extracts only necessary to soak the material 24 hours with frequent agitations. The total yield is nearly as great as that which is obtained by the steam cooker, but the volume is not so large as that obtained by the steam cooker because of the condensation of steam by the latter method. However, this diminished volume of yield is more than compensated for by the cheapness of the method over that of the steam cooker. The method of cooking with an open iron kettle is not to be recommended because of the small amount of extract obtained, as well as the small yield of nicotine.

We found that when we soaked the leaves or stems, twenty four hours, the volume of the extract and the per cent of nicotine obtained were about equal to the best results obtained by cooking.

The farmer, therefore, can soak the leaves in barrels for 24 hours with frequent stirring and get equally as good results as if he heated the water to boiling. This will enable him to prepare a number of barrels and in this way he will have enough to spray a large orchard. However, we found that fermentation took place rapidly and the extract is liable to spoil if kept over two or three days. He could prevent this fermentation, however, by using some antiseptic, like Bi-Chloride of Mercury or formaldehyde, but this is not to be recommended owing to the cost.

From the above work it seems that it is practical, as well as economical, for an apple grower to make his own nicotine spray solution from tobacco stems which may be obtained at about $20 per ton. This would make the solution cost about $1 per hundred gallons, outside of the cost of labor while the amount of "Black Leaf 40" necessary to make the same strength solution would cost $1.50.

Aside from the value of tobacco stems for their nicotine content, they make an excellent fertilizer. The fertilizing constituents are:

Potash (K_2O)	5 - 8%
Nitrogen	2 - 3%
Phosphoric Acid (P_2O_5)	3 -5%

A large part of the nitrogen is eliminated during the extraction but all of the phosphoric acid and some of the potash remains in the stems after the extraction. Putting a value of five cents per pound on the K_2O and two and one half cents per pound on the phosphoric acid, at the lowest estimate these two constituents alone are worth at least thirty cents per hundred pounds as a fertilizer.

Not only are tobacco stems extracted valuable as a fertilizer but also when put on the ground around the trees may deter insects, such as the woolly aphis from attacking their roots. Aside from the nicotine content one can safely put the value of stems at fifty cents per hundred pounds.

Where the whole tobacco plant can be grown or obtained cheaply, it is advisable to use fifty pounds of the dried material per 100 gallons of water and then add one part of water to each part of solution obtained. This will give a solution of more than sufficient strength to kill apple aphis.

Work on the Insecticidal Value of Nicotine.

The value of nicotine as an insecticide has long been known. In Europe its value has been appreciated. In this country, companies have been formed to manufacture nicotine solutions. In some of their concentrated extracts, the nicotine is in the form of a salt (Nicotine sulphate) while in others it is in the free state.

If nicotine can be obtained at a small cost by buying tobacco stems or leaves, the fruit growers will be able to produce high grade fruit cheaper than if they had to pay a high price for the commercial products.

In order to test out the efficiency of different nicotine extracts for killing apple aphis, spray mixtures of various strengths were prepared and used on the green apple aphis.

About the first of April, numerous cuttings were collected from the orchard and brought to the laboratory. The cuttings with a known number of lice, were placed in dishes and sprayed with the different strength solutions by a hand sprayer. After standing for different intervals of time, each dish was examined with a magnifying glass and the number of lice living and dead recorded. Afterwards the laboratory spraying was duplicated in the orchard. The number of lice on the buds were counted and sprayed with the same strength nicotine solution. The field results confirmed those obtained in the laboratory.

Table VI gives the results of the work done both in the laboratory and in the field. The kind of spray and its effect on the lice after a stated time is recorded.

TABLE VI.

Materials Used.	Place.	Time between spraying and counting.	No. Lice Sprayed.	No. Dead.	No. Alive
1. B.L.40 ¾-100 gal. 1-8 Con.L.S	Lab.	24	10	10	0
2. B.L.40 ¾-100 gal. L&S 1-40	"	24	10	9	1
3. B.L.40 ¾-100 gal. L&S 1-40 Soap 4-100	"	24	10	7	3
4. B.L.40 ¾-100 gal. L&S 1-40 Soap 4-100	Field	24	12	10	2
5. Homemade Nicotine .05% L&S 1-40	Lab	24	10	10	0
6. H.M.Nico. .05% Soap 4-100	"	24	9	7	2
7. .1% Nicotine	"	24	10	10	0
8. .08% "	"	24	20	20	0
9. Check on No. 8	"	24	10	0	10
10. Nicotine 05% Soap 4-100	Field	48	21	21	0
11. Nicotine .025% Soap 4-100	"	24	27	27	0
12. Nicotine.0125% Soap 4-100	"	24	27	21	0
13. Nico. 05% L&S 1-40 Soap 4-100	"	24	44	8	36
14. B.L.40 ¾-100 gal. L&S 1-40 Soap 4-100	"	24	12	10	2
15. L&S 1-40	Lab	24	10	1	9
16. L&S 1-40 Soap 1-50	"	24	9	2	7
17. L&S 1-40 Soap 2-50	"	24	10	3	7
18. B.L.40 ¾-100 L&S 1-40 As.of Lead 5-100	Field	48	27	27	0
19. B.L.40 ¾-100 As. of Lead 5-100	"	48	14	14	0
20. H.M.Nicotine.05% L&S 1-40 As. of Lead 5-100	"	48	32	32	0
21. H.M.Nico.05% As.of Lead 5-100	"	48	14	14	0

22. B.L.40 .01% Soap 4-100	Lab	48	8	6	2
23. B.L.40 .02% Soap 4-100	"	48	8	8	0
24. B.L.40 .03% Soap 4-100	"	48	10	10	0
25. B.L.40 .04% Soap 4-100	"	48	10	10	0
26. B.L.40 .05% Soap 4-100	"	48	10	10	0
27. H.M.Nico. .01% Soap 4-100	"	24	10	10	0
28. H.M.Nico. .02% Soap 4-100	"	24	10	10	0
29. H.M.Nico. .03% Soap 4-100	"	48	20	20	0
30. H.M.Nico. .04% Soap 4-100	"	24	12	12	0
31. H.M.Nico. .05% Soap 4-100	"	24	10	10	0
32. B.L.40 3 oz-100 Soap 8-100	"	24	9	9	0
33. B.L.40 6 oz-100 Soap 8-100	"	24	9	9	0
34. B. L.40 12 oz-100 Soap 8-100	"	24	10	10	0
35. Soap 8-100	"	18	9	12	7
36. Soap 8-100	"	42	9	4	5
37. Soap 8-100	"	72	9	7	2
38. B.L.40 1 oz-100 Soap 8-100	"	18	9	6	3
39. B.L.40 1 oz-100 Soap 8-100	"	42	9	7	2
40. B.L.40 1 oz-100 Soap 8-100	"	72	9	9	0
41. B.L.40 2 oz-100 Soap 8-100	"	18	10	7	3
42. B.L.40 2 oz-100 Soap 8-100	"	42	10	9	1
43. B.L.40 2 oz-100 Soap 8-100	"	72	10	10	0
44. B.L.40 3 oz-100 Soap 8-100	"	18	11	7	4
45. B.L.40 3 oz-100 Soap 8-100	"	42	11	9	2
46. B.L.40 3 oz-100 Soap 8-100	"	72	11	11	0
47. B.L.40 6 oz-100 Soap 8-100	"	18	11	11	0
48. B.L.40 12 oz-100 Soap 8-100	"	18	12	12	0
49. Nico-Fume 1 oz- 100 Soap 8-100	"	18	11	3	8

50. Nico-Fume 1 oz-100 Soap 8-100	Lab	42	11	7	4
51. Nico-Fume 1 oz-100 Soap 8-100	"	72	11	9	2
52. Nico-Fume 2 oz-100 Soap 8-100	"	18	14	12	2
53. Nico-Fume 2 oz-100 Soap 8-100	"	42	14	12	2
54. Nico-Fume 2 oz-100 Soap 8-100	"	72	14	14	0
55. Nico-Fume 3 oz-100 Soap 8-100	"	18	11	9	2
56. Nico-Fume 3 oz-100 Soap 8-100	"	42	11	9	2
57. Nico-Fume 3 oz-100 Soap 8-100	"	72	11	11	0
58. Nico-Fume 6 oz-100 Soap 8-100	"	18	11	8	3
59. Nico-Fume 6 0z-100 Soap 8-100	"	42	11	11	0
60. Nico-Fume 12 oz-100 Soap 8-100	"	18	10	7	3
61. Nico-Fume 12 oz-100 Soap 8-100	"	42	10	8	2
62. Nico-Fume 12 oz-100 Soap 8-100	"	72	10	10	0
63. Stem Solution. .05% nicotine	Field	24	6	m6	0
64. Stem Solution .027% Nicotine	"	24	10	10 -	0
65. Stem Solution .018% Nicotine	"	24	8	8	0
66. Leaf Solution .064% Nicotine	"	24	3	3	0
67. Leaf Solution .042%Nicotine	"	24	5	5	0
68. Leaf Solution .032%Nicotine	"	24	3	3	0
69. Leaf Solution .025%Nicotine	"	24	12	5	7
70. Leaf Solution .021%Nicotine	"	24	5	1	4
71. Leaf Solution .018%Nicotine	"	24	11	5	6
72. H.M.Nico .1%	Lab	24	10	9	1
73. H.M.Nico. .08%	"	24	10	8	2
74. H.M.Nico. .05%	"	24	10	10	0
75. H.M.Nico .025%	"	24	10	10	0
76. H.M.Nico..025% Soap 2-100	"	24	10	10	0
77. H.M.Nico..025% Soap 2-100	"	24	10	10	0
78. H.M.Nico..025% Soap 2-100	"	24	10	10	0

79. H.M.Nico .0125% Soap 2-100	Lab	18	10	10	0
80. H.M.Nico..0125% Soap 2-100	"	18	10	10	0
81. H.M.Nico..0125% Soap 2-100	"	18	10	10	0
82. H.M.Nico..0125% Soap 2-100	"	18	10	10	0
83. H.M.Nico..006% Soap 2-100	"	18	10	3	7

THe results of these experiments show that a solution or extract of tobacco containing .01% nicotine killed the aphis. Somewhat better results were obtained when whale oil soap was added to the extract. In preparing the extract, enough soap was added to equal 4 lbs. per 100 gallons.

While extracts containing only .01% nicotine killed the lice, we think stronger solutions should be used as the spraying may be imperfectly done and since nicotine is highly volatile, larger quantities would undoubtedly last longer and be more effective.

The addition of lime-sulphur to nicotine solutions diminishes its insecticidal value very considerably; in some cases, a solution containing .05% of nicotine and lime-sulphur in the proportion of 1-40 was not sufficient to kill all of the insects in 24 hours. When soap is added to such a solution, a calcium soap precipitates. However, this precipitate is not heavy enough to interfere with spraying. Lime-sulphur, when used alone in the proportion of 1-40, the strength of a summer spray, does not kill apple aphis. Likewise, a soap solution of 8 pounds per 100 gallons does not prove sufficient to kill these lice. The use of lead arsenate in the proportion of 5 pounds per 100 gallons in conjunction with nicotine, lowers the insecticidal value of the nicotine solution for aphis but a solution containing .05% of nicotine and 5 pounds of arsenate of

lead per 100 gallons and lime-sulphur, 1 part to 40, is of sufficient strength to kill the lice. If it is desired to use arsenate of lead for the codling moth and nicotine for the aphis, the two substances may be mixed together as a spray in the proportion of five pounds of arsenate of lead per 100 gallons and nicotine of .05% strength. Also, Bordeaux mixture may be used with a nicotine solution of .05% strength and not impair its insecticidal value. Such a solution does not injure the foliage of the apple tree when applied just as the leaves have opened out.

How much tobacco to use in making a tobacco decoction is a difficult problem that will confront the farmer, unless he knows the percentage of nicotine contained in the stems or leaves. A chemical analysis will give him this information but that is out of the question because he cannot get the analysis made for him without cost. However, he can use enough tobacco to make a more concentratedextract than the experiments show is necessary thereby insuring sufficient nicotine to kill the lice.

Nicotine content of tobacco varies greatly. Some varieites of tobacco may have different amounts of nicotine depending on many factors. The fertility of the soil and the kind of soil both have influence. The method of curing, unless watched carefully, may drive off some of the nicotine. The temperature is often allowed to run too high and the nicotine is lost by volatilization.

How to prepare a Tobacco Extract.

The farmer, if he has a lime sulphur cooker can soak the tobacco in water and then turn on the steam. As soon as the solution reaches the boiling point the steam should be shut off and the barrel or kettle allowed to cool and the content strained to free it of the stems or leaves. If possible he may use some means to press out the refuse. The farmer could use an open kettle to prepare an extract but the yield of nicotine will be less, owing to volatilization and evaporation.

From our experiments, we believe the farmer, for all practical purposes, can prepare an extract with nearly as good results as cooking with steam by simply allowing the tobacco or stems to soak for 24 hours, and strain.

Table V gives the percentage of nicotine and the number of pounds to use to give a 100 gallon extract containing the following percentages: .06%, .05%, .025%, and .01%, assuming that 75% of the nicotine originally present is extracted. By a careful study of this table one can make a fairly close approximation of the proper amount of the tobacco which is to be used. For the sake of economy only the stems and sweepings should be used in making tobacco extracts. If the leaves are of good quality they are far too valuable to be used in making nicotine solutions. However, if a small amount of tobacco can be grown on the farm for this purpose, it would be a very economical thing to do. In purchasing stems or sweepings,

for this purpose, one should get those from tobaccos of high nicotine content, such as the Narrow Leaf Orinocco, Burley, and other dark varieties.

Table V.

Lab. No.	Kind.	Where From.	%Nicotine.	No. Lbs per 100 gallons necessary to make solutions containing different percentages of nico. .06%	.05%	.025%	.01%
1	Stems	Richmond, Va.	.481	145	121	61	24
2	"	Danville, Va.	.609	110	91	45	18
3	Sweepings	Danville, Va.	.884	74	62	61	12
4	N.L.Orinocco	Appomattox, Va.	5.535	12¼	10 1/3	5	2
5	Olive	Powhatan, Va.	3.367	19½	16¼	8	3¼
6	Light	Danville, Va.	2.984	22	18	9	3½
7	Sweepings	Ky.Tob.Prod.Co., Louisville, Ky.	.753	91	85	42½	15
8	Smoker	Chatham, Va.	2.306	28½	23½	11¼	4¾
9	Wrapper	Chatham, Va.	3.05	21½	18	9	3½
10	Cutter	Chatham, Va.	3.466	19	15	7½	3
11	Dark	Appomattox, Va.	2.835	23¼	19¼	9½	4
12	N.L.Orinocco	Bowling Green, Va.	5.529	11¾	10	5	2
13	Med. Smoker	Chatham, Va.	3.766	17½	14½	7¼	3
14	Common "	Chatham, Va.	2.47	26	21½	10¾	4¼

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