THE PREPARATION OF A NICOTINE HYDROCHLORIDE DUST INSECTICIDE AND THE ECONOMIC FEASIBILITY OF GROWING HIGH NICOTINE CONTENT TOBACCO AS A CASH CROP

by

John C. Bowers

A Thesis Submitted for Partial
Fulfillment of the Requirements
for the Degree of

MASTER OF SCIENCE

in

CHEMICAL ENGINEERING

Approved:

____
In Charge of Investigation

____
Head of Major Department

____
Dean of Engineering Division

____
Director of Graduate Studies

Virginia Polytechnic Institute

Blacksburg, Virginia

1947

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I. INTRODUCTION

Nature, it seems, has provided for every crop at least one pest. The competition thus created is a fight the world over between man and pest, and it has aptly been called the greatest war of all times. For a time, it seemed as if man was going to be the loser. But, in order to circumvent the law of nature that decrees that for every crop there shall be two claimants: on the one side, he who planted it and expects to harvest it as a rightful product of his labor; and on the other, the many forms of insects and fungi which live upon this crop, apparently no less anxious than the grower himself to reap the harvest, man has resorted to a scientifically conceived and executed plan. Insecticides in the form of solids, liquids, and gases have been used. Records show that insecticides were used as long ago as 1,000 B.C. However, the first insecticides were more often useless than useful. The essential property of the early insecticides was a disagreeable odor rather than a poisonous nature.

Nicotine solutions and tobacco dust were among the first insecticides used. Nicotine solutions and compounds proved to be very successful and still are dependable and quite safe so far as delicate plant tissue is concerned. Nicotine is the only insecticide of plant origin produced in the United States of which there is an ample source. However, the demand for tobacco, the source of nicotine, for other purposes resulted in high prices being paid for tobacco. This just about eliminated the use of tobacco for producing nicotine except for that tobacco which is fit for no other purpose. Hence, the resultant shortage of nicotine and its compounds, and the directing of research toward providing other and cheaper insecticides.

This investigation deals with the preparation of a nicotine hydrochloride dust from the entire tobacco plant and the economic possibility of the tobacco farmer cultivating tobacco for the purpose of selling his tobacco to manufacturers that produce the insecticide. Some work is being done along this line by the Agricultural Department. In 1943, the Department's tobacco diversion program was successful in adding 1,800,000 pounds of nicotine sulfate solution to the regular supply of nearly 2,250,000 pounds, making a total of about 4,000,000 pounds.

The purpose of this investigation is:

1. Preparation of a nicotine hydrochloride dust insecticide from the entire tobacco plant.

2. To make a study of the costs of cultivating flue cured tobacco and the costs of cultivating tobacco that could be used for the preparation of the dust.

3. To determine the price at which the tobacco farmer could sell high nicotine content tobacco and still make the profit he realizes from the cultivation of flue cured tobacco.

II. LITERATURE REVIEW

The Cost of Producing and Selling Flue Cured Tobacco

The difference between gross and net producer income from tobacco is accounted for by the cost of production and sale. These costs are both monetary and non-monetary, and the latter are usually difficult of exact evualuation(1).

Direct Costs. The monetary costs of production are the actual investments in land and capital, the cost of fertilizers, the cost of production, credit, and the wages paid to hired labor. The monetary costs of sale are transportation and storage costs, (if not supplied from farm facilities), and the warehouse charges themselves. Non-monetary costs comprise losses in land fertility, the farmer's and his family's labor, work hours from farm live stock and equipment, and fuel for curing, (if cut off the farm; if bought, this becomes a monetary cost). From farm to farm, the various costs shift between monetary and non-monetary. Some farmers hire labor and others use family labor; some use horse drawn equipment, some hand operated, and others use tractor drawn machines. Thus it is difficult to differentiate between the categories of cost, or to determine a fair cost per unit of production for all the flue cured farms(1).

Woofter[49] states the cost-relationships of the tobacco farmer rather aptly: a very considerable proportion of the costs are accounted for by the labor (about 80%) either performed by the farmer and his family or hired. Thus, tobacco cultivation is merely a divise for the farmer to use his land and sell his labor rather cheaply and occasionally make a small profit.

In studies which have been made of the costs of raising an acre of tobacco, there have been no consistent practices of inclusion or exclusion of most items. However, all the studies do recogonize the fact quoted above, and endeavor to meet it by assuming all man hours of labor as hired at the prevailing rate. On this basis, Underwood[28] arrived at an average cost of $127.35 for raising and transporting one acre of tobacco (warehouse costs are not included) in Pittsylvania County, Virginia in 1933. Of this amount, $86.24 represented the cost of 438.6 man hours of labor at the rate of $0.197 per hour. If it is assumed that the entire labor bill was provided by unpaid family labor (including the farmer's own), then the cash cost of raising and transporting to market one acre of tobacco was $41.11 (it should be noted that this figure still includes such indirect and imputed costs as fuel, and the interest and user costs for land, machinery, etc. Many of these costs do not necessitate any out-of-pocket expenditures). For the same tobacco the average yield was 642 pounds per acre, and the average warehouse charge was $0.62 per 100 pounds, or

$3.98 per acre. Thus using these figures and assuming all labor furnished by the family, the average cash cost of raising and selling one acre of flue cured in that area was not more than $45.09. If this figure can be considered as representative of the entire Virginia Old Belt, which in 1933 had an average flue cured value per acre (based on acreage and total farm value of the flue cured crop) of $118.00, then the net money income in that area from one acre of tobacco was $73.00. For farms studied (by Underwood) the average tobacco crop value per acre was nearly $93.00.

Similar studies made in other sections of the flue cured area produce a wide range of total costs per acre, depending on the studies. Calculation such as the above, which assume all labor unpaid, furnish the lower estimates and those assuming all labor paid furnish the higher ones. Actual costs probably average between these two limits. However, it is apparent from the figures given above that the tobacco farmer seldom gets less from his crop than his actual cash outlays. Woofter (49) says, "When it is stated that the farmer loses money on his crop it does not generally mean that he sells for less than the cash invested in production, but that he does not earn, for his labor, a wage equal to the usual level of farm wages".

Indirect Costs. It is distinctly possible that one of the most important production costs of tobacco culture is loss of soil fertility, either through erosion or through depletion.

Since organic matter in the soil is not consistent with high quality tobacco, there are few alternate uses of possible soil-building rotations that prove attractive to the flue cured producer. Thus the soil is used rather continuously, without cover crops, and more constant danger of loss of fertility(1).

Conclusions. The farmer who raises flue cured seldom fails to realize more for his crop than his actual cash investment. However, in most years he and his family receive less for their labor than does the average hired agricultural laborer. In addition, the fact that tobacco (a soil depleting crop) cannot be grown in rotation with soil building crops, and the fact that good tobacco fields are usually under almost continuous cultivation, intensify the possibilities of erosion and loss of soil fertility. If the irreplaceable soil is added to the high unpaid labor bill, it is quite possible that tobacco culture is in the long view, a distinctly unprofitable enterprise(1).

Detailed Costs

Costs of Growing Tobacco. The cost of growing tobacco as computed by Underwood(29) included all costs incurred before harvesting the crop, whether the items represented cash outlay or not. It included the cost of performing all operations in producing plants, land preparations, transportation, transplanting, and all the work done between plant setting and the beginning of harvest. Fertilizer, seed, manure, cover crop expense, poison, crop insurance, and land use were included as growing costs.

Cost of Producing Plants. About one-half of the total labor required in producing plants was spent in preparing, sowing, and fertilizing the bed. About two-fifths of the total labor of producing plants was absorbed in weeding and watering the beds. Work animals were used mostly in cleaning the ground of roots and stumps and hauling water. The total cost of doing all the work of producing plants, including horse work and the use of cover cloth and equipment, averaged $12.77 per one hundred square yards of plant bed. This accounted for 84.5% of the total cost of producing plants. About two-thirds of the total cost represented man labor. Fertilizer and manure accounted for 12.6% of the total cost. The average total cost of producing five thousand plants, or about enough to plant an acre of tobacco was $4.24[29].

Table I, page 8, gives a tabulation of the cost incurred in producing tobacco plants[29], and Table II, page 9, gives a summary of the costs of producing tobacco plants[30].

Cost of Plowing. The average cost of plowing (or fallowing) tobacco was $3.64 per acre. The average amount of labor was 8.5 man hours and 14.5 horse hours. The average cost for the use of equipment was $0.21 per acre. Average plowing cost

Table I Labor, power, and equipment costs in producing tobacco plants

Operation	Average for 100 square yards of plant bed					
	Man labor		Horse work		Equipment cost	Total cost
	Hours	Cost	Hours	Cost		
Preparing, sowing, fertilizing	24.8	$5.17	7.5	$0.90	$0.18	$6.25
Fencing and covering	3.4	0.69			1.15*	1.84
Weeding	15.2	3.08	0.1	0.01		3.09
Watering	4.6	1.00	3.7	0.45	0.05	1.50
Top dressing	0.3	0.06				0.06
Miscellaneous	0.1	0.03				0.03
Total	48.4	$10.03	11.3	$1.36	$1.38	$12.77

*Includes annual charge for cover cloth.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p.43, (1939).

Table II Summary of costs of producing tobacco plants

	Average for 100 square yards		Average for 5,000 plants		Percent of total cost
	Amount	Cost	Amount	Cost	
Man labor,hours	48.4	$10.03	13.6	$2.81	66.3
Horse work,hours	11.3	1.36	3.2	0.38	9.0
Cover cloth		1.15		0.32	7.6
Equipment use		0.23		0.06	1.4
Seed,ounces	0.7	0.41	0.2	0.12	2.8
Fertilizer,pounds	159.8	1.72	44.8	0.48	11.3
Manure,pounds	74.8	0.19	21.0	0.06	1.4
Poison		0.03		0.01	0.2
Total		$15.12		$4.24	100.0

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64 p. 44, (1939).

varied with the kind of implement, size of tobacco field, and kind of soil.

In this operation, 63.1% broke their tobacco land with two-horse turning plows, and 27.6% used one-horse turning plows. The average cost per acre for this operation was slightly less for two-horse than for one-horse turning plows. A saving in man labor more than off set the higher horse work and equipment cost incurred in the use of two-horse plows(30).

The average cost of plowing on the farms using tractors was only $2.40 per acre.

Table III, page 11, gives the comparative costs of plowing tobacco land with different implements(31).

<u>The Cost of Cultivating or Rebreaking</u>. This operation was a cultivation of the field in preparation for planting. It was not necessary and was performed by only about half of the farmers covered in this study. The average cost of this operation was $1.78 per acre covered once. The average labor requirement was 5.9 man hours and 6.1 horse hours per acre per operation(31).

Table IV, page 12, gives the comparative cost of cultivating tobacco land before planting with different implements(32).

<u>Cost of Harrowing</u>. The average cost of harrowing tobacco land was $0.71 per acre. More than 75% of the farmers who harrowed their tobacco land used two-horse teams. The average

Table III Comparative costs of fallowing tobacco land with different implements

Kind of plow	Average costs per acre					
	Man labor		Horse work		Equipment	Total
	Hours	Cost	Hours	Cost		
One-horse turning plow	10.2	$2.10	10.2	$1.58	$0.15	$3.38
Two-horse turning plow	7.9	1.62	15.9	1.66	0.21	3.49
Double shovel, colter or single shovel[1]	12.5	3.73	12.5	4.29	0.12	8.14
Colter and two-horse turning plow	10.6	2.31	14.5	2.23	0.31	4.85
One- and two-horse turning plow	10.0	1.81	13.4	1.65	0.23	3.69
Part or all tractor plowed	2.4	0.62	0.4	0.18	1.60[3]	2.40
All farms[2]	8.5	$1.75	14.5	$1.68	$0.21	$3.64

[1]Includes 4 farms using both colter and one-horse turning plow.

[2]Information regarding kind of plow not obtained on three farms, while one farm did not fallow tobacco land but opened the rows after cultivating once.

[3]Includes 2 hours tractor use at $1.29 per acre.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 45. (1939).

Table IV Comparative costs of cultivating tobacco land before planting, by kind of implement[1]

Kind of implement	Average cost per acre per cultivation					
	Man labor		Horse work		Equipment	Total
	Hours	Cost	Hours	Cost		
One-horse cultivator	4.6	$0.79	4.6	$0.45	$0.04	$1.28
Double shovel	5.8	1.17	5.8	0.62	0.09	1.88
One-horse turning plow	8.4	1.59	8.4	0.73	0.09	2.41
All kinds	5.9	$1.12	6.1	$0.59	$0.07	$1.78

[1]Includes only farms using one kind of implement.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 46, (1939).

cost of harrowing an acre once with two-horse harrows was $0.66 as compared to $0.87 for one-horse harrows. The farmer that harrowed with a tractor accomplished the harrowing at a cost of $0.50 per acre(32).

Table V, page 14, gives the comparative cost of harrowing tobacco land with one or two horses(33).

Cost of Disking. About 75% of the farmers who reported this operation used horse drawn disks, mostly with two-horse teams. Two-horse disking was considerable cheaper than that done with one-horse. Tractor disking was more expensive than horse disking because the saving in man and horse hours was not sufficient to offset the cost of operating the tractor and the use of a more expensive disk. The average cost of all disking was $1.02 per acre and required 2.2 man hours and 4.5 horse hours(33).

Table VI, page 15, gives the average costs of disking tobacco land with different implements(33).

Cost of Dragging or Logging. The average cost of dragging was $0.69 per acre covered once. This operation required 1.8 man hours and 3.0 horse hours. The cost of dragging or logging with twe-horse teams was less than dragging with one-horse teams(33).

Table VII, page 16, gives the average cost of dragging or logging tobacco land(34).

Cost of Laying Off Rows. The average cost of laying off tobacco rows was $0.84 per acre. This was a one-horse job,

Table V Comparative costs of harrowing tobacco land with one or two horses

Number of horses	Average cost per acre per harrowing					
	Man labor		Horse work		Equipment	Total
	Hours	Cost	Hours	Cost		
One	2.2	$0.47	2.2	$0.37	$0.03	$0.87
Two	1.6	0.31	3.1	0.32	0.03	0.66
All farms	1.6	$0.34	3.0	$0.34	$0.03	$0.71

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 47, (1939).

Table VI Average costs of disking tobacco land

Size of team	Cost per acre per disking							
	Man labor		Horse work		Tractor use		Disk use	Total
	Hours	Cost	Hours	Cost	Hours	Cost		
One-horse	6.9	$1.08	6.9	$0.66			$0.06	$1.80
Two-horse	2.3	0.48	4.6	0.42			0.03	0.93
Three- or four-horse	2.0	0.38	7.2	0.64			0.05	1.07
All horse-disking	2.4	0.49	5.1	0.46			0.04	0.99
Tractor	0.9	0.18			0.9	$0.76	0.30	1.24
All disking	2.2	$0.45	4.5	$0.41	0.1	$0.09	$0.07	$1.02

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 47. (1939).

Table VII Average costs of dragging or logging tobacco land

Size of team	Cost per acre per dragging					
	Man labor		Horse work		Use of drag	Total
	Hours	Cost	Hours	Cost		
One-horse	2.6	$0.46	2.6	$0.33	$0.03	$0.82
Two-horse	1.5	0.31	3.0	0.32	0.03	0.66
All farms	1.8	$0.34	3.0	$0.32	$0.03	$0.69

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 48, (1939).

requiring 2.4 hours per acre(34).

Cost of Fertilizing and Listing. The most common method of fertilizer application was to open the row with a single shovel, distribute the fertilizer in the bottom of the furrow, either with a drill or by hand, and list the row. Listing was usually accomplished by going twice to the row with a one horse turning plow to prepare a ridge, or bed, in which the plants were set. Most of the fertilizer was bought on a delivered basis at the farm, so that hauling did not represent a direct labor cost on tobacco. However, a certain amount of hauling to the field was included in calculating the cost of fertilizing tobacco. The total cost of hauling and distributing fertilizer, exclusive of the price of fertilizer, averaged $1.21 per acre. This operation required 3.9 man hours and 2.6 horse hours. The labor of listing averaged 4.7 man hours and 4.6 horse hours per acre. The average total cost per acre, including equipment used was $1.60. In some cases a combination fertilizer-drill and lister was used. Including the extra men required for handling the fertilizer, the man labor used with the lister averaged 3.6 hours per acre, which was five hours less than the total for performing the two separate operations of fertilizing and listing. The total cost of using the combination fertilizer-drill and lister was only $1.28 per acre listed, as compared to $2.81 per acre for the combined cost of fertilizing and listing when performed as separate operations(34).

Table VIII, page 19, gives the average cost of fertilizing and listing tobacco land(34).

Cost of Marking Hills. The labor of marking hills varies from merely walking across the field and stepping in the places where the plants were to be set, to drawing a two or three row marker across the rows and working up a small area for each plant with the hoe. The average amount of labor used in making hills was 4.1 man hours and 0.5 horse hours per acre. The total cost averaged $0.90 per acre marked(35).

Cost of Planting. Transplanting required 17.4 hours of man labor per acre. This includes the labor of drawing the plants from the bed. Other expenses incurred in planting of the tobacco were watering expenses and replanting expenses. The total cost of transplanting, watering, and replanting averaged $4.74 per acre(35).

Table IX, page 20, gives a tabulation of the cost incurred in planting tobacco(35).

Growing After Planting. The cost of growing tobacco from transplanting to harvest included all tillage operations, topping, suckering, and worming. The tillage operations included siding down, cultivating, hoeing, chopping, splitting middles, laying by, using sweep, and hilling. The cost of labor, power, and equipment for these operations averaged $22.65 per acre, of which 88% represented man labor. The average labor requirement was slightly more than 100 man hours and 21 horse hours per acre. All hand operations combined accounted for

Table VIII Average costs of fertilizing and listing tobacco land

Operation	Average costs per acre					
	Man labor		Horse work		Equipment	Total
	Hours	Cost	Hours	Cost		
Hauling and distributing fertilizer	3.9	$0.81	2.6	$0.32	$0.08	$1.21
Listing	4.7	0.95	4.6	0.54	0.11	1.60
Total	8.6	$1.76	7.2	$0.86	$0.19	$2.81
Combination drill and lister[1]	3.6	$0.68	4.5	$0.44	$0.16	$1.28

[1] The combination fertilizer drill and lister was definitely reported used on 9 farms. The average acreage of tobacco grown on these farms was 13.5, as compared to 8.1 on the farms that reported fertilizing and listing as separate operations.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 48, (1939).

Table IX Costs of planting tobacco

	Average per acre, all farms					
	Man labor		Horse work		Equipment use	Total cost
	Hours	Cost	Hours	Cost		
Transplanting	17.4	$3.49	0.3	$0.02	$0.01	$3.52
Watering	0.5	0.09	0.3	0.04		0.13
Replanting	5.6	1.09				1.09
Total	23.5	$4.67	0.6	$0.06	$0.01	$4.74

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 49, (1939).

about four-fifths of the total man labor for growing after planting(35).

Table X, page 22, gives the labor, power, and equipment cost of growing tobacco after planting(36).

Cost of Land Use. The average value of the land on which tobacco was grown on these farms in 1933 was $335.28 per acre. The cost of the use of this land averaged $2.26 per acre. The charge for the use of owned land was slightly higher than for rented land because the owned land was more valuable, and interest on the value of the land constituted 7% of the total charge. Taxes and miscellaneous charges each averaged 25 cents per acre(37).

Table XI, page 23, gives cost of land use for tobacco(38).

Cost of Fertilizer. Fertilizers applied on tobacco were charged at their actual cost to the growers and the cost of hauling and application were included with labor and equipment charges. However, most of the fertilizer was bought on a delivered basis at the farm. The average cost of fertilizer used on tobacco fields was $20.34 per ton, or $8.50 per acre fertilized. The total cost of fertilizer, including the value of the fertilizer, was $9.78 per acre fertilized(37).

Table XII, page 24, tabulates these costs(38).

Cost of Manure. Where manure was used, the average of the growers' estimates of the value of the manure used on the tobacco fields, including the cost of hauling and spreading

Table X Labor, power, and equipment costs of growing tobacco after planting

Operation	Average per acre, all farms					
	Man labor		Horse work		Equipment cost	Total cost
	Hours	Cost	Hours	Cost		
Siding down	0.2	$0.05	0.1	$0.01		$0.06
Cultivating	14.9	3.04	15.0	1.71	$0.22	4.97
Hoeing	21.2	4.14			0.02	4.16
Chopping	4.9	0.94			0.01	0.95
Splitting middles	0.6	0.13	0.6	0.07	0.01	0.21
Laying by	3.8	0.76	3.8	0.43	0.07	1.26
Using sweep[1]	1.6	0.33	1.6	0.18	0.02	0.53
Hilling	0.7	0.16				0.16
Topping	8.4	1.67				1.67
Suckering and worming	43.7	8.66				8.66
Spraying and applying poison	0.1	0.02				0.02
Total	100.1	$19.90	21.1	$2.40	$0.35	$22.65

[1]The terms "sweep" and "Buzzard wing" are used interchangeably in this tabulation.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 50. (1939).

Table XI Cost of land use for tobacco

Item	Average per acre		
	Owned[1]	Rented[1]	All farms
Average value	$36.60	$33.56	$35.28
Taxes	$ 0.23	$ 0.27	$ 0.25
Interest	1.83	1.67	1.76
Other costs	0.25	0.26	0.25
Total	$ 2.31	$ 2.20	$ 2.26

[1]Does not include farms on which part of the land was owned and part rented.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p.52, (1939).

Table XII. Costs of fertilizing tobacco fields

Item	Average per acre fertilized	
	Quantity	Cost
Man labor	3.9 hours	$0.81
Horse work	2.6 hours	0.32
Other equipment		0.09[1]
Total labor and equipment		$1.22
Fertilizer	842.1 pounds	8.56
Total cost		$9.78

[1]Includes 91 miles of truck use at 10.1 cents per mile.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 52, (1939).

was $5.21 per ton. The average cost for manure used on tobacco fields on all farms represented 1.3% of the total cost of growing tobacco to harvest time and averaged $0.71 per acre of tobacco grown[38].

<u>Cost of Cover Crops.</u> The average cost of producing cover crops was $5.29 per acre of cover crop. The production of rye (the most common cover crop), wheat, and crimson clover required more man and horse work per acre than cowpeas so that the average cost for rye was somewhat higher than for all cover crops combined. The average cost for rye cover crop was $5.59 per acre[39].

Table XIII, page 26, gives the cost of cover crops for tobacco[39].

<u>Cost of Spray and Dust.</u> In addition to the use of poisons in the form of spray or dust on plant beds, poison was applied on tobacco fields to assist in the control of worms. The labor of applying poison was enumerated separetely from that for suckering and worming. The average cost of this operation was $1.17 per acre of tobacco. Man labor averaged 3.1 hours per acre and constituted 71% of the total cost of the operation[39].

Table XIV, page 27, gives the cost of applying poison on tobacco fields[40].

<u>Cost of Insurance.</u> The average amount of insurance carried on farms on which this item was reported was $76.00 per acre and the average premium paid was $10.28 per $100.00 of insurance. This gave an average premium of $7.32 per acre

Table XIII Costs of cover crops for tobacco

Item	Average per acre of cover crops			
	Rye		All cover crops	
	Quantity	Cost	Quantity	Cost
Seed	1.0 bushel	$1.00		$1.03
Fertilizer			2.2 pounds	$0.02[1]
Man labor	9.5 hours	1.97	9.1 hours	1.83
Horse work	17.1 hours	2.21	16.0 hours	1.99
Equipment use		0.41[2]		0.42[2]
Total		$5.59		$5.29

[1] One farm applied 400 pounds of fertilizer worth $3 on two acres of cowpeas.

[2] Includes 7 hours of tractor use at 74 cents per hour on 2 farms.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 53. (1939).

Table XIV Costs of applying poison on tobacco fields

Item	Average per acre covered	
	Quantity	Cost
Man labor	3.1 hours	$0.83
Equipment use		0.03
Total labor and equipment		$0.86
Materials		0.31
Total cost		$1.17

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 54, (1939).

insured. Insurance on tobacco buildings and equipment was included in the cost of the use of these items[40].

Cost of Interest. Interest on equipment, land, and buildings was included in the cost calculated for these items. Interest charges were also calculated on costs at a rate of 6% per annum from the date each item of cost was incurred to the date the tobacco was marketed. They were calculated on both labor and materials used in production. Interest charges on growing cost averaged $1.57 per acre[40].

Cost of Harvesting. The average labor and equipment cost of harvesting all tobacco on all farms was $15.80 per acre, of which man labor constituted 87.7%. The average labor requirement per acre was 71.5 man hours and 17.1 horse hours. Total harvesting cost, including interest on costs and miscellaneous charges, averaged $16.24 per acre. Harvesting by priming required 50% more man labor per 100 pounds of tobacco than harvesting by cutting. On the basis of the average yield per acre of tobacco produced on these farms, the difference would amount to 28 hours per acre[41].

Table XV, page 29, gives the costs of harvesting tobacco[41].

Cost of Curing. The chief cost of curing tobacco was the labor of tending fires. This operation requires the constant watchfullness of an attendant during the entire period. However, the attendant was often able to sleep for short periods of time while keeping a careful watch on his fires.

Table XV Costs of harvesting tobacco

Item	Average per acre, all tobacco	
	Hours	Cost
Man labor	71.5	$13.86
Horse work	17.1	1.73
Equipment use		0.21
Total labor and equipment		$15.80
Interest on costs		0.23
Miscellaneous		0.21
Total harvesting cost		$16.24

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 55, (1939).

Only the actual labor of attending the curing barn has been included in the cost of curing tobacco. This represents about three-fourths of the total time for which the fires were operated. The average amount of labor required for firing a curing of primed tobacco was 68.7 hours. Generally, cut tobacco required a longer period and slightly more labor than primed tobacco, because of the moisture content of the stem, and the fact that some of the leaves on the cut tobacco were immature and required more heat for proper curing than leaves primed at the proper stage of maturity. The total curing cost averaged $3.95 per one hundred pounds of tobacco, or $35.33 per acre of tobacco[42].

Table XVI, page 31, gives a tabulation of the costs incurred in the curing of tobacco[42].

Marketing. In this study the farm cost of marketing tobacco included all costs involved in handling the crop after curing. All preparation for market as well as the grower's part in the actual selling was classed as farm costs of marketing. This included all work of moving the tobacco in the barn, rehanging, moving to sweat-house or pit, packing down, ordering, stripping, sorting, tying, loading, hauling, and attending to the sale of the crop. Warehouse charges were deducted from the amount received for the tobacco and were not included as farm costs of marketing[43].

Cost of Storing and Ordering. In this study, moving,

Table XVI Costs of curing tobacco

Item	Average per 100 pounds	
	Quantity	Cost
Man labor, hours	11.2	$2.25
Barn use		0.67
Flues		0.10
Sticks		0.10
Other equipment		0.01
Wood, cords	0.3	0.76
Kerosene		0.01
Interest on costs		0.05
Total		$3.95

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 56, (1939).

rehanging, and packing have been combined and called storing, while moving to a sweat-house or pit and ordering have been combined and called ordering. Labor of storing and ordering counted for a little more than 18% of the total farm labor used in marketing. The average cost of storing was $0.983 and the average cost of ordering was $0.288 per one hundred pounds (44).

Table XVII, page 33, gives the cost of storing and ordering tobacco[44].

Cost of Stripping, Sorting, and Tying. Labor of stripping, sorting, and tying in "hands" averaged about 13.5 hours per one hundred pounds and the cost averaged about $2.62 per one hundred pounds[45].

Table XVIII, page 34, gives the cost of stripping, sorting, and tying tobacco[44].

Costs of Loading, Hauling, and Selling. Farm labor used in loading and hauling tobacco and attending the auction averaged 2.53 hours per one hundred pounds sold. The average farm costs of these operations was $0.79 per one hundred pounds. The cost of disposing of cut tobacco was $0.09 per hundred greater than for prime tobacco[45].

Table XIX, page 35, gives the cost of loading, hauling, and selling tobacco[44].

Summary of Costs and Returns. The average net total cost of producing flue-cured tobacco was $126.70 per acre. With yields of saleable tobacco averaging 641.4 pounds per

Table XVII Cost of storing and ordering tobacco

Item	Average costs per 100 pounds	
	Amount	Cost
Storing:		
Man labor, hours	2.28	$0.432
Horse work, hours	0.93	0.093
Equipment use		0.013
Building use		0.240
Interest on tobacco		0.195
Total		$0.973
Ordering:		
Man labor, hours	1.38	$0.269
Horse work, hours	0.15	0.017
Equipment use		0.002
Total		$0.288

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64, p. 58, (1939).

Table XVIII Cost of stripping, sorting, and tying tobacco

Item	Average costs per 100 pounds	
	Amount	Cost
Stripping, sorting, tying:		
Man labor, hours	13.46	$2.563
Stovewood, cords	0.02	0.047
Equipment use		0.008
Total		$2.618

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 58, (1939).

Table XIX Cost of loading, hauling, and selling tobacco

Item	Average costs per 100 pounds	
	Amount	Cost
Loading, hauling, selling:		
Man labor, hours	2.53	$0.499
Horse work, hours	0.52	0.041
Truck use, miles	0.47	0.027
Automobile use, miles	1.99	0.079
Other equipment use		0.005
Hired hauling[1]		0.138
Miscellaneous		0.001
Total		$0.790

[1]The average cost of hired hauling was 32.6 cents per 100 pounds hired hauled.

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 58, (1939).

acre, this was a net cost of $19.75 per one hundred pounds tobacco sold. The average price received for all tobacco sold was $13.67 leaving a net loss of $39.01 per acre or $6.08 per on hundred pounds.

The largest single item in the cost of production was labor, which amounted to about 44 days per acre and constituted slightly more than two-thirds of the total cost. The average value of labor used in producing tobacco was $0.197 per hour. However, the major portion of this labor did not represent a cash cost, since it was the time spent by farm operators, croppers, and unpaid members of their families. The return to pay for labor, above all other costs, averaged $0.108 per hour.

Horse and mule work averaged 85.5 hours per acre, at a cost of $0.11 per hour. More than two-thirds of this was used in growing the tobacco to harvest time. Other power and equipment use combined averaged $3.75 per acre, or 2.9% of the total cost.

About three-eighths of the total labor on tobacco was spent in conducting the operations required for growing the crop, one-sixth was for harvesting, one-sixth for curing, and a little more than one-fourth for marketing. Labor constituted about three-fifths of the growing costs, 85% of the harvesting costs, 57% of curing costs, and about four-fifths of the costs of marketing.

Of the total cost of growing tobacco to harvest time, averaging $55.84 per acre, commercial fertilizer constituted

16.2%. Total fertility costs, including commercial fertilizer, barnyard manure, and costs of producing cover and green manure crops for tobacco averaged $9.96 per acre of tobacco, or 17.8% of the growing cost of harvest time.

Other important items of cost were wood, land and building use, and interest. The use of wood averaged 2 cords per acre, at a cost of $2.58 per cord. Land and building use averaged $8.08 per acre and constituted 6.4% of the total cost of production. Interest charges on costs and on harvested tobacco until date of sale averaged $3.40 per acre, or 2.7% of the total cost.

Growing costs accounted for 43.8%; harvesting costs, 12.8%; curing 19.9%; and marketing, 23.5% of the total cost of production[28].

Table XX, page 38, gives summary of costs and returns in producing tobacco[46].

Table XX Summary of all costs and returns in producing tobacco

Item	Average per acre		Percent of total
	Quantity	Value	
Growing:			
Man labor, hours	169.00	$33.78	60.5
Horse or mule work, hours	58.10	6.68	12.0
Tractor use, hours	0.04	0.03	
Truck use, miles	0.02		
Plant bed cloth		0.36	0.7
Other equipment		0.99	1.8
Seed and plants		0.13	0.2
Fertilizer, pounds	888.60	9.07	16.2
Manure, pounds	272.90	0.71	1.3
Cover crop		0.18	0.3
Spray and dust		0.02	
Insurance		0.06	0.1
Interest		1.57	2.8
Land use		2.26	4.1
Total		$55.84	100.0
Harvesting:			
Man labor, hours	71.5	$13.86	85.4
Horse or mule work, hours	17.1	1.73	10.6
Other equipment		0.21	1.3
Interest		0.23	1.4
Other harvesting costs		0.21	1.3
Total		$16.24	100.0
Curing:			
Man labor, hours	72.10	$14.45	57.1
Other equipment		1.30	5.1
Building use		4.28	16.9
Wood, cords	1.90	4.87	19.2
Interest		0.35	1.4
Other curing costs		0.08	0.3
Total		$25.33	100.0
Marketing:			
Man labor, hours	126.00	$24.13	80.6
Horse or mule work, hours	10.30	0.96	3.2
Truck use, miles	3.00	0.18	0.6
Automobile use, miles	12.80	0.51	1.7
Other equipment		0.17	0.6
Hired hauling, pounds	275.20	0.89	3.0
Storage building use		1.54	5.1
Interest on tobacco		1.25	4.2
Stovewood, cords	0.10	0.30	1.0
Other marketing costs		0.01	
Total		$29.94	100.0
Summary:			
Man labor, hours	438.60	$86.24	67.7
Horse or mule work, hours	85.50	9.37	7.4
Tractor use, hours	0.04	0.03	
Truck use, miles	3.02	0.18	0.1
Automobile use, miles	12.80	0.51	0.4
Use of other equipment		3.03	2.4
Seed and plants		0.13	0.1
Fertilizer, pounds	888.60	9.07	7.1
Manure, pounds	272.90	0.71	0.6
Cover crop		0.18	0.1
Spray and dust		0.02	
Curing wood, cords	1.90	4.87	3.8
Stovewood, cords	0.10	0.30	0.2
Land use		2.26	1.8
Building use		5.82	4.6
Insurance		0.06	0.1
Interest		3.40	2.7
Hired hauling		0.89	0.7
Miscellaneous		0.28	0.2
Total, all costs		$127.35	100.0
Credit for stems, pounds	214.10	0.65	
Net costs		$126.70	
Tobacco sales, pounds	641.40	87.69	
Net loss		$39.01	

Underwood, F. L. Flue Cured Tobacco Farm Management. Va. Agri. Expt. Sta. Bull. 64. p. 67, (1939).

Growing Tobacco as a Source of Nicotine

Introduction

Nicotine has long been used as an insecticide. At present approximately a million pounds of the alkaloid are produced annually in the U.S., the product being marketed chiefly in the form of nicotine sulfate. Up to the present the supply of nicotine has been derived almost entirely from byproducts of tobacco manufactures, particularly stems, leaf scrap, and clippings, and from low grade or damaged leaf poorly suited for the manufacture of tobacco products and therefore commanding very low prices. In view of the fact that in recent years there has been a marked increase in demand for nicotine as an insecticide, it has seemed desirable to study experimentally the possibilities in growing tobacco primarily for extraction of nicotine[(14)].

The tobacco crop of this country as now grown is estimated to contain an average of 2.5 to 3.0 per cent nicotine (in the leaf), and the average yield of leaf is about eight hundred pounds per acre. Because of the woody character of the stalks, their content of nicotine is too low to justify their utilization for extraction purposes, but this does not exclude their use in a dust made from grinding the tobacco. The upper range in nicotine content of the leaf, which is attained only occassionally, is from 6 to 8 per cent[14].

For the ten year period of 1929-38 the estimated average acre values of the tobacco crop was $135.00 or about $0.17 per pound. The corresponding output of nicotine per acre, even for those types of tobacco having the highest content, would be only 30 to 40 pounds, and on the basis of a dollar per pound farm value for the nicotine, which is materially higher than prevailing prices applying to nicotine in tobacco byproducts, the gross return would be $30.00 to $40.00 per acre. Actually the farm value of such tobacco when diverted to nicotine extraction has averaged $0.02 to 0.025 per pound, or not more than about $20.00 per acre. Obviously the per acre production of nicotine must be greatly increased or the cost of growing the crop must be greatly lowered to make it possible for the grower to receive a reasonable return on the crop which is to be used solely for insecticidal purposes. The necessary increase in nicotine production per acre can be accomplished only by increasing the tonnage of tobacco or the percentage content of the alkaloid or both[14].

In addition to ordinary tobacco, Nicotiana tabacum L., the only species of Nicotiana showing any promise of meeting these requirements was Nicotiana rustica L. Preliminary tests with this species were begun more than 25 years ago, but it has been possible only in recent years to undertake sufficiently comprehensive experiments to yield definite results, especially as to the most favorable conditions of soil and climate for obtaining very high nicotine production. During this interval, reported results of experiments with N. rustica in western New York State, indicated a yield of one hundred to one hundred fifty pounds of nicotine per acre(14).

Present commercial tobacco culture is sharply localized. However, the tobacco plant can be readily grown under a very wide range of soil and climatic conditions if the quality of the product is ignored, so that culture for insecticidal purposes need not be restricted to present tobacco producing areas. On the other hand, environment is an important factor affecting the quantity of nicotine formed in the plant(14).

Available information shows that three major factors or groups of factors influence the tonnage of dry matter produced per acre and the nicotine content of tobacco(14):

1. The species, variety, and strain used;
2. the environment in which the plant is grown, primarily the conditions of soil and climate;
3. the cultural, curing, and handling methods employed as e.g., time of transplanting and harvesting, spacing of plants, topping, and suckering operations.

Relative Efficiencies of Species, Varieties, and Strains for Production of Nicotine

There are many species of Nicotiana, but only two have the growth characteristics and content of nicotine that would justify consideration for present purposes, namely, N. tabacum, which is the ordinary tobacco of commerce, and N. rustica, also utilized for production of smoking and chewing tobaccos in certain foreign countries(15).

Although from the earlier observations N. rustica was known to contain as a rule a higher percentage of nicotine, because of its relatively small size, as compared with most varieties of N. tabacum, there remained a question whether it would consistently give higher yields of nicotine per acre. Systematic tests were therefore conducted to determine which would produce the greatest amount of nicotine when grown under the same conditions at different locations. In table XXI, page 43, it is evident that N. rustica has usually produced a much higher yield of nicotine per acre than ordinary tobacco. For reasons that were not definitely determined, the growth of N. rustica at Lakin, West Virginia, was unsatisfactory, except in 1938, resulting in a low yield of nicotine. At the other locations the nicotine produced by N. rustica, in pounds per acre, was almost double that obtained from ordinary tobacco. However, where temperature, soil conditions and pests appear to

Table XXI Summary of yields of tobacco, percentage content of nicotine in the tobacco, and production of nicotine per acre in plantings of N. rustica and ordinary tobacco at Davis, Calif., Madison, Wis., Lakin, W. Va., and Arlington Farm, Va., during the period 1934-38

Location	Year	Yield per acre (20-percent-moisture basis)		Nicotine content (20-percent-moisture basis)		Production of nicotine per acre	
		N. rustica	Ordinary tobacco	N. rustica	Ordinary tobacco	N. rustica	Ordinary tobacco
		Pounds	Pounds	Percent	Percent	Pounds	Pounds
Davis, Calif.	1934	2,144	2,945	6.97	2.45	149.5	72.2
	1935	3,488	2,500	5.68	3.68	198.2	92.0
	1936	3,010	2,859	5.55	3.72	167.0	106.4
Average		2,881	2,768	6.07	3.28	171.6	90.2
Madison, Wis.	1934	3,098	1,396	3.37	3.13	104.5	43.7
	1935	3,513	1,156	4.30	2.62	151.0	30.3
	1936	1,625	1,244	3.02	2.10	49.1	26.1
	1937	2,891	1,168	5.37	4.51	160.0	52.7
	1938	3,629	1,692	2.80	2.69	101.6	45.5
Average		2,969	1,331	3.77	3.01	113.2	39.7
Lakin, W. Va.	1934	1,111	2,698	3.37	4.02	36.1	108.5
	1935	1,088	1,220	3.46	2.88	37.7	35.1
	1936	1,379	1,781	3.63	3.93	50.0	70.0
	1937	1,358	3,010	4.37	3.13	59.4	94.2
	1938	4,150	2,263	3.42	2.69	142.1	60.9
Average		1,817	2,194	3.65	3.33	65.1	73.7
Arlington Farm, Va.	1934	2,317	2,787	7.03	3.19	162.9	89.2
	1935	3,472	2,671	5.68	4.59	197.1	122.6
	1936	5,365	2,664	4.75	4.47	254.7	119.2
Average		3,718	2,707	5.82	4.08	204.9	110.3

McMurtrey, J. E., Bacon, C. W., and Ready, D. Growing Tobacco as a Source of Nicotine. U.S.D.A. Tech. Bull. 820. p. 10-20. (1942).

interfere with the growth of N. rustica the tobacco does not always double the quantity of nicotine. This was usually the result of a higher nicotine content, although in some instances an increased tonnage of dry matter is the chief factor. It is surprising that N. rustica, which is a much smaller plant than N. tabacum, was able to produce in many instances a larger amount of dry matter than ordinary tobacco. Some of the difference is due to the inclusion of the stalk in the N. rustica yields, whereas weights shown for ordinary tobacco represent only the leaf, but the greater amount was due to the increased thickness and weight per unit area of the N. rustica leaf[15].

In addition to its practical importance, the possibility of further increasing nicotine yields from N. rustica is a matter of some theoretical interest. The principal avenue of approach appears to be in greater yields per acre of dry material. This might be accomplished by producing strains of N. rustica with larger leaves, by increasing the thickness or density of the leaves, or by obtaining combinations of these with an increased number of leaves[15].

Effect of Cultural Methods on Nicotine Yields

The use of suitable cultural methods often constitute the principal means the grower has for better adapting the tobacco plant to some particular use. This is particularly true where the production of nicotine is of chief concern. Some cultural methods or procedures that may be modified are: Topping and suckering of the plants, spacing in the fields, varying the transplanting and harvest dates, use of irrigation, and applying manures and fertilizers[16].

Conditions and Practices for Growing and Handling High-Nicotine Tobaccos

In order to produce a crop that will yield large quantities of nicotine per acre it is important to give close attention to certain details. As the *N. rustica* species produces much higher yields of nicotine than ordinary tobacco under suitable conditions, the requirements, for growing this species to best advantage, which differ somewhat from those of ordinary tobacco, should be the principal consideration. *N. rustica* will not produce maximum quantities of nicotine if it is allowed to become stunted even for comparatively short periods during the seedling or later growth stages. It is therefore important to transplant promptly from the seedbed as soon as the plants attain sufficient size. Transplanting to the field as soon as danger of frost is past is also recommended, as this species makes the best growth during cool moist periods, such as are commonly prevalent early in the season[17].

Use of a vigorously growing strain of N. rustica that does not mature too rapidly is another important point in growing this plant for profitable production of nicotine. Although the planting distance in the field required for best results may vary somewhat, depending upon conditions, as a rule a distance of 15 to 18 inches in rows 30 to 36 inches apart will give satisfactory results[17].

Topping and suckering are operations of the utmost importance if maximum yields of nicotine are to be obtained. Suckering should begin even prior to topping in order to encourage leaf development. Hard and fast rules cannot be given in regard to height of topping. There appears to be no consistent advantage for low topping over high topping but it is evident that topping of some kind is absolutely necessary if good yields of nicotine are to be obtained. The suckers of lateral branches should be removed after topping at regular intervals of 7 to 10 days for the first two or three operations, after which the intervals may be somewhat longer[17].

Possibly the most important consideration relative to building up a high content of nicotine in N. rustica is that the plants should be allowed to remain in the field for as long a time as possible- up to 60 days after topping- provided there are no serious losses of the lower leaves by drying or disease[17].

To avoid important losses of nicotine the curing or drying process should be as rapid as possible and should take place under conditions that will prevent leaching of the dried or partially dried material by rain or water from other sources. Once the crop is dry it will keep for a considerable period without much loss of nicotine if the moisture content does not again become high as a result of high atmospheric humidity or other conditions. The crop possibly could be baled in the same manner as hay, but the moisture contained in the leaf at the time of baling or packing must be low to avoid fermentation. Important losses of nicotine may be expected if there is any considerable fermentation at any stage of curing or drying[17].

The Cost of Growing N. Rustica as a Source of Nicotine

In the small plantings used in the present studies concerning the quantity of nicotine produced by N. rustica it has not been possible to obtain accurate data on the labor requirements for growing the crop. As the number of plants grown per acre is essentially the same as in the case of the burley type of tobacco, the labor requirement surveys that have been made for the latter should be more or less applicable to N. rustica with respect to most of the important cultural operations. One group of operations, however, would not be the same, namely, topping, worming, suckering and spraying or dusting. This complex item would correspond more nearly to the requirements for dark-fired tobacco except that, since there are approximately twice the number of plants per acre, this figure would need to be doubled. Figures for the several major operations, derived in this way from surveys of labor requirements that have been made in the burley area and the dark-fired area of Kentucky, are shown in table XXII, page 49[18][19]. It is evident that inexperienced labor ordinarily could not accomplish the necessary operations in the time indicated. These figures do not include many other items of cost in producing the crop, such as rent of the land, drying sheds, and the cost of marketing; and these figures may be expected, also, to vary considerably in different localities[18].

Table XXII Approximate hours of man labor and horse work required for producing an acre of <u>N. rustica</u> tobacco and their distribution between the principal operations of culture, on the basis of requirements for growing burley and dark-fired tobacco in Kentucky

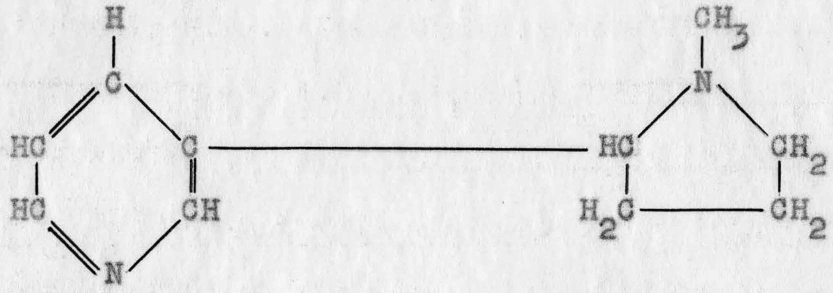
Operation	Average man labor	Average horse work
	Hours	Hours
Plant bed	17	5
Field preparation	19	38
Transplanting	31	10
Cultivating with plow	13	15
Cultivating with hoe	24	
Topping, worming, suckering, and spraying or dusting	108	
Cutting and housing	58	22
Curing	1	
Total	271	90

McMurtrey, J. E., Bacon, C. W., and Ready, D. Growing Tobacco as a Source of Nicotine. U.S.D.A. Tech. Bull. 820. p. 36-37, (1942).

Nicotine Insecticides

Need For Effective Insecticides. The seriousness of insect pests was realized quite some time ago by the country at large. This was due largely to the tremendous devestation of such pests as the Japanese beetle, which made its appearance in New Jersey in 1916. The insecticide manufacturers made considerable progress in the commercial production of high quality insecticides, and growers all over the country rapidly replaced their crude homemade material, which consisted in most cases of some form of nicotine spray or dust, with commercial products, which are chemically controlled, uniform products[10]. Lead arsenate was used by all fruit growers as an insecticide until the year 1926. However, people objected to the arsenical residue left on the fruits after it had dried[47]. The following season many arsenical insecticides were studied anew, and many new insecticides were developed[6]. This caused the rebirth of the use of nicotine sulfate.

The Structure and Properties of Nicotine. Nicotine, the alkaloid of tobacco, is one of the simplest of the alkaloids. It is a tertiary secondary base containing a pyridine ring and hydrogenated pyrrote ring with a methyl group attached to the nitrogen atom[7]. The structural formula for beta-nicotine is given as follows:



Pure nicotine is an oily liquid and one of the most deadly poisons known. Chemically, it is a weak base, uniting with acids to form salts. It is therefore possible to obtain a great many different compounds of nicotine, and it is reasonable to suppose that some of these may be more toxic to insects than others. In experiments, however, nothing has been found to be decidedly superior to nicotine itself. Nicotine has been combined in the theoretically proper proportions with the following acids: acetic, boric, carbolic, cresylic, formic, hydrochloric, hydrocyanic, hydrofluoric, oleic, salicylic, stearic, and tartaric. Of these compounds none has shown positive superiority over nicotine itself[22].

<u>Determination of the Nicotine in the Tobacco</u>. The determination consists essentially of steam-distilling the nicotine into an acid solution and then precipitating the nicotine by adding silicotungstic acid. The following method may be used for determining the nicotine content of either tobacco leaves or of nicotine insecticides[1][3].

For the determination, use a sample that contains from 5 to 10 mg. of the nicotine. Weigh this, then cover the sample with 2 or 3 ml. of water. Now add two drops of phenolphthalein. Next, add in excess a 40% solution of sodium hydroxide. Steam is passed into the flask and is kept at a pressure of 1.5 to 2 feet of water.

The beaker used to receive the distillate must contain 3 ml. of hydrochloric acid (1-4) and 5 ml. of water. The distillation is continued for 30 minutes and 100 ml. of the distillate is collected.

To precipitate the nicotine, 1 ml. of silicotungstic acid (12% solution) is used for each 10 mg. of nicotine or less. After the precipitation, the covered samples are treated with heat over a steam bath for 15 minutes and then cooled slowly to room temperature and maintained at 0°C. overnight. The precipitate is filtered through C. S. & S. No. 589, white ribbon filter paper and washed with 100 to 200 ml. of hydrochloric acid (1-2000).

Transfer the paper and precipitate to a weighed crucible and heat this for seven minutes at a temperature of 960°F. or until all the carbon is destroyed. Then the crucible is heated over a Teclu or Meeker burner for not more than 10 minutes. The weight of the residue multiplied by 0.114 will give the weight of the nicotine present in the sample.

<u>Nicotine Sulfate as an Insecticide</u>. Nicotine sulfate is the most extensively used of all the nicotine compounds. It is used by florists and greenhouse men for spraying and fumigation. In animal husbandry, it is used extensively for dipping

cattle for scab-mite, lice, and sheep-tick. In the poultry field, nicotine is used for internal parasite control and for fighting body lice and mites. In the truck gardening field, application as a spray or in dust form is quite wide for the control of many insects. It is used extensively in connection with commercial fruit growing[#48].

Similarity of the Sulfate and the Chloride. The hydrogen chloride and the hydrogen sulfate radical will both attach themselves onto the nicotine formula in the same manner and position. The two compounds being similar in properties is the reason that the chloride will be equal or even better as an insecticide than the sulfate[12]. Crawley[8] proved the latter to be true.

Advantages of a Dust Insecticide Over a Spray. Insecticides are usually applied either as a dust or as a spray. An insecticide dust has several advantages over a liquid spray. It can be applied much more easily and quickly than a spray, and a dust hopper can be refilled in much less time than a spray tank. The dust particles reach more insects than the spray, particularly on the under side or in curled leaves. Power dusters cost less than power sprayers, while hand dusters are easier to carry and to operate than hand sprayers. Usually much less than 100 pounds of dust is required to cover an acre, while to spray the same area requires at least 100 gallons, weighing at least 800 pounds; on this account dusters can be operated on wet ground and hill sides inaccessible to sprayers. For spraying with nicotine sulfate, a soap spreader is required, and the whale oil or fish oil commonly used must be heated to dissolve it, which causes delay and is a disagreeable task avoided when the dust is used[5].

Properties of an Insecticidal Dust. In order for insecticidal dust to be effective, it must be finely powdered. At least 50% to 75% of the dust must pass a 300 mesh sieve. It must possess adhesive qualities. In the case of nicotine insecticide, the dust must contain between 0.05 and 0.06% nicotine. The nicotine is always present in the form of a salt[8].

Disadvantages of Nicotine Dusts. Nicotine dusts so far used consist of a mixture of nicotine sulfate and some dust as powdered fullers earth, kaolin, hydrated lime, calcium carbonate, gypsum, sulfur, or other absorbent material. These dusts have all proven more or less unstable and must be used soon after they are made. McDonnell[13] found that the factors influencing the loss of nicotine from the dusts were principally the physical and chemical nature of the absorbent used and the container in which the dusts were stored. Obviously, then, the consumer may unknowingly use a worthless product.

How the Dust Acts. The nicotine dust, in which the nicotine is present in the form of a salt, can be made much more powerful by adding hydrated lime. When the dust becomes wet with dew, the lime and nicotine sulfate or hydrochloride react to form the chloride or sulfate of lime and free nicotine. This free nicotine is much more effective in killing insects than is nicotine in the salt form[8].

The main object to be kept in mind is to produce the most powerful dust possible with the minimum amount of nicotine. The toxic strength of the dust is influenced both by the form and amount of nicotine used and the nature of the filler. Two alternatives are presented in utilizing a given amount of nicotine[23].

1. To make the dust of such a nature that the nicotine will be as volatile as possible and the maximum amount be given off in the minimum of time. This means, of course, that the dust will lose its efficiency very soon after application.
2. To make the nicotine less volatile, so that the dust will act more slowly but for a longer time.

Chemical Nature of the Filler

Effect on Nicotine. When nicotine sulfate is mixed with koalin or any other inert material the nicotine remains in the form of nicotine sulfate. When hydrated or quick lime is used, however, a reaction takes place forming sulfate of lime and free nicotine. The latter is more volatile than nicotine sulfate and for this reason dust made with quick lime or hydrated lime is more powerful for the same amount of any dust made with any inert filler[24].

Drying Effect of the Filler. It is possible to use as the filler some materials, such as quick lime, which combine chemically with water as well as simply absorbing it. This is a great advantage in making nicotine dust[24].

Irritating or Burning Effect of the Filler. Nothing should be used as the filler which might possibly cause injury to animal or vegetable life[24].

Physical Nature of the Filler

Fineness. The more finely divided the filler the better dust it will make. It will blow and stick better and thus the nicotine will be brought more closely into contact with the insects[25].

Weight. At first thought a light, bulky material would seem the best for dusting, but this is only partially true. To some extent at least a fairly heavy material is desirable because it can be blown into thick vegetation with more force than a very light material[25].

Dryness. It is very desirable that the filler be as dry as possible before adding the nicotine[25].

Absorptiveness. Experience has indicated that nicotine is more freely given off from materials which do not absorb it readily and that fillers of the latter class therefore produce a quicker acting dust, which seems to be best[25].

Materials Which Have Been Used as Fillers

Kaoline. It is a white, soft, extremely fine substance, of rather light weight, quite absorptive of moisture, and drying into soft, chalky, easily pulverized lumps. It is free from chemical effects, and forms in many respects an ideal filler(26).

Hydrated Lime. Its absorptive, blowing, and sticking qualities are excellent. It reacts with nicotine sulfate, forming free nicotine. The chief objection to hydrated lime is the irritating effect which it has on the skin and eyes of the operator(26).

Quick Lime. Quick lime takes up a large amount of moisture in slaking or hydrating. This is a great advantage in making nicotine dust. The disadvantage of quick lime is the heat which is generated in the reaction, causing the loss of some of the nicotine(26).

Lime Carbonate. This is available in various forms such as air-slaked lime, refuse sugar beet lime, etc. When dried and finely pulverized such materials make a very satisfactory filler. They have no effect upon nicotine sulphate(26).

Gypsum-Plaster. All forms of gypsum and plaster are heavier and not so dusty as kaolin. They are also apt to be coarser unless reground. The various building plasters, from which a portion of the water has been removed by heating, act as driers and do not produce heat. They have the disadvantage of forming hard lumps in drying. None of the materials mentioned react with nicotine sulfate[26].

Diatomaceous Earth. This is extremely light and absorptive material, but it does not form a good dust. It tends to cling together in flakes and particles. Its great absorptiveness seems to be a disadvantage rather than an advantage, since the nicotine is not given up freely and quickly, as is most desirable[26].

Talc. When thoroughly pulverized it makes a very good dust and sticks well. It does not react with nicotine sulfate [26].

Sulfur. Sulfur is frequently added to nicotine dust on account of its value as an insecticide or fungicide, rather than simply as a mechanical carrier. In the latter capacity it is heavy nonabsorptive and rather expensive. However, when mixed with sulfur a given amount of nicotine is more effective against insects than with any of the other fillers mentioned. This may be due to the fact that sulfur, being a poor absorbent, gives off the nicotine more rapidly than do some of the other materials. Only the finest grade of sulfur should be used[26].

Tobacco Dust. When well ground this is a very absorptive material and makes a good dust. Tobacco dusts act more slowly than nicotine dusts containing the same amount of nicotine[26].

Methods of Preparation. Greever[9] prepared nicotine hydrochloride using hydrochloric acid."The leaves were ground into particles the size of salt crystals. This gives the acid a better chance to react with the tobacco, thus forming nicotine hydrochloride. After enough water had been added to dilute the acid to 36.0 per cent, a 1000 liter round bottom flask was filled about half way. Plenty of room was left for agitation of the mixture. This mixture was allowed to set for a week or ten days with occassional shaking to aid the reaction. At the end of this time a complete reaction had taken place in the flask.

The mixture was filtered and the residual tobacco powder was left in the filter paper and discarded. The liquid was a concentrated solution of nicotine hydrogen chloride. This liquid was placed in a large beaker and steam distilled until an almost pure solution of the nicotine hydrochloride had resulted."

Crawley[8] prepared nicotine hydrochloride using HCl gas. The following procedure was used:

Gassing of the Tobacco."The tobacco samples to be gassed were placed inside the gas chamber on a tray made of one-fourth inch mesh wire screen. After fastening the top securely on the chamber, the gas generator was started and the hydrogen chloride gas was passed into the chamber. An effort was at first made to maintain a gas pressure of about two inches of water in the chamber, but this was later found to be unnecessary. All that was necessary was to maintain a slight pressure to be certain that the chamber was full of the gas throughout the run. As suggested by Land[11], 100% gas was used in treating the tobacco. The gas was taken directly from the generator without passing it through a metering device.

Preparation of the Dust. After the tobacco was treated with hydrogen chloride gas to form nicotine hydrochloride, the material was ground in an ordinary meat chopper. The resulting tobacco was placed in a ball mill and was pulverized until the dust was fine enough for at least 50% to pass a 200 mesh sieve. The dust was then ready for application as an insecticide.

Preparation of Samples for Analysis. In order to determine the per cent nicotine activated by the gassing, it was necessary to first separate the activated nicotine, or nicotine hydrochloride, from the pure nicotine. This separation was based on the difference in solubilities of the two substances. The samples to be analyzed were first ground in a mortar and were then placed in a large filtering funnel fitted with ordinary filter paper. The tobacco was then washed with water at a temperature between 90 and 100°F., until no precipitation was obtained when a portion of the filtrate was treated with silicotungstic acid. The nicotine hydrochloride was washed out of the sample, leaving the nicotine. The analysis for the unreacted nicotine was the standard A. O. A. C. silotungstic acid method of analysis, as already given in the review of literature."

III. EXPERIMENTAL

Purpose of the Study

This study was undertaken for the following reasons:

1. Nicotine and its compounds have long been used as insecticides, and are in great demand at the present.
2. Tobacco, the source of nicotine, is such a valuable commodity that only waste has been used as a source of nicotine for years.
3. N. rustica tabacum, a species of tobacco, has a high nicotine content, and has been grown under climate and environment suitable for other types of tobacco.

The purpose of the investigation is as follows:

1. Preparation of a nicotine hydrochloride dust insecticide from the entire tobacco plant.
2. To make a study of the costs of cultivating flue cured tobacco and the costs of cultivating tobacco that could be used for the preparation of the dust.
3. To determine the price at which the tobacco farmer could sell high nicotine content tobacco and still make the profit he realizes from the cultivation of the flue cured tobacco.

Plan of Investigation

This investigation will be conducted in the following steps:

1. Review of literature
    a. Costs of growing flue cured tobacco.
    b. Costs of growing high nicotine content tobacco.
    c. Preparation of nicotine dust insecticides.
2. Construction of equipment necessary
    a. Equipment for analysis of tobacco to determine percentage of nicotine present.
    b. Equipment for the treatment of the tobacco with HCl gas to convert the nicotine to nicotine hydrochloride.
3. Collection of operating data
    a. Percentage of nicotine originally present in tobacco.
    b. Percentage of nicotine present in tobacco after treatment with HCl gas for specific intervals of time before grinding.
    c. Percentage of nicotine present in tobacco after treatment with HCl gas for specific intervals of time after grinding.
    d. Size of particles after grinding in pebble mill using 0.5" diameter pebbles for specific intervals

of time before treatment with HCl gas.

e. Size of particles after grinding in pebble mill using 1" diameter pebbles for specific intervals of time before treatment with HCl gas.

f. Size of particles after grinding in rod mill using miscellaneous rods for specific intervals of time before treatment with HCl gas.

g. Size of particles after grinding in Lancaster Countercurrent Batch Mixer for specific intervals of time before treatment with HCl gas.

h. Size of particles after grinding in pebble mill using 0.5" diameter pebbles for specific intervals of time before treatment with HCl gas.

i. Size of particles after grinding in pebble mill using 1" diameter pebbles for specific intervals of time after treatment with HCl gas.

j. Size of particles after grinding in rod mill using miscellaneous rods for specific intervals of time after treatment with HCl gas.

k. Size of particles after grinding in Lancaster Countercurrent Batch Mixer for specific intervals of time after treatment with HCl gas.

4. Calculation of results

a. Costs of growing one acre of flue cured tobacco wherever it is necessary.

b. Costs of growing one acre of high nicotine content tobacco.

c. The price per pound at which a farmer would have to sell the high nicotine content tobacco in order to realize the profit he makes from the cultivation of flue cured tobacco.

d. Percentage of nicotine converted to nicotine hydrochloride after treatment with HCl gas for specific time intervals.

e. Percentage of particles of a given size after grinding for specific time intervals.

f. The optimum time of treatment with HCl gas and grinding or combination of the two.

Materials

Sodium Chloride, crystals. Used in gas generator to produce HCl gas. Mulkey Salt Co., Detroit, Michigan.

Acid Sulfuric, A. C. S. Std., sp. gr. 1.84, assay 95-96%, lot No. E-207018. Used in gas generator to produce HCl gas. J. T. Baker Co., Phillipsburg, New Jersey.

Acid Hydrochloric, A. C. S. Std., sp. gr. 1.19, assay 36.1%, lot No. 32546C. Used to prepare solutions of 1 to 4 and 1 to 2000 used in determination of nicotine in tobacco. J. T. Baker Co., Phillipsburg, New Jersey.

<u>Acid Silicotungstic</u>, crystals, C. P. Used to prepare 12% aqueous solution as analytical reagent in determination of nicotine in tobacco. Fisher Scientific Co., Pittsburgh, Pennsylvania.

<u>Sodium Hydroxide</u>, flake, 76% Na_2O, code 2256, lot No. C108J. Used to prepare 40% aqueous solution used in determination of nicotine in tobacco. General Chemical Co., New York, New York.

<u>Tobacco</u>, scrap, flue-cured, 1946 crop. Used to prepare a nicotine hydrochloride dust insecticide. Mr. W. L. Bowers, Rt. 3, Chase City, Virginia.

<u>Paint</u>, Tygon, code No. TP-61 White, Control No. 7154. Used to paint gas chamber, top and shelves to prevent corrosion by HCl gas. U. S. Stoneware, Akron, Ohio.

<u>Lime</u>, hydrate. Used in analytical determination of nicotine in tobacco after treatment with HCl gas. Obtained from New River Lumber Co., Blacksburg, Virginia.

<u>Gas</u>, Pyrofax, Used as fuel for Bunsen and Tirrill burners.

<u>Water</u>, distilled. Used to prepare all aqueous solutions used in the determination of nicotine in tobacco.

<u>Phenolphthalein</u>. Used as indicator in determination of nicotine in tobacco.

<u>Ammonium Hydroxide</u>, sp. gr. 0.60, 28% NH_3, code 1243, lot No. 6133. Used to furnish NH_3. General Chemical Co., New York, New York.

Apparatus

Flask, Pyrex round bottom, 1000 ml., (one). Used as steam generator for analytical determination of nicotine in tobacco.

Stopper, Rubber, three-hole, No. 8, (one). Used as top for steam generator.

Tubing, Soft glass, 6 mm. inside diameter, 3' long, (one). Used as pressure gage on steam generator.

Flask, Pyrex round bottom, 500 ml., (one). Used as distillation flask for analytical determination of nicotine in tobacco.

Stopper, Rubber, two-hole, No. 6, (one). Used as top for distillation flask.

Trap, Kjehdahl, (one). Used as liquid trap between distillation flask and condenser in analytical equipment for determination of nicotine in tobacco.

Condenser, Glass, water, (one). Used to condense distillate in the analytical determination of nicotine in tobacco.

Stopper, Rubber, one-hole, No. 4, (one). Used as top for condenser.

Beakers, Pyrex, 150 ml., (eight). Used as distillate receivers in the analytical determination of nicotine in tobacco.

Tubing, Soft glass, 3 mm. inside diameter, (various lengths). Used as connections between parts of analytical apparatus.

Tubing, Rubber, (various lengths). Used as connections between parts of analytical apparatus. (See Figure 1, page 69).

Burner, Bunsen, (one). Used to heat water in steam generator.

Burner, Tirrill, (one). Used to reduce volume of liquid in distillation flask.

Tripod, Iron, (one). Used to hold crucibles while heating residue over burner.

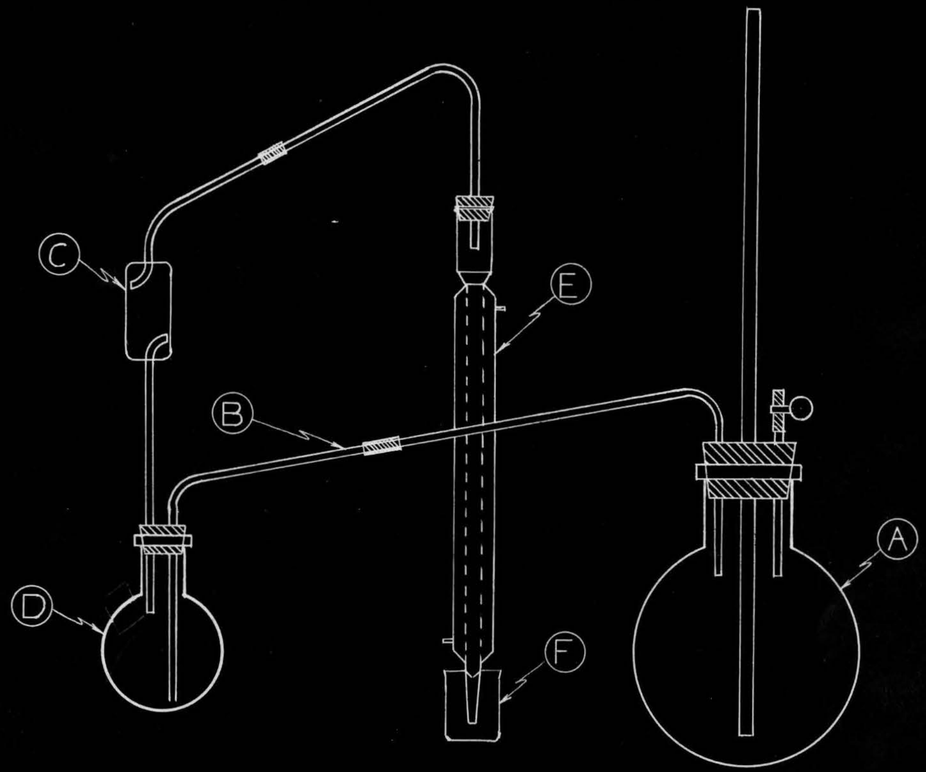
Gauze, Wire, asbestos center, (one). Used to hold crucibles while heating residue over burners.

Beaker, Pyrex, 250 ml. (one). Used as steam bath in analytical determination of nicotine in tobacco.

Rack, Funnel, wooden, (one). Used to hold funnels for filtrations in analytical determination of nicotine in tobacco.

Funnels, Pyrex, long stem, (two). Used to filter precipitate in analytical determination of nicotine in tobacco.

Paper, Filter, Whatman, No. 40, 11.0 cm., ashless. Used to filter precipitate in analytical determination of nicotine in tobacco. Manufactured by W. and R. Bolston, Ltd., England.



LEGEND

A Steam Generator
B Delivery Tube
C Safety Trap
D Distillation Flask
E Water Condenser
F Distillate Receiver

All stoppers and connections — rubber
Supports and Burners not shown

VIRGINIA POLYTECHNIC INSTITUTE
DEPARTMENT OF CHEMICAL ENGINEERING
Blacksburg, Virginia

APPARATUS FOR ANALYTICAL DETERMINATION OF NICOTINE

Drawn by: W.B.	Date: Sept. 1, 1947
Checked by: Frank C. Vilbrandt 9/8/47	Figure No. 1
Approved by: Frank C. Vilbrandt 9/8/47	Scale: None

Dishes, Evaporating, porcelain, Coors, 3" diameter, (four). Used to hold samples during heating in the determination of the moisture content of tobacco.

Crucibles, Porcelain, 20 ml. capacity, Coors, (ten). Used to fire smaples and to hold samples while reaction with lime was carried out.

Tongs, Monel metal, (one pair). Used to handle crucibles.

Dessicators, Glass, (two). Used to hold crucibles and residue while cooling.

Bottles, Glass, weighing, (four). Used to weigh samples of tobacco after drying in the determination of the moisture content in tobacco.

Rings and Clamps, Steel, (six). Used as supports for steam generator, distillation flask, and condenser.

Watchglasses, 2" diameter, (four). Used to weigh out samples of tobacco and as covers for distillate receivers during heating over steam baths.

Balance, Analytical, Becker Chainomatic, (one). Used to weigh all samples in analytical work. Manufactured by Seederer-Kohlbusch Inc., Jersey City, New Jersey.

Weights, Analytical (one set). Used in weighing all samples of tobacco in analytical work. Manufactured by Will Corporation, Rochester, New York.

<u>Oven</u>, Drying, Electric, (one), Serial No. 100-2761, Cat. No. 1250, Temp. range 35 to 180°C. Volts 110, Cycles 60, Watts 100, Amp. 5.5, Phases 1, Type A. Used to dry samples of tobacco in moisture determination. Manufactured by Precision Scientific Co., Chicago, Illinois.

<u>Furnace</u>, Electric, (one), Serial No. 1400, Type No. 9921, KW 1.35, Volts 220, Amp. 6.13, Safe working Temp. 1760°F., Maximum 1860°F. Used to fire samples after filtration. Manufactured by Cooley Electric Manufacturing Corporation, Indianapolis, Indiana.

<u>Pipette</u>, 10 ml., 0.1 ml. graduations, (one). Used to measure out small amounts of solutions.

<u>Pipette</u>, 2 ml., 0.1 ml. graduations, (one). Used to measure out small amounts of solutions.

<u>Cylinder</u>, Graduated, 100 ml., 1 ml. graduations, (one). Used to measure out large amounts of solutions.

<u>Jars and Flasks</u>, Glass, assorted, (seven). Used as containers for 1 to 4 HCl, 1 to 2000 HCl, 40% NaOH, 12% Silicotungstic acid, NaCl, distilled water, and lime.

<u>Mortar and Pestle</u>, Porcelain, Coors, (one). Used to pulverize samples of tobacco.

<u>Thermometer</u>, Glass, (one), -15 to 360°C., 1° graduations. Used to read temperature of electric drying oven. Brothcom, New York.

<u>Thermometer</u>, Weston, (one), 200 to 1000°F. 10° graduations. Used to read temperature of electric furnace. Manufactured by the Western Electrical Instrument Corporation, Newark, New Jersey.

<u>Jar</u>, Glass, one pound coffee, (one). Used as HCl gas generator.

<u>Funnel</u>, Separatory, 125 ml., (one). Used to run H_2SO_4 into the gas generator.

<u>Stopper</u>, Rubber, two-hole, No. 12, (one). Used as top of gas generator.

<u>Can</u>, Steel, Standard Oil Chassis Lubrication Grease, (one). Used as gas chamber. (See Figure 2, page 73).

<u>Cloth</u>, Hardware, 1/4" mesh, (200 sq. in). Used as shelves in gas chamber.

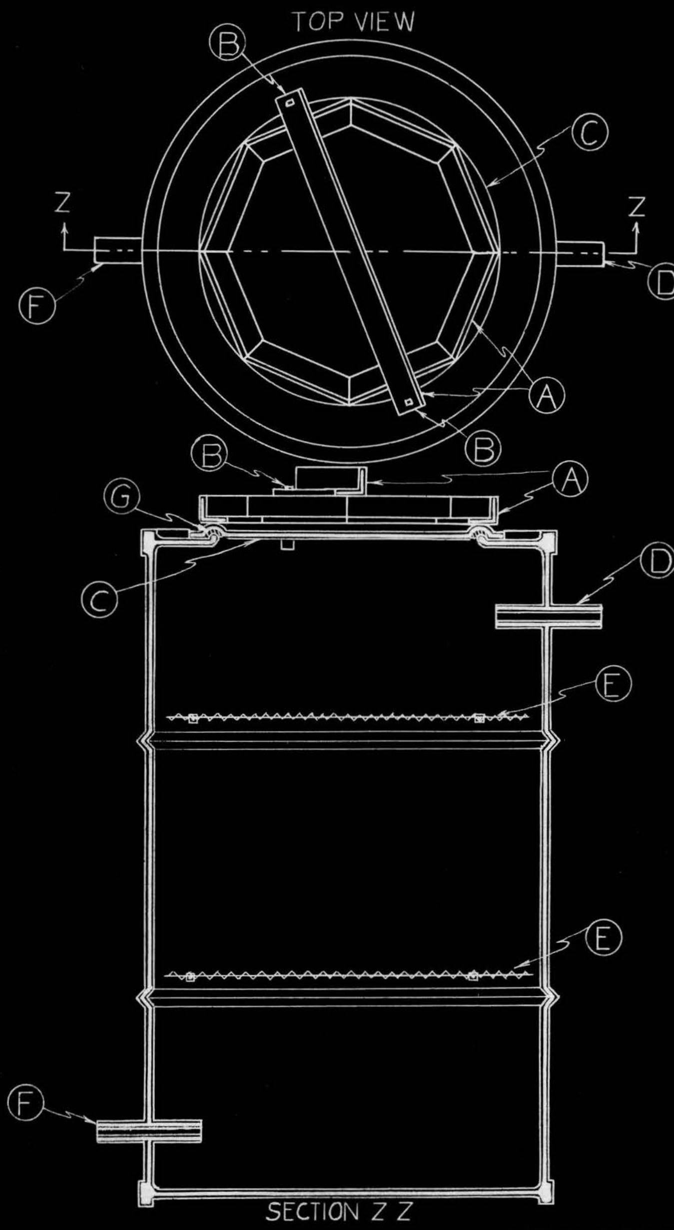
<u>Bolts</u>, Stove, round head, 4" x 1/8", (six). Used as shelf supports in gas chamber.

<u>Pipe</u>, Iron, 3/8" diameter, 6" long, (two). Used as inlet and outlet of gas chamber.

<u>Iron</u>, 1" angle, (39 3/16"). Used as clamps to hold top of gas chamber on securely.

<u>Bolts</u>, Machine, square head, 3" x 3/16", (two). Used to tighten and clamp top of gas chamber by screwing into the welds provided on the gas chamber.

<u>Pinchcocks</u>, (three). Used on inlet and outlet of gas chamber and on the safety valve of steam generator.



LEGEND

A CLAMP
B BOLTS
C TOP
D OUTLET
E SHELF
F INLET
G - GASKET

All parts of gas chamber painted with two coats of Tygon paint as indicated by section ZZ

VIRGINIA POLYTECHNIC INSTITUTE
DEPARTMENT OF CHEMICAL ENGINEERING
Blacksburg, Virginia

GAS CHAMBER

Drawn by: J.C.B.	Date : Nov. 1, 1947
Checked by: Frank C. Vilbrandt	Figure No. : 2
Approved by: F.C.V.	Scale : None

Bottle, Glass, 1/2 pint, (one). Used as H_2O trap between gas chamber and HCl gas absorber.

Stopper, Rubber, two-hole, No. 11, (one). Used as top of water trap.

Jar, Brown glass, one gallon, (one). Used as HCl gas absorber.

Stopper, Rubber, one-hole, No. 12, (one). Used as top for HCl gas absorber.

Tubing, Soft glass, 3 mm., inside diameter, (various lengths). Used as connection between pieces of gassing equipment.

Tubing, Rubber. Used as connections between pieces of gassing equipment. (See Figure 3, page 75).

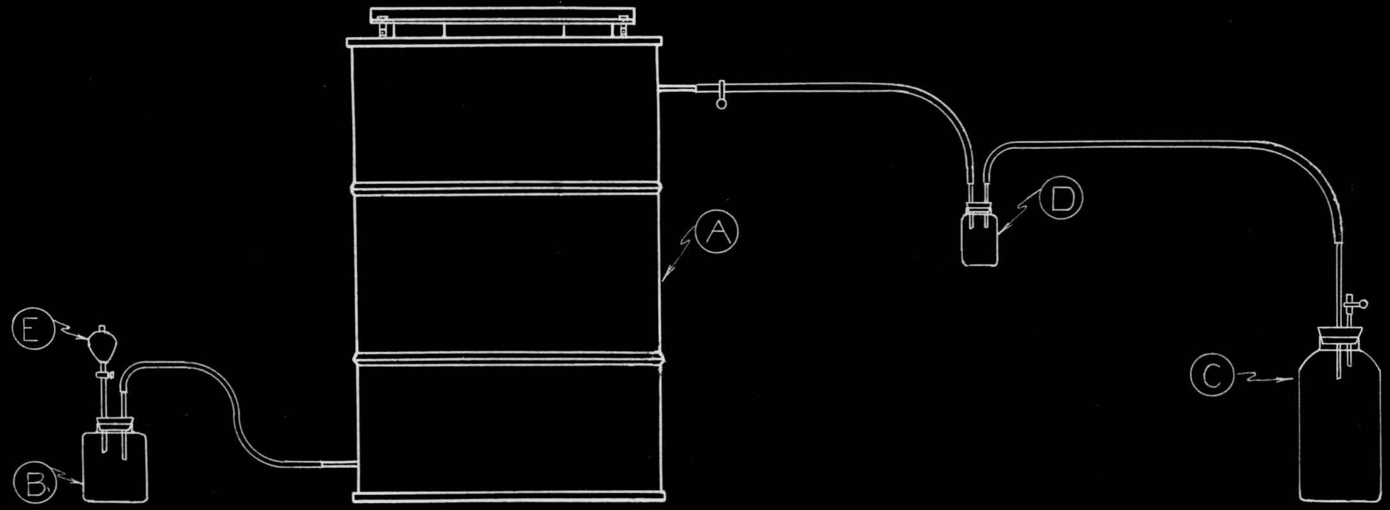
Mill, Patterson Rod and Pebble, Motor driven, (one). Used to grind tobacco.

Pebbles, Stone, 1" diameter (approx.), (10 kg.). Used as grinding unit in rod and pebble mill to grind tobacco.

Pebbles, Stone, 1/2" diameter (approx.), (10 kg.). Used as grinding unit in rod and pebble mill to grind tobacco.

Rods, Steel, miscellaneous, 18" lengths (approx.), (10 kg.). Used as grinding unit in rod and pebble mill to grind tobacco.

Box, Ice, Coco-Cola (one). Used to maintain samples at $3^{o}C$. overnight.



LEGEND

A GAS CHAMBER
B GAS GENERATOR
C ABSORPTION APPARATUS
D WATER TRAP
E SEPARATORY FUNNEL

VIRGINIA POLYTECHNIC INSTITUTE
DEPARTMENT OF CHEMICAL ENGINEERING
Blacksburg, Virginia

ASSEMBLED GASSING EQUIPMENT

Drawn by: J.A.B.	Date: Nov. 1, 1947
Checked by: F.C. Vilbrandt 11/5	Figure No.: 3
Approved by: F.C.V. 11/5	Scale: None

<u>Motor</u>, Induction, General Electric, (one), Type K, Model 5K204A2002, Frame 204, Volts 220/440, Amp. 3.38/1.69, Speed FL 1130, 1 hp., No. JU 2096. Used to drive rod and pebble mill. Manufactured by General Electric Company, Schenectady, New York.

<u>Mixer</u>, Lancaster Countercurrent Rapid Batch, (one), US Patents Nos. 1663830-1728598-1797301, Type SKH, Year 1940, No. 127. Used to grind tobacco. Manufactured by Lancaster Iron Works Inc., Lancaster, Pennsylvania.

<u>Motor</u>, Induction, General Electric, (one), totally enclosed, fan cooled, Model 5KG254920, Frame 254, Type KG, Volts 220/440, Cycles 60, 3 Phase, Amp. 10/5, Speed FL 1130, Patent 1695946-1695947, 3 hp., No. JU 1630. Used to drive Lancaster Countercurrent Rapid Batch Mixer. Manufactured by General Electric Company, Schenectady, New York.

<u>Riddle</u>, Combs Gyratory, (one). Used to separate particle sizes of ground tobacco. Manufactured by the Great Western Manufacturing Company, Leavenworth, Kansas.

<u>Motor</u>, Electric, General Electric, (one). Model 5KH65AB64, Type KH, 1 Phase, 1/6 hp., Cycles 60, Amp. 3.0, Volts 110, RPM 660, Temp. rise 55°C., Continuous. Used to drive riddle. Manufactured by General Electric Company, Schenectady, New York.

Screens, Tyler standard, with pan and top, (three), Nos. 100, 200, and 325. Used to separate particle sizes of ground tobacco. Manufactured by the W. S. Tyler Company, Cleveland, Ohio.

Balance, Triple beam, 500 gms. capacity, 0.1 gm. accuracy, (one). Used to weigh salt. Manufactured by Fisher Scientific Company, Pittsburgh, Pennsylvania.

Balance, Double beam, 21.1 kg. capacity, 1 gm. accuracy, (one). Used to weigh samples of tobacco during grinding. Manufactured by Newark Scale Works, Newark, New Jersey.

Plate, Hot, round, "Autemp" heater, 115 v., (one). Used to heat water for steam bath. Manufactured by Fisher Scientific Company, Pittsburgh, Pennsylvania.

Procedure

Preparation of Solutions. Since none of the solutions used in the analytical determination of nicotine were available, all had to be made up using materials that were available and distilled water.

1. Preparation of 40% Sodium Hydroxide Solution. Flake sodium hydroxide was available reported as 76% Na_2O. This corresponds to approximately 98% pure NaOH. To calculate how

much NaOH to weigh out for the solution, 1000 ml. of distilled water was used as a basis. The proportion 1000 - x : 100 = x : 40 was used. This gave a weight of 666.67 grams. Since the NaOH was only 98%, 666.67 divided by 0.98 gave a total weight of 680.28 grams of the flake NaOH. This amount was weighed out and dissolved in 1000 ml. of distilled water.

2. Preparation of 12% Silicotungstic Acid Solution. Crystal silicotungstic acid, C. P., was available. To calculate how much of the acid to weigh out for the solution, 1000 ml. of distilled water was used as a basis. The proportion 1000 - x : 100 = x : 12 was used. This gave a weight of 136.36 grams of silicotungstic acid. This amount was weighed out and dissolved in 1000 ml. of distilled water.

3. Preparation of 1 to 4 Hydrochloric Acid. Hydrochloric acid, sp. gr. 1.19, assay 36.1%, was available. To calculate how many ml. of the acid to measure out, 1 liter of solution, or 1000 grams, was used as a basis. Each ml. of the available acid contained 1.19 x 0.361 or 0.42959 grams of HCl. A 1 to 4 solution contains 1 gram of HCl to 4 grams of water. Therefore, a liter of the solution, or 1000 grams, will contain 800 grams of water and 200 grams of HCl. The proportion 0.42959 : 1 = 200 : x was used. This gave 465.56 ml. as the amount of original acid to measure out. This amount was measured, put into a 1000 ml. volumetric flask and then the flask was filled to the 1000 ml. mark by adding distilled water.

4. Preparation of 1 to 2000 Hydrochloric Acid. Hydrochloric acid, sp. gr. 1.19, assay 36.1%, was available. To calculate how many ml. of the acid to measure out, 1 liter, or 1000 grams, was used as a basis. Each ml. of the available acid contained 1.19 x 0.361 or 0.42959 grams of HCl. A 1 to 2000 solution contains 1 gram of HCl to 2000 grams of water, or 0.5 grams of HCl to 1000 grams of water. The proportion 0.42959 : 1 = 0.5 : x was used. This gives 1.16 ml. as the amount of original acid to measure out. This amount was measured, put into a 1000 ml. volumetric flask, and then the flask was filled to the 1000 ml. mark by adding distilled water. This did not give an exact 1 to 2000 solution, but since measuring out 1.16 ml. was only an estimate between 1.10 and 1.20 ml., it gives a solution that is adequate since the solution is only used for washing purposes.

Construction and Assembly of Equipment. The equipment used in this investigation can be divided into four major categories. These are: analytical equipment, gassing equipment, grinding equipment, and auxiliary equipment. Only the gassing equipment required any construction.

1. Assembly of Analytical Equipment. All the pieces of apparatus used in the analytical equipment can be obtained from any chemical stock room. The analytical apparatus was assembled on a frame work of iron pipe. A ring was used as a support for the bottom of the 1000 ml. Pyrex round bottom flask and a clamp was used to support the neck. A No. 8,

three-hole rubber stopper was used as the top. A piece of soft glass tubing 3 feet long with an inside diameter of 6 mm. was put into one of the holes so that the end of the tubing was below the surface of the water in the flask. This served as a pressure gage. A 6" piece of 3 mm. inside diameter soft glass tubing was put into another hole in the stopper. A piece of rubber tubing was put on the upper end of this piece of glass tubing and a pinchcock fastened onto the rubber tubing. This served as a safety valve to release excess pressure in the flask. From the third hole, another piece of 3 mm. inside diameter soft glass tubing served as a delivery tube to carry the steam generated to the distillation flask.

The distillation flask, a 500 ml. Pyrex round bottom flask, was supported in the same manner as the steam generator. A No. 6 two-hole rubber stopper was used as the top. Into one hole, a piece of 3 mm. inside diameter, soft glass tubing was inserted so that the end of the tube was only a small distance from the bottom of the flask. This was done so that the end of the tube would be below the surface of the very small amount of liquid that would be in the flask during distillation. This piece of glass tubing was bent and fitted with a small piece of rubber tubing on the end in such a manner that a connection could be made with the delivery tube from the steam generator. The lower portion of a Kjeldahl trap was inserted into the other hole, and the top portion of the trap was bent so as to lead to the top of a water condenser. The water condenser was fitted with a No. 4 one-hole rubber stopper at the top. A piece of 3 mm., inside diameter, soft glass tubing was inserted into this hole, and the upper section of the tubing was bent so that a connection could be made with the upper portion of tubing of the Kjeldahl trap. The connection was made by using a small piece of rubber tubing.

Rubber tubing was used as connections from the water tap to the lower part of the condenser and from the upper part of the condenser to a drain. A Bunsen burner was used to heat the water in the steam generator and a Tirrill burner was used to reduce the volume of liquid in the distillation flask during the distillation. Rubber tubing was used as connections from the gas main to the burners.

Figure 1, page 69, gives a schematic diagram of the assembled analytical apparatus.

2. <u>Construction of Gas Chamber</u>. A "Standard Oil Chassis Lubrication Grease" steel can was used as a gas chamber. The can was provided with a top that was fitted with a rubber gasket. The can was first cleaned inside and out with gasoline to remove all the grease. Then a hole, 3/8" diameter, was bored 2" from the bottom of the can. Another hole, 3/8" diameter, was bored opposite the first hole and 2 3/8" from the top of the can. A 6" piece of 3/8" iron pipe was then welded in these holes to serve as

inlet at the bottom, and outlet at the top, for HCl gas.

A 1/8" diameter hole was then bored in the side of the can 8" above the bottom. Then two more holes were bored on approximately 120° angles from the first hole. A 1/8" round head stove bolt, 4" long, was put into each of these holes and a nut screwed on them on the inside of the can to secure them. These three bolts served as the supports for a 100 sq. in shelf of 1/4" mesh hardware cloth.

Three more 1/8" diameter holes were bored in the can in the same manner 17" above the bottom of the can. These holes were fitted with stove bolts for another shelf of the hardware cloth.

In order for the gas chamber to be leak proof, some clamp had to be devised for clamping the top of the can on securely. The can was provided with two wells exactly opposite each other into which 3/16" bolts would fit. A piece of 1" angle iron 28" long was formed into an octagon with 3 1/2" sides. Two holes, 3/16" diameter, 10 1/2" center to center were bored in one of the sides of a 11 3/16" piece of the angle iron. These two pieces of angle iron served as a clamp for the top. The octagon was placed on the top, exerting pressure at eight points on the top. The straight piece of angle iron was then put across the octagon and a square head machine bolt 3/16" diameter and 3" long was inserted through the hole in each end and screwed into the wells provided in the can.

Shelves of 1/4" mesh hardware cloth, 10" square, was then cut and put into the can.

HCl gas is very corrosive, so the can had to be protected from this. The can, inside and out, including shelf supports, top, clamp, and inlet and outlet pipes, was painted with a thick coat of Tygon white paint. This first coat was allowed to dry for about a week, and then another thick coat of Tygon was applied.

Figure 2, page 73, gives a drawing of the completed gas chamber.

3. <u>Assembly of Gassing Equipment</u>. No construction was required for the rest of the gassing apparatus. All that was necessary was to assemble the individual pieces. A one pound glass coffee jar was used as the HCl gas generator. A No. 12 two-hole rubber stopper was used as a top. A 125 ml. separatory funnel was inserted into one of the holes to provide a means of running the H_2SO_4 into the generator to react with the NaCl and produce HCl gas. A piece of 3 mm. inside diameter soft glass tubing was then used to connect the gas generator with the inlet of the gas chamber. This piece of rubber tubing was provided with a pinchcock so that the flow of HCl gas to or from the gas chamber could be stopped or started at will.

The gas generator and the gas chamber were the two main pieces of the gassing equipment. The HCl gas absorber was provided to absorb all excess HCl gas that would be present after the termination of an experimental run.

A water trap was provided between the gas chamber and absorber to absorb any water vapor that might get into the chamber from the HCl gas absorber. A 1/2 pint glass jar was used as the water trap. This jar was filled approximately one third full with H_2SO_4 to absorb the water. A No. 11 two-hole rubber stopper was used as the top. Into each of the two holes, a piece of 3 mm., inside diameter soft glass tubing was inserted. One of these was connected to the outlet of the gas chamber by using rubber tubing. This piece of tubing was also provided with a pinchcock so that the flow of HCl gas from the chamber could be started or stopped at will. The piece of tubing to be connected with the absorption chamber was adjusted so that the end of the tubing was just above the surface of the H_2SO_4 in the water trap.

A one gallon brown glass jar was used as the HCl gas absorber. This was filled about two thirds full with water. A No. 12 one-hole rubber stopper was used as a top. A piece of 3 mm., inside diameter soft glass tubing was inserted into the hole and adjusted so that the end of the tubing was just above the surface of the water. The water trap and HCl gas absorber were connected with a piece of rubber tubing.

Figure 3, page 75, gives a drawing of the assembled gassing equipment.

4. Grinding and Auxiliary Equipment. All pieces of grinding equipment and all pieces of auxiliary equipment were either standard equipment in the Unit Operations Laboratory of the Chemical Engineering Department of the Virginia Polytechnic Institute or could be obtained from any chemical stock room. Hence no construction or assembly was necessary.

Sampling. Both whole leaf and finely ground tobacco were used in the analytical determination of nicotine. Hence, a method of sampling that would give a uniform sample had to be devised for each.

1. Sampling Leaf Tobacco. About two pounds of the tobacco were selected as the material for tests involving the whole tobacco leaf. The leaf was torn up into smaller pieces of different sizes; the smallest pieces still being large enough to retain its leafy characteristics. Then all of this was mixed thoroughly with the hands and placed in a container. When a sample was desired, pieces were removed from the container, and smaller pieces torn from them to be weighed as the sample. When enough had been obtained for a single sample, the remaining pieces were put back into the container and the whole lot remixed with the hands.

2. Sampling Ground Tobacco. No special method of sampling was used. As a sample was needed, it was taken from the container, the assumption being that a thorough mixing had occurred during the grinding process and shaking on the riddle.

Weighing of Samples. All samples used in the moisture determinations and determinations of nicotine content were weighed on a Chainomatic analytical balance. The weighing of the residue in the analytical determination of nicotine was also done on the analytical balance. The weighing of the salt used in the gas generator to produce the HCl gas was done on a triple beam laboratory balance of 500 gms. capacity.

1. Weighing Samples for Moisture Determination and Nicotine Determinations. A 2" watchglass was first weighed accurately. Then an adjustment of the weights was made so that when enough sample was added to balance the weights, a sample of 0.5000 gm. or 1.0000 gm. was weighed out, depending on the adjustment of the weights.

2. Weighing of Residue. The residue was weighed in a porcelain crucible. The crucible had been weighed previously and the weight of the residue was obtained by subtracting the tare weight of the crucible from the total weight of the crucible and residue.

3. Weighing of Samples After Drying in Moisture Determination. The dried sample was weighed in a glass weighing bottle. The weighing bottle had been weighed previously and the weight of the dried sample was obtained by subtracting the tare weight of the weighing bottle from the total weight of the bottle and dried sample.

4. Weighing of Salt. A sheet of paper was placed on the balance and weighed. The weights on the beam of the triple beam laboratory balance were then adjusted so that when enough salt had been put on the paper to balance the weights, 100 gms. of salt would be weighed out. Extreme accuracy was not necessary in this operation.

Determination of Moisture Content. Each time that samples were weighed out for the determination of nicotine, a moisture determination was made. The method used was that suggested and used by Wells[48]. A 1.0000 gm. sample of tobacco was weighed out and put into an evaporating dish. The sample was then put into the electric drying oven. The temperature was maintained at 140°C. At the end of 24 hours, the sample was removed from the oven and immediately transferred to a glass weighing bottle to prevent the dried sample from picking up moisture from the atmosphere. The weight of the dried sample was then determined as previously mentioned. The weight of the dried sample was then subtracted from the original weight and the difference was divided by the original weight to determine the percentage moisture.

Gassing of Samples. A description of the procedure for one gassing operation will suffice since each was done in exactly the same manner. The sample to be gassed was placed on the shelf, E, (Figure 2) of the gas chamber (the leaf tobacco on a piece of paper and the ground tobacco on a watchglass). The top, C, was then placed on the chamber and clamped tightly on by screwing the bolts, B, of the clamp, A, into the wells provided in the can. The outlet, D, of the gas chamber was closed by means of the pinchcock on the rubber tubing connecting the outlet of the chamber to the water trap, C, (Figure 3). One hundred grams of NaCl was put into the gas generator, B, and the generator was connected to the gas chamber. Forty ml. of H_2SO_4 was then put into the separatory funnel, E. The entire amount of H_2SO_4 was then allowed to run into the gas generator producing the HCl gas. After one hour had passed, the outlet of the gas chamber was opened and the excess HCl was absorbed in the absorption apparatus, D. This was done only if the duration of the gassing operation was one hour. If the duration of the operation was more than one hour, at the end of each hour the inlet, F, (Figure 2) to the gas chamber was closed and the gas generator was disconnected, washed out and recharged with 100 gms. of salt. Then 40 ml. more of HCl gas was run into the generator, and the inlet opened by removing the pinchcock. Then at the end of the second hour in a two hour operation, or at the end of the third hour in a three hour run, the excess HCl gas was absorbed as described above. The top of the gas chamber was then removed and the gassed samples removed from the gas chamber.

Preparation of Samples for Analysis. A sample of lime equal in weight to the gassed sample was weighed out while the sample was being gassed. Immediately upon removal of the gassed sample from the gas chamber, it was mixed with lime. Approximately

20 drops of water was then added to facilitate the reaction between the lime and any nicotine hydrochloride. The sample was then allowed to set from 40 to 48 hours before the nicotine determination was made. Blank samples (not gassed) were handled in the same manner.

Analytical Determinations of Nicotine. A description of one analytical determination of nicotine will suffice since each individual analysis was done in exactly the same manner.

The sample to be analyized was put into the distillation flask, D, (Figure 1). Using a pipette, 3 ml. of distilled water was put into the flask. Five ml. of water was put into the distillate receiver, F. Then 3 ml. of 1 to 4 HCl was put into the distillate receiver. The distillate receiver was then put into place at the foot of the condenser, E, so that the end of the condenser was below the surface of the liquid in the distillate receiver. Two drops of phenophlthalein was then put into the distillation flask. Since the samples had already been mixed with lime, it was alkaline, but 0.5 ml. of 40% NaOH was added to the distillation flask. Meanwhile the Bunsen burner had been heating the water in the steam generator, A, to the boiling point, and the cold tap water had been turned on so that it was running through the condenser. The connection was made between the distillation flask and the condenser. As soon as the water in the steam generator was boiling steadily, the connection was made between the steam generator and the distillation flask, and the distillation begun. As soon as the distillation was going smoothly, heat was applied to the distillation flask by means of the Tirrill burner. This was done to reduce the volume of the liquid in the flask. A pressure of 2.5 to 3.0 feet of water was maintained in the steam generator. This was observed by means of the pressure gage. Distillation was continued until about 100 ml. of distillate was collected and the liquid in the distillation flask was reduced to dryness. One ml. of 12% silicotungstic acid was then added to the distillate to precipitate the nicotine that had been distilled over. During the distillation, water in a 250 ml. beaker had been boiling on a hot plate. The sample was put on this steam bath for 15 minutes. While the sample was on the steam bath, the analytical apparatus was disassembled and the distillation flask, trap, condenser and delivery tube were washed out and reassembled for the next run. At the end of the 15 minutes, the sample was removed from the steam bath and allowed to cool slowly to room temperature. The sample was then placed in an ice box and maintained at approximately 3°C. overnight. The precipitate was filtered out on Whatman No. 40 filter paper. To make sure that all of the precipitate was gotten out of the distillate receiver, half of the filtrate was poured back into the distillate receiver and refiltered, and then a quarter of the filtrate was poured back into the distillate receiver and refiltered. The precipitate was then washed with 150 ml. of 1 to 2000 HCl. The filter paper was then placed in a crucible and put into the electric furnace. The temperature of the furnace was maintained at 960°F. As soon as the filter paper had been completely burned, the crucible was removed from the furnace and heated over the Bunsen burner for 10 minutes. The crucible was then placed in a desiccator and allowed to cool for 1 hour. At the end of the cooling period, the weight of crucible and residue was determined as previously described. The weight of the residue was multiplied by 0.114 to give the weight of nicotine. The weight of nicotine was divided by the weight of sample to give the nicotine percentage on the wet basis. Further division of this by 100% minus the moisture content gave the nicotine content on the dry basis.

Calculation of Percentage Nicotine Lost By Vaporization After Gassing and Mixing With Lime. The dry basis nicotine content of a gassed sample was divided by the dry basis nicotine of the blank (not gassed) sample. This gave the percentage of original nicotine still present in the sample. This amount subtracted from 100% gave the percentage of original nicotine lost by vaporization. This is reported as the percentage nicotine converted.

Grinding Tobacco. The grinding with each of three grinding units; 1" pebbles, 1/2" pebbles, and rods was done in exactly the same manner in the Patterson Rod and Pebble Mill. Five hundred grams of tobacco was weighed out and put into the mill. Then 10 kg. of the grinding unit was put into the mill. The top of the mill was put into place and clamped. The mill was started and the grinding operation was continued for 5 hours. At the end of the 5 hours, the grinding unit was removed from the mill, caution being taken to brush off all that was possible of the tobacco that had adhered to the unit, Then the ground tobacco was brushed from the mill into the screens for a screen analysis. After the screen analysis, the total sample was put back into the mill, the grinding unit was again put into the mill, and the grinding operation carried out for another 5 hours. In practice only that portion insufficiently ground would be returned to the mill. In this study, the degree of grinding in a definite length of time was desired.

Screen Analysis. The screens used were 100, 200, and 325 mesh with pan and top. The ground tobacco was put into the 100 mesh screen. The top was placed on this and the set of screens was placed in the riddle. The riddle was run for 5 hours. The screens were then removed from the riddle and the individual screens and pan with the tobacco on it were weighed. The weight of the screen and tobacco minus the tare weight of the screen, which had been determined previously, gave the amount of tobacco that had passed through the screen above the screen being weighed. This weight of tobacco divided by the total weight gave the percentage of tobacco through the screen above the screen being weighed.

Method of Increasing Moisture Content. Some of the ground tobacco that had passed through a 200 mesh screen was placed on a piece of cotton cloth. The cloth was then folded to form a bag when held in the hands. This bag was then held in the stream of steam issuing from the delivery tube, B, (Figure 1) of the steam generator. The bag was rotated so that steam would strike all sides of it. From time to time the tobacco in the bag was agitated to expose all the tobacco to the steam. This was continued for about 15 minutes. Then the tobacco was put into a glass jar and allowed to set for several days to give the moisture a chance to become evenly distributed through the entire mass of tobacco.

Determination of Free and Combined Nicotine in Tobacco. A sample (2-3 grams) of tobacco was placed in the distillation flask and covered with H_2O. No caustic was added. Ten minutes after distillation was begun the distillate receiver was removed and another inserted in its place. This was repeated at the end of each 10 minute interval until 80 minutes had passed. As each distillate receiver was removed, the nicotine distilled was precipitated with silicotungstic acid. Then the rest of the procedure in the analytical determination of nicotine was carried out. Upon determining how much nicotine had been distilled each 10 minutes, a curve was plotted with time in minutes as the abscissa and the weight of nicotine in mg. distilled per 10 minutes as the ordinate. By assuming that the hydrolysis of the combined nicotine to free nicotine and the subsequent distillation of the free nicotine would take place in a straight line relationship, and that the free nicotine originally present would distill off during the first time intervals, the relative proportion of free nicotine to total nicotine content could be observed.

Treatment with NH_3. The treatment of the tobacco with the NH_3 was very similar to treatment with HCl gas. Approximately 100 ml. of NH_4OH was put into the gas generator. The samples of tobacco to be treated were put into the gas chamber and the top was clamped into place. With the outlet of the chamber closed, the NH_4OH in the gas generator was heated with a hot plate. This liberated NH_3 from the NH_4OH. The treatment with the NH_3 was for 1 hour.

Tests with N. rustica. All tests performed with N. rustica were exactly the same as with ordinary tobacco with the exception that in the analysis for nicotine only 0.2000 gram samples were used because of the high nicotine content. Also, in grinding, the N. rustica was ground for only 1 hour with rods.

Calculation of Cost of Growing One Acre of N. rustica. The cost of growing one acre of N. rustica was calculated on the same basis as Underwood[28] calculated the cost of growing one acre of flue cured tobacco. The cost of man labor was calculated at $0.197 per hour and the cost of horse work was calculated at $0.11 per hour. For other cost items, such as use of tools, land, etc., the cost charged to one acre of N. rustica was the same as charged by Underwood. In calculating the cost of marketing the N. rustica, some items had to be determined by other means. For instance, the item- Truck use- was determined by the price charged ($0.30 per hundred pounds) at the present time by truck owners to haul the flue cured tobacco from surrounding territory to the Chase City, Va. tobacco market. Also the item- hired hauling- was calculated on the railroad freight rate for carload lots of cut tobacco in no containers ($1.13 per hundred pounds) from Chase City, Va. to Richmond, Va. The figure for the total poundage produced by one acre of N. rustica was the average poundage produced during the three years of experimentation at Arlington Farms, Va.

Data and Results

The following tables and figures give the observed data and the results calculated during the course of this investigation.

Table XXIII, page 98, gives a detailed statement of the cost of producing one acre of N. rustica tobacco.

Table XXIV, page 99, gives a summary of all costs and returns in raising one acre of N. rustica tobacco. The values marked with an asterick(*) are figures for flue cured tobacco taken from Table XX, page 38. These figures were included for purposes of comparison.

Table XXV, page 100, gives the effect of particle size, moisture content, treatment with NH_3, and time of exposure to HCl gas, on the conversion of nicotine in tobacco to nicotine hydrochloride.

Table XXVI, page 101, gives the results of screen analysis of tobacco after being ground in the rod and pebble mill using 1" pebbles, 1/2" pebbles, and rods as the grinding unit.

Table XXVII, page 102, gives the effect of grinding on the loss of nicotine from tobacco by vaporization due to heat generated during the grinding.

Figure 4, page 103, is the plot of the data contained in Table XXVI.

Table XXVIII, page 104, gives the results of steam distillation of ordinary tobacco to determine the proportion of free nicotine to total nicotine content.

Table XXIX, page 105, gives the results of steam distillation of N. rustica tobacco to determine the proportion of free nicotine to total nicotine content.

Figure 5, page 106, is the plot of the data contained in Tables XXVII and XXVIII.

Table XXIII

Detailed Statement of the Cost of Producing One Acre of
N. rustica Tobacco

Item	Average Per Acre		Total
	Quantity	Value	
Growing:			
Man labor, hours @ $0.197	212.00	$41.76	
Horse or mule work, hours @ $0.11	68.00	7.48	
Tractor use, hours	0.14	0.03	
Truck use, miles	0.02		
Plant bed cloth		0.36	
Other equipment		0.99	
Seed and plants		0.13	
Fertilizer, pounds	888.60	9.07	
Manure, pounds	272.90	0.71	
Cover crop		0.18	
Spray and dust		0.02	
Insurance		7.32	
Interest @ 6% per annum		1.57	
Land use		2.26	$71.88
Harvesting:			
Man labor, hours	58.00	$11.43	
Horse or mule work, hours	22.00	2.42	
Other equipment		0.21	
Interest		0.23	
Other harvesting costs		0.21	$14.50
Curing:			
Man labor, hours	1.00	$ 0.20	
Other equipment		1.30	
Building use		4.28	
Interest		0.35	
Other curing costs		0.08	$ 6.21
Marketing:			
Man labor, hours	10.00	$ 1.97	
Horse or mule work, hours	20.00	2.20	
Truck use		11.15	
Automobile use		1.60	
Other equipment		0.17	
Hired hauling, pounds	3,718.00	42.01	
Storage building use		1.54	
Interest on tobacco		1.25	
Other marketing costs		0.01	$61.90
Grand Total			$154.49

Table XXIV

Summary of All Costs and Returns in Raising One Acre of N. rustica Tobacco

Item	Average Per Acre	
	Quantity	Value
Man labor, hours	281.00	$55.36
Horse or mule work, hours	110.00	12.10
Tractor use, hours	0.04	0.03
Automotive transportation		12.75
Use of other equipment		3.03
Feed, fertilizer, and poison		10.11
Use of land and buildings		8.08
Insurance		7.32
Interest		3.40
Hired hauling, pounds	3,718.00	42.01
Miscellaneous		0.30
Total all costs		$154.49
Tobacco sales, pounds @ $0.04	3,718.00	148.72
Net loss	$39.01*	5.77
Unpaid labor	86.24*	55.36
Net profit	47.23*	49.59

*These figures do not apply to N. rustica. They are the figures for flue cured tobacco inserted here for comparison purposes.

Table XXV

Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride

Test No.	Weight of Sample gm.	Type of Tobacco	Size of Tobacco	Time of Treatment With HCl hrs.	Time of Treatment With NH_3 hrs.	Moisture Content 140°C 24 hrs. %	Moisture Content 105°C 24 hrs. %	Yield of Nicotine mg.	Nicotine Content (Dry basis) %	Nicotine Converted %
1-3	0.5000	Ordinary	Leaf	-	-	16.39	-	6.2928	1.51	-
4-6	0.5000	Ordinary	Leaf	1	-	16.39	-	5.2906	1.27	15.89
7-9	0.5000	Ordinary	Leaf	2	-	18.23	-	5.2896	1.29	14.57
10-12	0.5000	Ordinary	Leaf	3	-	18.01	-	5.3124	1.30	13.91
13-16	0.5000	Ordinary	Ground	-	-	18.50	-	3.8532	0.95	-
17-19	0.5000	Ordinary	Ground	1	-	18.50	-	3.4086	0.84	11.58
20-22	0.5000	Ordinary	Ground	2	-	17.47	-	3.4884	0.85	10.53
23-26	0.5000	Ordinary	Ground	1	-	45.04	-	2.3256	0.85	10.53
27-29	0.5000	Ordinary	Ground	-	-	18.39	14.18	5.7114	1.40	-
30-35	0.5000	Ordinary	Ground	1	1	18.39	14.18	4.3890	1.08	22.86
36-37	0.2000	rustica	Ground	-	-	15.48	-	4.5114	2.67	-
38-40	0.2000	rustica	Ground	1	1	15.48	-	2.1090	1.25	53.18

Table XXVI

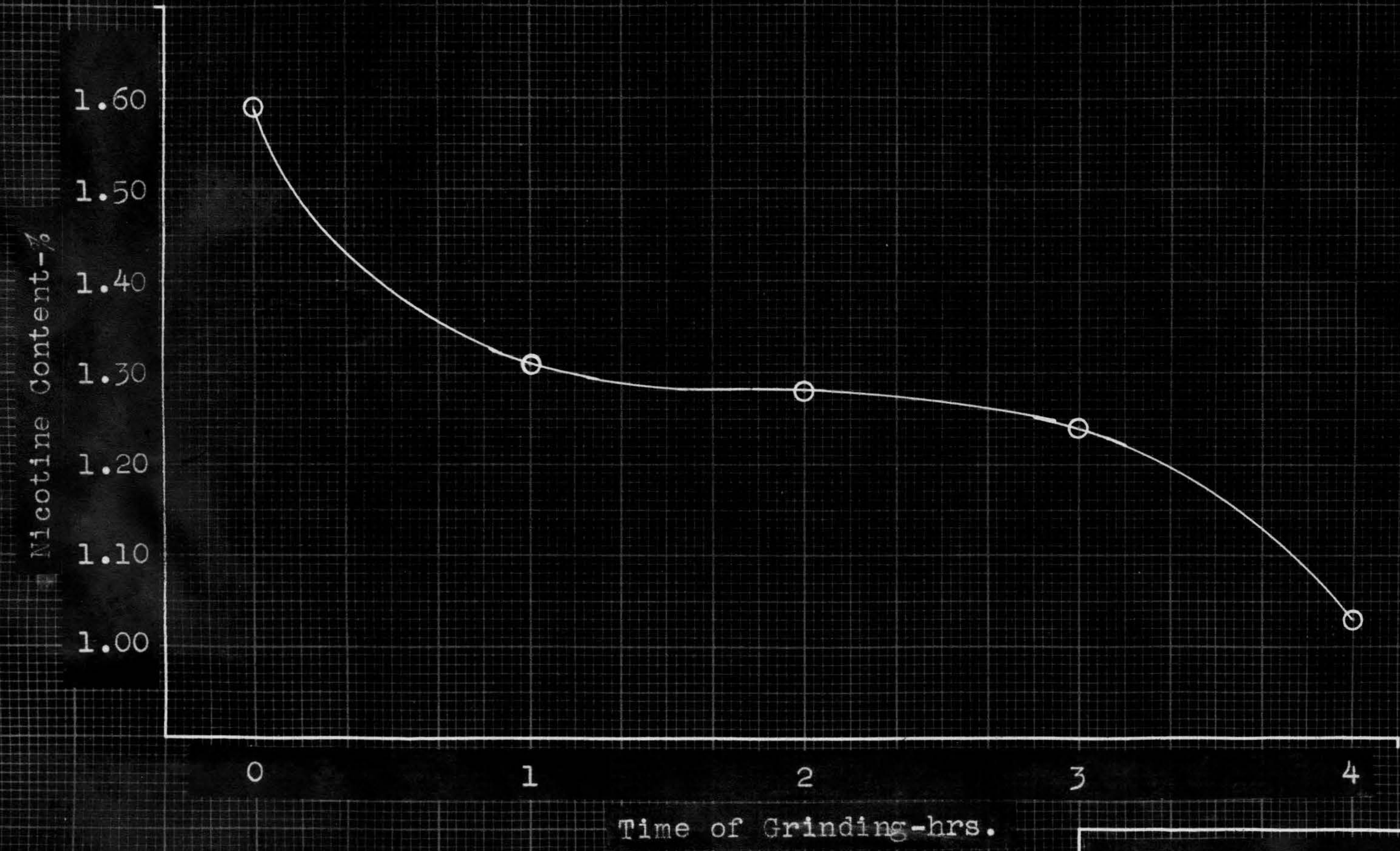
Results of Screen Analysis of Tobacco After Being Ground in the Rod and Pebble Mill Using 1" Pebbles, 1/2" Pebbles, and Rods as the Grinding Units

Test No.	Grinding Unit	Time of Grinding hrs.	Moisture Content 140°C 24 hrs. %	Retained on 100 Mesh Screen %	Through 100 & Retained on 200 Mesh Screen %	Through 200 & Retained on 325 Mesh Screen %
1a	½"peb.	5	18.67	27.80	17.20	47.00
1b	½"peb.	10	18.67	18.80	15.20	55.20
2a	1"peb.	5	17.70	23.80	26.20	46.60
2b	1"peb.	10	17.70	14.20	60.20	20.60
3a	rods	5	17.70	26.20	20.20	53.60
3b	rods	10	17.70	11.40	25.20	66.60

Table XXVII

Effect of Grinding With Rods on the Loss of Nicotine From Tobacco by Vaporization Due to Heat Generated During the Grinding

Test No.	Time of Grinding hrs.	Weight of Sample gm.	Yield of Nicotine mg.	Moisture Content 140°C. 24 hrs. %	Nicotine Content (Dry basis) %
1-4	-	0.5000	6.8058	14.49	1.59
5-8	1	0.5000	5.5974	15.27	1.31
9-12	2	0.5000	5.4720	14.70	1.28
13-16	3	0.5000	5.2654	15.04	1.24
17-20	4	0.5000	4.4232	14.28	1.03



Department of Chemical Engineering
Virginia Polytechnic Institute
Blacksburg, Virginia

Time of Grinding
vs
Nicotine Content

Drawn by: J.W.B. Date: Nov. 25, '47
Approved by: F.C.V. Figure No.: 4

Table XXVIII

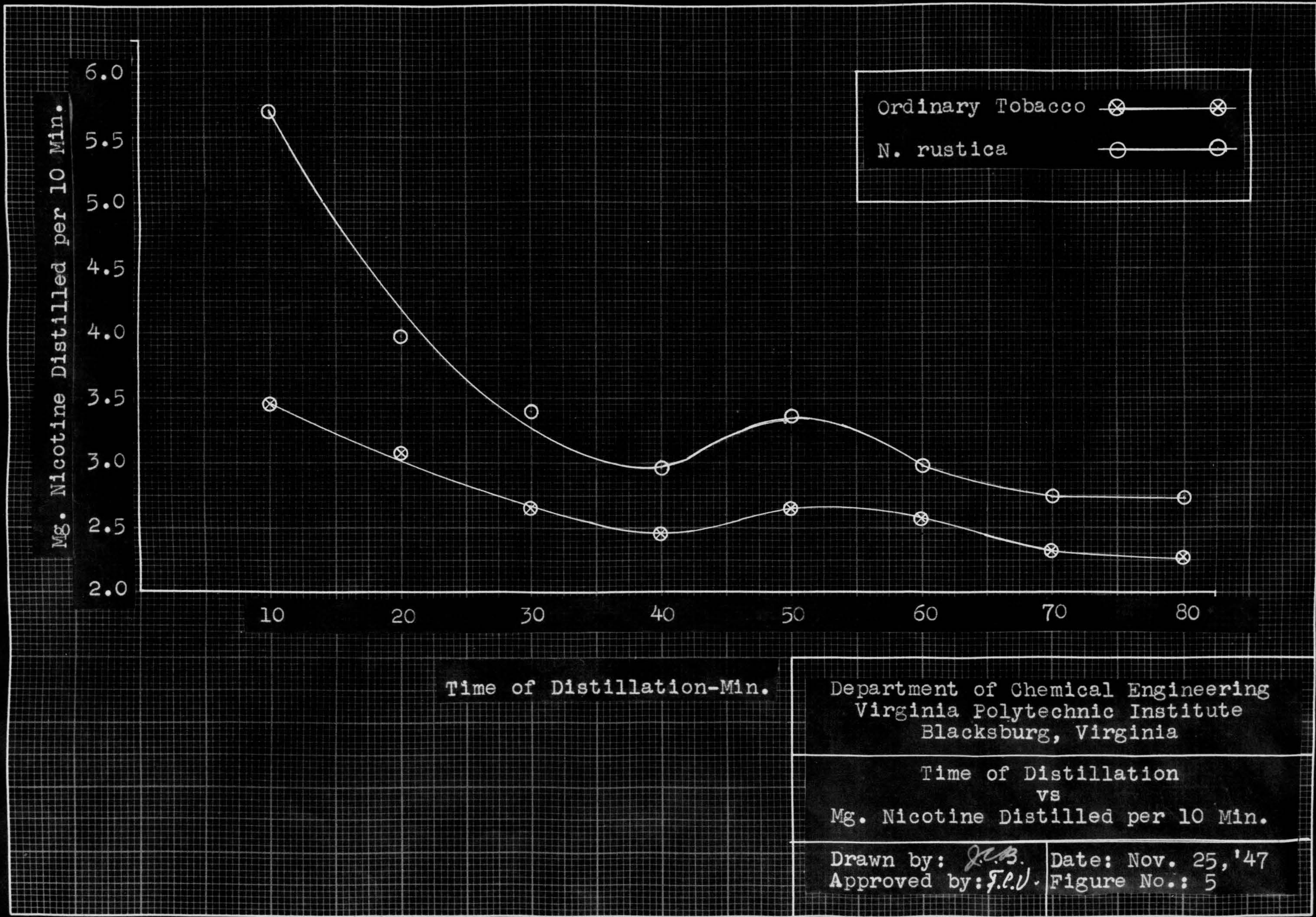
Results of Steam Distillation of Ordinary Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content

Sample No.	Time of Distillation min.	Yield of Nicotine mg.
1	10	3.4542
2	20	3.0780
3	30	2.6676
4	40	2.4510
5	50	2.6676
6	60	2.5878
7	70	2.3142
8	80	2.2572

Table XXIX

Results of Steam Distillation of N. rustica Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content

Sample No.	Time of Distillation min.	Yield of Nicotine mg.
1	10	5.7000
2	20	3.9786
3	30	3.3402
4	40	2.9526
5	50	3.3516
6	60	2.9868
7	70	2.7474
8	80	2.7360


Ordinary Tobacco
N. rustica
Mg. Nicotine Distilled per 10 Min.
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
10
20
30
40
50
60
70
80
Time of Distillation-Min.
Department of Chemical Engineering
Virginia Polytechnic Institute
Blacksburg, Virginia
Time of Distillation
vs
Mg. Nicotine Distilled per 10 Min.
Drawn by: J.C.B.
Approved by: F.C.V.
Date: Nov. 25, '47
Figure No.: 5

IV. DISCUSSION OF RESULTS

Economic Feasibility of Growing N. rustica as a Cash Crop. The economic study of growing flue cured tobacco made by Underwood[28] was for the 1933 flue cured tobacco crop in Pittsylvania County, Va. The records on which the study was based were obtained by the survey method. It was the object of the study to collect and analyze statistically data concerning the organization and operation of a comparatively large number of tobacco farms for the purpose of determining the relative importance of the major factors associated with variations between farms in the returns from tobacco.

Economic conditions are constantly changing, and a given set of conditions may not be repeated for many years. Hence, the economic situation in 1939, when the results of the survey were published, was not the same as it was at the time the survey was made. Neither is the situation the same today as it was in 1933 or in 1939.

N. rustica has never been grown as a commercial crop in the United States. In fact, the extent to which N. rustica has been grown has been limited to small experimental patches. In growing these small experimental patches, the emphasis has been on the maximum production of nicotine per acre, either by increasing the percentage of nicotine, increasing the poundage per acre, or by a combination of both.

Very few farmers would be able to state at once the total hours of labor required to produce an acre of tobacco, but most of them can name fairly accurately the tasks that were performed and the time required for each, such as fallowing or breaking the land and setting plants, so that a correct total may be obtained. In the case of N. rustica such information was not available. The labor requirements for N. rustica have therefore been determined by other means. As the number of plants grown per acre is essentially the same as in the case of the burley type of tobacco, the labor requirement surveys that have been made for the latter should be more or less applicable to N. rustica with respect to most of the important cultural operations. One group of operations, however, would not be the same, namely, topping, worming, suckering, and spraying or dusting. This complex item would correspond more nearly to the requirements for dark-fired tobacco except that, since there are approximately twice the number of plants per acre, this figure would need to be doubled. Figures for the several major operations are derived in this way. This item is highly important because the labor costs in growing tobacco is a high percentage of the total cost. For example, the cost of man labor and horse work constituted 75.1% of the total cost of growing flue cured tobacco as computed by Underwood(28). For N. rustica, the cost of man labor and horse work was 57.8% of the total cost. The important thing to remember is that while the man labor is charged in the cost accounting, it seldom represents a cash outlay since the labor is done by the farmer and members of his family. After all other expenses have been met, the farmer can then pay himself for his labor. By dividing the total number of hours worked into the money left, the farmer can determine his hourly wage rate.

In the case of the flue-cured tobacco there existed a net profit of $42.23 for 438.6 hours. This gives an hourly rate of $0.108. For N. rustica, the economic study based on the study made by Underwood(28) shows a net profit of $49.59 for 281 hours. This gives an hourly rate of $0.177. Therefore, the farmer is being paid more for his labor and at the same time has 157.6 hours in which to engage in some other profitable enterprise.

As stated before, economic conditions vary, and a given set of conditions may not be repeated for many years. The time at which Underwood conducted his survey could not be considered normal. Neither can the present be considered normal. Certainly, then, any conclusions drawn from the economic study can not apply today, nor can they apply to what may be considered a normal year.

However, unless the methods of cultivation in growing tobacco change radically, and this is very unlikely, the labor required will continue to constitute a majority of the cost of growing either flue cured tobacco or N. rustica. If the price paid for tobacco, the production per acre, and costs other than labor all vary from time to time, and these items are known, they can be substituted for the values as given in Table XX, Summary of All Costs and Returns in Producing Tobacco, Table XXIII, Detailed Statement of the Cost of Producing One Acre of N. rustica Tobacco, and Table XXIV, Summary of All Costs and Returns in Raising One Acre of N. rustica Tobacco, and it can be determined whether or not it would be economical to grow N. rustica as a cash crop as compared to growing flue cured tobacco as a cash crop.

Determination of Moisture in Tobacco. The purpose of the moisture determination was to obtain data so that all nicotine percentages could be expressed on the same basis-the dry basis. It is a known fact that the moisture content of tobacco exposed to the atmosphere will change with a change in atmospheric conditions. This plus the fact that the investigation was to extend over a long period of time made the dry basis the only logical basis for expressing the nicotine content, and frequent moisture determinations were therefore necessary. Wells[48] cites five methods of determining moisture in tobacco. One method was heating the tobacco for 24 hours at 140°C. and determining weight loss. This method was used throughout the investigation.

Loss Due to Heating at 140°C. for 24 Hours. During the course of the investigation when it was discovered that subjecting the tobacco to heat would probably cause some of the nicotine to volatilize off, a nicotine analysis was made on a sample of tobacco that had been used in a moisture determination. The results of this analysis proved that this particular method of moisture determination was unsatisfactory. For samples of 0.2000 gm. an average residue of 0.0050 gm. was obtained; this is a nicotine content of 0.285%. The nicotine content of a sample that had not been dried was 2.76% on the dry basis.

Loss Due to Heating at 105°C. for 24 Hours. It was suggested that the 140°C. was too high and that heating at a lower temperature such as 105°C. would be better. An analysis of samples of tobacco that had been dried at such a temperature proved that this also was unsatisfactory. For samples of 0.2000 gm. an average residue of 0.0130 gm. was obtained. This is a nicotine content of 0.74%. This was also a drop from 2.76%.

Loss of Bound Moisture by Increase in Drying Temperature. Another difference was observed in the results of using the two temperatures in the moisture determinations. With samples which were weighed out at the same time, those which had been dried at 140°C. for 24 hrs. gave an average moisture content of 18.39% while those which had been dried at 105°C. for 24 hrs. gave an average moisture content of 14.18%. This was a difference of 4.21% which was too much to be accounted for by the amount of nicotine vaporized. Therefore, the drying at 140°C. was either decomposing some constituent in the tobacco or driving off bound moisture; the latter is most likely true.

These observations, along with the fact that there was a loss of nicotine in grinding due to heat generated, indicate that the use of heat to the extent where it would be positive that all the moisture will be driven off, and determining the loss by weighing would be an unsatisfactory method.

All nicotine contents in this investigation are reported on the so called "dry-basis" but which in reality is a moisture free-nicotine free basis. In the case of the ordinary tobacco where the nicotine content never exceeded 1.60%, the nicotine content on an actual dry basis would not differ to any great extent from that which is reported. However, with N. rustica of 5-7% nicotine or higher, the difference would be considerable.

Importance of Sampling. The nicotine content of tobacco varies a great deal. It is known that the nicotine content of tobacco varies from one part of the leaf to another; from plant to plant, and from field to field. For example, the distribution of nicotine in N. rustica has been reported as follows: percentage (water-free basis) of leaf tissue, 8.08-12.80; percentage (water-free basis) of leaf stem or midrib, 2.99-3.34; and percentage (water-free basis) of main plant stalk, 1.65-2.79. In flue cured tobacco a proportional distribution could be expected. Therefore, it is very essential that representative samples be obtained if the data obtained by such a sample is to be used in processing a large amount.

In this investigation, such a representative sample was not obtained. All the ordinary tobacco used was scrap and it came from three different farms. Also, being scrap, the tobacco contained varying amounts of "dead" tobacco, that which had been left in the field too long before harvesting. These factors added up to give a material with a large range of nicotine content with which to work.

While a satisfactory method of sampling was worked out for small amounts, no method of sampling so that the results obtained could be applied to all of the tobacco was attempted or developed.

As an example that the method of sampling used in the case of both leaf and ground tobacco was satisfactory for small amounts, attention is called to the results of Test Nos. 10-12 and 13-16, Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride. For Test Nos. 10-12, the residues from which the average nicotine content was obtained were: 0.0466, 0.0473, and 0.0458. For Test Nos. 13-16, the residues from which the average nicotine content was obtained were 0.0334, 0.0345, 0.0342, and 0.0332. Comparable results were obtained throughout the investigation. However, if out of four samples, three results obtained were of the same order of magnitude such as listed above, and the other was far out of line, it was discarded. For example, the residues obtained in Tests Nos. 17-19 were: 0.0296, 0.0293, 0.0307. A fourth residue was 0.0257. Obviously, this was out of line with the other three; hence, it was discarded.

Determination of Nicotine Content. Even with a satisfactory method of sampling worked out, consistent results such as those cited above were not obtained until each analytical determination was carried out in a very exact manner. It was found necessary to evaporate the liquid in the distillation flask to dryness. Also, the weight of sample, amount of water and caustic added and time of distillation was the same for each determination. This was done to reduce the possibility of error in an analytical determination that requires a high degree of technique.

Nicotine Content of N. rustica as Compared With Flue-Cured Tobacco. The maximum total nicotine content of the flue-cured tobacco was 1.59%. This was the content of the sample of leaf tobacco. Hence, there had been no loss by vaporization of the nicotine by grinding nor had there been any dilution effect of considerable amounts of leaf stem or stalk.

No analysis of a leaf sample of N. rustica was made. This was not done because the purpose of the investigation was the preparation of the insecticide from the entire tobacco plant. The total nicotine content of the ground N. rustica (entire stalk) was 2.76%. This does not seem to be such a great increase over the nicotine content of the flue cured tobacco. But, the aforementioned facts must be considered. Had the entire plant of flue cured tobacco been available, the total nicotine content most likely would have been lower, and the differences between the two would have been more prominent.

Grinding Tobacco. The original plan of investigation was not carried out in this part of the thesis. Several observations made during the course of the study made it apparent that carrying out the original plan would be a waste of time and no important data could be obtained by such actions.

Changes in Plan of Investigation. Chief departure from the orginal plan was the decision not to attempt any grinding after the tobacco had been treated with HCl gs. It was observed that with the tobacco which had not been treated with HCl, the tendency of the tobacco to stick to the grinding unit and sides of the mill was pronounced. Only when the tobacco was very brittle and the mill and the grinding unit were dry would there be negligible loss due to sticking to the grinding unit or mill. Leaf tobacco which had been treated with HCl gas became so limp and soft, it would have been impossible to grind the tobacco in such a condition.

Another part of the proposed plan that was not carried out was attempting to grind the tobacco in the Lancaster

Countercurrent Batch Mixer. Preliminary tests with this piece of equipment gave proof that such an operation was out of the question. The tobacco was not heavy enough to cause the wheel to turn and produce the grinding action. In the preliminary tests only about 1% of the tobacco being ground passed through a 200 mesh screen.

Grinding Capacity Per Time Interval of the Different Grinding Units. Table XXVI, Results of Screen Analysis of Tobacco After Being Ground in the Rod and Pebble Mill Using 1" Pebbles, 1/2" Pebbles, and Rods as the Grinding Units, gives the results of grinding the tobacco with 1" pebbles, 1/2" pebbles, and rods. In this investigation the grinding capacity of the different units for a given time interval was being sought. In actual practice, no screen analysis of the ground product would be made with the exception of the separation of the product into a size that would meet specifications and that which would not. Also, in practice, all the original charge would not be returned to the mill for additional grinding. Only that part insufficiently ground would be returned. The results of Table XXVI seem to indicate that none of the grinding units were capable of attaining the grinding proficiency desired. All of the tobacco that passed through the 200 mesh screen was retained on the 325 mesh screen. However, the data does not tell the complete story. After the particle size had been reduced so that it would pass through a 200 mesh screen there was an increase in the tendency of the particles to stick together. It did not form hard cakes or balls, but small balls that could be disintegrated with a very slight touch. Also, during one of the preliminary tests, shaking on the riddle overnight resulted in about 10% of the total tobacco ground passing through the 325 mesh screen. These observations indicate that some of the tobacco was reduced in size to the extent that it would have passed through the 325 mesh screen, although this is not indicated by the results in Table XXVI, Results of Screen Analysis of Tobacco After Being Ground in the Rod and Pebble Mill Using 1" Pebbles, 1/2" Pebbles, and Rods as the Grinding Units.

The percentages of Table XXVI are based on the original charge to the mill of 500 gms. of tobacco. The weight of the grinding unit in each case was 10 Kg. For grinding capacity for a given time interval, the rods proved the best. At the end of both the 5 hour and 10 hour grinding intervals the rods gave a higher efficiency of size reduction. At the end of 5 hours the percentage that passed through the 200 mesh screen was 53.60% compared to 47.00% and 46.60% for the 1/2" pebbles and 1" pebbles respectively. At the end of 10 hours, the percentages were 66.60% for rods, 55.70% for 1/2" pebbles and 20.60% for 1" pebbles. This latter figure is not a true indication of the grinding capacity of the 1" pebbles, as indicated by the drop from 46.60% at the end of 5 hours to 20.60% at the end of 10 hours. A late afternoon rain on a hot summer day raised the relative humidity to about 90% at the time the tobacco was on the riddle.

The slight increase in the percentage that passed through the 200 mesh screen after 5 additional hours of grinding would not be enough to justify more grinding after 5 hours. However, the trend of the results indicate that this makes no difference in the rating of the grinding units on the grinding capacity per interval of time basis.

The rods were the best on this basis with the 1/2" pebbles second, and 1" pebbles third. This can be explained by taking into consideration the grinding area per unit volume of grinding unit. Except for irregularities on the surfaces of the rods, the entire length of the rod can serve as grinding surface. In the case of the pebbles, this is not true. One pebble can come into contact with another pebble at only one point which is a very small percentage of the total surface area of the pebble. With the 1" pebbles, the number of contact points, and hence, grinding surface, is smaller per unit volume than the number of contact points of the 1/2" pebbles.

Loss of Nicotine Due to Heat Generated While Grinding. The grinding capacity per time interval is not the only thing that has to be considered. At first this was thought to be true. However, with the completion of Test Nos. 13-16, Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl gas, on the

Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, using tobacco ground with the rods, the total nicotine content dropped from 1.51% as determined in Test Nos. 1-3 to 0.95%. There were several possible explanations. A check on the total nicotine content by analysis of more samples gave comparable results and eliminated the possibility of such great error in Test Nos. 13-16. Therefore, two possibilities remained. The first of these was the dilution effect of the leaf stem due to lower nicotine content of the leaf stem. Upon grinding, the proportion of leaf stem to leaf tissue would be greater than in a sample of leaf tobacco. The other possibility was that the heat generated during grinding would vaporize some of the nicotine.

Table XXVII, Effect of Grinding on the Loss of Nicotine From Tobacco by Vaporization Due to Heat Generated During the Grinding, and Figure 4 show the results of the time of grinding upon the loss of nicotine. This grinding was done with rods. The nicotine content dropped from 1.59% in a sample that was not ground to 1.03% in a sample that was ground 4 hours. The sharp drop between the tobacco that had not been ground and the tobacco that had been ground for 1 hour probably indicates that the dilution effect of the leaf stem is a factor to be considered. The slight decrease between 1 and 2 and 2 and 3 hours grinding indicate that in that interval of time, the amount of heat generated was insufficient to cause very much of the nicotine to volatilize. However, at the end of four hours, the nicotine content showed another considerable decrease and it was evident that the continuous grinding action had generated enough heat to cause serious loss of the nicotine by vaporization.

Tendency of Individual Grinding Unit to Generate Heat. Still another factor to be considered in selecting the best grinding unit is the tendency of the unit to generate the heat by friction, the specific heat, and such other factors as would determine whether the heat would be dissipated quickly or taken up and held by the unit. No specific data was obtained on this, but Test Nos. 27-29, Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, were conducted with tobacco that had been ground for 10 hours with the 1/2" pebbles. The nicotine content in this case was 1.40%, a reduction of only 0.11% from the 1.51% of Test Nos. 1-3. On the other hand Test Nos. 13-16 were performed on tobacco that had been ground for 10 hours with rods. The nicotine content in this case was 0.95%, a reduction of 0.56% from the 1.51% of Test Nos. 1-3.

This is not conclusive evidence, but it indicates that there is a difference. More data would have to be obtained to form any conclusion.

Conversion of Nicotine to Nicotine Hydrochloride. The effect of several variables upon the conversion of nicotine to nicotine hydrochloride by treatment of the tobacco with HCl gas was investigated. These variables were; particle size, time of treatment with HCl gas, and moisture content. The amount of HCl gas per unit of time, the amount of tobacco being treated, the temperature, and the pressure were constant. The amount of HCl gas per hour was the amount generated by the reaction between 100 gms. NaCl and 40 ml. of H_2SO_4. The temperature was always room temperature, and the pressure was only that exerted by the generated HCl gas.

Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, is a compilation of the results of these investigations.

Time of Treatment With HCl Gas. The first thing that is apparent from the results of Table XXV, is that 1 hour of treatment with HCl gas is sufficient to convert all the nicotine that is available for conversion. With the leaf tobacco treatment with HCl gas for 2 and 3 hours gave no better results than treatment with HCl gas for 1 hour. With the ground tobacco, treatment for 2 hours gave no better results than treatment for 1 hour. In fact, in both cases, the percentage of nicotine converted became smaller as the time of treatment was increased. This may have been due to differences in the nicotine content of the samples. However, it seems to be more than a coincidence that this should happen with such regularity. Probably, the continued exposure to the HCl gas results in the formation of another compound other than nicotine hydrochloride. The formation of nicotine hydrochloride is an addition reaction, the HCl molecule being added to the nicotine molecule. Perhaps the continued exposure to the HCl gas caused some breakdown or a substitution reaction to take place. Such an entirely different compound would not undergo the same reaction as nicotine hydrochloride. However, since such a reaction would be defeating the purpose of this investigation, it is desirable to suppress it as much as possible.

Another possibility to be considered is that exposure to the HCl gas for more than 3 hours may result in the hydrolysis of some of the nicotine esters giving more free nicotine.

Effect of Particle Size. The effect of varying the particle size of the tobacco next comes under observation. Only two sizes were used in the investigations; leaf tobacco and that which had passed through a 200 mesh screen. By comparison of the results of Test Nos. 4-12 and 17-22, Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, it seems that a decrease in the particle size results in a slight decrease in the percent of nicotine converted. However, later developments in the investigation gave an explanation of this which clearly demonstrated why this was not the case. A study of Table XXV, Table XXVIII, Results of Steam Distillation of Ordinary Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, and Table XXIX, Results of Steam Distillation of N. rustica Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, and Figures 4 and 5 provide the explanation. It was shown in Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, and Figure 4 that some nicotine was lost by vaporization during grinding. It was not until later that a suggestion resulted in the data given in Tables XXVIII, Results of Steam Distillation of Ordinary Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, and XXIX, and the curves of Figure 5. This indicated that the nicotine in the tobacco exists in two forms- that as free nicotine and that which existed in some combined form. It is reasonable to expect then that the free nicotine would be more susceptible to vaporization by heat than the combined form. Therefore, the proportion of free nicotine to combined form is reduced with a given amount of vaporization by heating. For example, suppose the total nicotine content of tobacco is 1.50% of which 1.00% exists in combined form. This would make one third of the total nicotine content available for conversion to nicotine hydrochloride immediately. Now suppose upon heating, the total nicotine content is reduced to 1.00%. It is in order to expect that a higher proportion of the free nicotine to vaporize than the combined nicotine. Suppose then, three parts of free nicotine were lost for two parts of combined. Then the 1.00% would consist of 0.80% combined nicotine and 0.20% free nicotine. Therefore there would be only one fifth of the total nicotine content available for conversion to nicotine hydrochloride upon treatment with HCl gas.

In working out this explanation, the reason for such low percentages of the total nicotine content being converted to nicotine hydrochloride was explained. Figure 5 indicates that the ratio of free nicotine to combined nicotine was small. Actually then, it is possible that all the free nicotine was being converted.

Effect of Gas Treatment on Grinding Properties of Tobacco. Even if the treatment of the leaf tobacco with HCl gas had resulted in a higher percentage conversion, it would have been no advantage to do so. The tobacco after the treatment with the HCl would have been too soft and limp to be ground.

Effect of Moisture Content of Tobacco. Before the fact was brought out that the nicotine did exist in two forms in the tobacco it was thought that the HCl gas was not coming into contact with the tobacco sufficiently to give a complete reaction. Therefore, it was decided to increase the moisture content of the tobacco with the thought in mind that the HCl gas, being very soluble in water, would be absorbed by the water and thus come into contact with the tobacco. Of course, when it was later brought out about the nicotine existing in two states in the tobacco, the explanation was apparent, but the results of Test Nos. 23-26 of Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, was enough to conclude that an increase in moisture had no effect upon the percentage of nicotine converted to nicotine hydrochloride.

Existence of Nicotine in Two Forms in Tobacco. After varying both time of treatment with HCl gas and the moisture content had failed to produce an increase in the percentage of nicotine converted, the suggestion was made about the nicotine existing as the free nicotine and the combined form. This led to the investigation that resulted in the data of Table XXVIII, Results of Steam Distillation of Ordinary Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, Table XXIX, Results of Steam Distillation of *N. rustica* Tobacco Without Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, and Figure 5, This investigation clearly showed that the proportion of free nicotine to combined nicotine in the flue cured tobacco was small. It also showed that in the case of *N. rustica* the proportion of free nicotine to combined nicotine was greater. And this latter fact also explained the much higher percentage of conversion when the *N. rustica* was treated with the HCl gas.

Effect of NH_3 Treatment Before HCl Gas Treatment. The problem of treating the tobacco in some manner to reduce the combined nicotine to free nicotine before treatment with HCl was then considered. It was decided to treat the tobacco with NH_3 before treatment with HCl to attempt to change some of the combined nicotine to a form so that the treatment with HCl would convert the nicotine to nicotine hydrochloride. It was known that the addition of NaOH in the analytical determination produced a hydrolysis of the combined nicotine to a state such that it was easier to steam distill off. Esters also undergo ammonalysis with NH_3 to form amides. Other types of organic compounds react with NH_3. So it was decided to treat the tobacco with NH_3 before treatment with HCl to see if such reactions would take place and convert the combined nicotine to some form such that subsequent treatment with HCl would produce nicotine hydrochloride. The results of Test Nos. 30-35 of Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, indicate that such a procedure does have an effect upon the percentage of nicotine finally converted and lost by vaporization during the reaction with lime. Considerable investigation along this line is desirable.

No data was obtained as to the result of not using NH_3 before HCl in the treatment of N. rustica. Table XXIX, Results of Steam Distillation of N. rustica Tobacco Without

Caustic to Determine the Proportion of Free Nicotine to Total Nicotine Content, and Figure 5, indicate that the proportion of free nicotine to combined nicotine in the N. rustica was considerably higher than in the flue cured tobacco.

Validity of Method Used to Determine the Amount of Nicotine Converted to Nicotine Hydrochloride. The method used to determine the percentage of the total nicotine content that was converted to nicotine hydrochloride was based on the established practice of adding lime to the insecticide dust as an activator.

Reaction to Liberate Nicotine. The lime reacts with the nicotine salt, such as nicotine sulfate or nicotine hydrochloride, in the presence of moisture, and forms free nicotine and calcium chloride. Then the nicotine is free to volatilize off, making the action of the insecticide that of a fumigant.

A question may be asked as to why the nicotine is converted to nicotine hydrochloride and then changed back to free nicotine. The nicotine in the plant is volatile, but unless it is under the influence of some driving force such as heat, the loss is very slow. If this were not true all the nicotine would be lost from tobacco such as the flue cured tobacco used in this investigation which was at least a year old.

Alternatives in Producing Dust Insecticide. It must be remembered that there are two alternatives in producing a dust insecticide. One is to make the dust of such a nature that the nicotine will be as volatile as possible and the maximum amount be given off in the minimum of time. The other is to make the nicotine less volatile, so that the dust will act more slowly but for a longer time. The first of these is the important one here.

<u>Acclerated Vaporization of Nicotine in Dust</u>. The nicotine freed as a result of the reaction between the nicotine hydrochloride and the lime is in a state that would be more susceptible to vaporization than free nicotine in the original tobacco. The heat of solution of hydrated lime in water is 2.79 kg. cal./gm. mol diluted in 2500 mols of water. The heat of solution of nicotine hydrochloride in water is 6.56 kg. cal./gm. mol at infinite dilution. Since the reaction takes place in the presence of moisture, the resultant heat is sufficient to vaporize the free nicotine freed as a product of the reaction.

<u>Combined Nicotine Liberated by Lime</u>. The question may also be asked about the effect of the reaction of the lime with some of the combined nicotine which results in changing the combined nicotine to the free state just as the addition of caustic liberates the nicotine in the analytical analysis. The undoubetedly takes place and is an advantage. After all, the purpose is to make the dust such that the nicotine will be as volatile as possible and the maximum amount be given off in the minimum of time. But, since the blank (ungassed) samples were mixed with lime before analyzing just as the

gassed samples were, the per cent of nicotine converted was based on the total nicotine content after such a reaction had taken place. The check on Test Nos. 13-16 of Table XXV, Effect of Particle Size, Moisture Content, Treatment With NH_3, and Time of Exposure to HCl Gas, on the Conversion of Nicotine in Tobacco to Nicotine Hydrochloride, that was previously mentioned was with tobacco that had not been mixed with lime. This gave a total nicotine content of 1.01% as compared to 0.95% of Tests Nos. 13-16.

Difference in Actual and Test Conditions. When the dust is being used, the dew provides the water and the air currents carry the vaporized nicotine off. Also, the dust is spread out in a very thin layer and all the free nicotine formed has a chance to be carried off. This was not the case in this investigation. The lime was mixed with the gassed tobacco and put into a crucible. This was allowed to set in a room with no more circulation than the normal ventilation. Also the tobacco and the lime filled the crucible to considerable depth such that perhaps some of the nicotine freed was prevented from being carried off after it was liberated. Therefore, the results obtained might be lower than what could result in actual use.

Recommendations

The following recommendations are made for future work on this subject with the thought in mind of developing a continuous process for the production of a nicotine hydrochloride dust insecticide from the entire N. rustica plant.

Sampling for Carload Lots. The first operation recommended would be a cutting of the tobacco plant, stalk and all, into small pieces before feeding to the grinding mill. This would make it easier for the stalk to be ground and become evenly distributed throughout the ground leaf tissue. Then, after grinding, a sample taken by an approved standard method would contain an equal distribution of the three components of the plant and an overall nicotine content could be obtained. If the stalk were not chopped up into smaller pieces, the leaf tissue and leaf stem would be ground much easier than the stalk and the sample taken would not contain an adequate proportion of the ground stalk.

Method of Determination of Moisture Content. The following method of determination of moisture content is recommended. A sample of 50 gms. of tobacco and 200 cc. of gasoline are put in a flask to which is attached a verticle condenser. Heat is applied, and the moisture in the tobacco vaporizes with the gasoline. The vapors condense and drip down into a tube calibrated in cc. The condensate separates into two immiscible layers. When the volume of water in the calibrated tube has become constant, indicating that all of the moisture in the tobacco has been evaporated off, the volume of water in the 50 gms. of tobacco is read off in cc., from which the per cent moisture is calculated. The nicotine in the tobacco would undoubtedly be driven off by the heat, but it would not be measured as it would in the case of taking the loss in weight[50].

Time of Treatment With HCl Gas. One hour was shown to be sufficient time of treatment with HCl. However, this does not mean that some time under 1 hour would not be sufficient. Therefore, it is recommended that the treatment with HCl for intervals of time less than 1 hour be investigated to determine the minimum time required to convert the available nicotine to nicotine hydrochloride.

Effect of Curing and Drying Process on the Proportion of Free Nicotine to Combined Nicotine in Tobacco. It is recommended that a series of investigations be made to determine the proportion of free nicotine to combined nicotine at different stages of drying of the tobacco from immediately after the tobacco is cut until it has been completely dried or cured.

Treatment With NH_3. It is recommended that further investigations be made to determine the effect of treatment with NH_3 to change the combined nicotine to the free state, and the optimum time of such treatment[51].

<u>Method to Determine Percentage of Nicotine Converted to Nicotine Hydrochloride and Give Data For Material Balance</u>. It is recommended that the following procedure be followed to determine the percentage of nicotine converted to nicotine hydrochloride and at the same time secure data that could be used in making a material balance. The nicotine content is determined in the usual manner. Then samples of a specific amount are weighed out and subjected to treatment with HCl gas. At the end of the gas treatment, the samples are weighed again to determine the increase in weight due to the addition of the HCl molecule to the nicotine molecule. The gassed tobacco and lime are mixed and spread in a thin layer on the porous bottom of a container. Such a container can be made by inserting some porous substance such as "Filtros" into a large glass tube. Enough moisture is added to cause the reaction between the nicotine hydrochloride and lime. A slight current of air from a compressed air source is directed up from the bottom of the glass tube and passed through the porous plate to carry off the nicotine vapors as it is vaporized. After a reasonable time, the mixture of lime and tobacco is weighed again to determine the loss in weight. The loss in weight divided by the weight of nicotine in the original sample would give the percentage of the nicotine converted to nicotine hydrochloride and then lost by vaporization of the free nicotine formed by the reaction between the nicotine hydrochloride and lime. At the same time, valuable data that

could be applied to the problem of making a material balance would be obtained.

Other Grinding Equipment. It is recommended that the use of a hammer mill be investigated in grinding the tobacco. Perhaps the hammers would be just the correct thing for breaking up the plant stalk. Also, the rubbing action of a grinding unit is not present in this type of mill, and consequently the heat generated would be very small.

Limitations

The experimental part of this investigation was conducted under the following limiting conditions.

1. Time of treatment with HCl gas: 1,2, and 3 hours.
2. Size of tobacco- Ground (200 mesh) and unground leaf.
3. Moisture content of tobacco- 15.48%-18.67%; 45.04%.
4. Temperature- 60-90°F.
5. Pressure- Atmospheric.
6. Time of grinding- 5 and 10 hours.
7. Grinding equipment- Patterson Rod and Pebble Mill using 1/2" pebbles, 1" pebbles, and rods as grinding units.
8. Time of treatment with NH_3- 1 hour.
9. Moisture determination- Loss after drying at 140°C. for 24 hours.

V. CONCLUSIONS

On the basis of the results obtained in this investigation the following conclusions are made:

1. Using the survey made by Underwood[28] on the economics of growing flue cured tobacco as the basis, N. rustica could have competed with flue cured tobacco as a commercial crop at a selling price of $0.04 per pound in 1933 on basis of economic crop production.

2. Upon drying at 140°C. for 24 hours the nicotine content of N. rustica was reduced from 2.76% to 0.285%. Upon drying at 105°C. for 24 hours, the nicotine content of N. rustica was reduced from 2.76% to 0.74%.

3. The amount of tobacco that passed through the 200 mesh screen after 5 hours of grinding was 53.60%, 47.00%, and 46.60% of the original charge to the grinding mill using rods, 1/2" pebbles, and 1" pebbles, respectively. At the end of 10 hours grinding, the amount of tobacco that passed through the 200 mesh screen was 66.60% and 55.20% of the original charge to the grinding mill using rods and 1/2" pebbles, respectively.

4. The Lancaster Countercurrent Batch Mixer is unsuitable for grinding tobacco. At the end of a preliminary grinding test, 1% of the original charge to the equipment passed through the 200 mesh screen.

5. The tobacco has to be ground before treatment with HCl gas because treatment of the leaf tobacco causes it to become soft and limp so that grinding could not be accomplished by the use of rods, 1/2" pebbles, or 1" pebbles in the Patterson Rod and Pebble Mill.

6. At the end of 1 hour grinding with rods, the nicotine content was reduced from 1.59% to 1.31%. At the end of 2 hours grinding, the nicotine content was 1.28%. At the end of 3 hours it was 1.24% and at the end of 4 hours grinding, the nicotine content was reduced to 1.03%.

7. The nicotine content of tobacco was reduced from 1.51% to 0.95% by grinding 10 hours with rods. It was reduced to 1.40% by grinding 10 hours with 1/2" pebbles.

8. One hour of treatment with HCl gas was sufficient to convert all the available nicotine to nicotine hydrochloride. Treatment of leaf tobacco with HCl gas and mixing with lime resulted in 15.89%, 14.57%, and 13.91% of the total nicotine content lost by vaporization at the end of 1, 2 and 3 hours of treatment with HCl gas, respectively. Treatment of ground (200 mesh) tobacco with HCl gas and mixing with lime resulted in 11.58% and 10.53% of the total nicotine content lost by vaporization at the end of 1 and 2 hours of treatment with HCl gas, respectively.

9. The maximum percentage of total nicotine content of leaf tobacco lost by vaporization by treatment with only HCl gas and mixing with lime was 11.58%.

10. The percentage of total nicotine content of tobacco, containing 45.04% moisture, lost by vaporization was 10.53%. The percentage of total nicotine content of tobacco containing 18.50% moisture, lost by vaporization was 11.58%.

11. Treatment of the tobacco with NH_3 for 1 hour, subsequent treatment with HCl gas for 1 hour, and mixing with lime gave a loss by vaporization of 22.86% of the total nicotine content. The maximum percentage of total nicotine loss by vaporization by treatment with only HCl gas and mixing with lime was 15.89%.

12. The treatment of N. rustica with NH_3 for 1 hour, subsequent treatment with HCl gas for 1 hour, and mixing with lime resulted in a loss by vaporization of 53.18% of the total nicotine content.

VI. SUMMARY

Nicotine or nicotine compounds have long been used as insecticides. Nicotine insecticides have many uses and are in great demand. However, tobacco, the source of nicotine is such a valuable commodity that only scrap tobacco is used for nicotine production. Consequently, there is a shortage of nicotine for insecticidal purposes.

There are two species of tobacco that are suitable as a source of nicotine. These are N. tabacum which is ordinary tobacco, and N. rustica. In experimental plots, N. rustica has consistantly produced more nicotine per acre than ordinary tobacco. However, the N. rustica has never been grown as a commercial crop.

One purpose of this investigation was to determine the economic feasibility of growing N. rustica as a cash crop as compared to flue cured tobacco by determining the price per pound that a tobacco farmer would have to sell N. rustica to make the same profit he realizes from flue cured tobacco. The other objective was to produce a nicotine hydrochloride dust insecticide from the entire tobacco plant by treatment of the tobacco with HCl gas.

A review of the literature was conducted. This review was divided into three sections: cost of growing flue cured tobacco; cost of growing N. rustica; and preparation of nicotine dust insecticides.

From the review of the literature, the economic feasibility of growing N. rustica was determined.

In the preparation of the nicotine hydrochloride dust insecticide, the effect of several variables upon the percentage of total nicotine converted to nicotine hydrochloride was investigated. These variables were: size of particle, time of treatment with HCl gas, moisture content, and treatment of the tobacco with NH_3 before treatment with HCl gas. The temperature, pressure, and amount of HCl gas used were held constant in that the temperature was room temperature, the amount of HCl gas was that generated by the reaction between 100 gms. of NaCl and 40 ml. of H_2SO_4, and the pressure was that exerted by the generated gas.

The grinding capacity of rods, 1/2" pebbles, and 1" pebbles in a Patterson Rod and Pebble Mill was also investigated. Also during the course of the investigation, the loss of nicotine due to vaporization during grinding, and the proportion of free nicotine to combined nicotine in the tobacco was investigated.

From the results of the investigation the following conclusions were made:

1. In 1933, at a price of $0.04 per pound, N. rustica could have competed with flue cured tobacco as a cash crop using as a basis the economic study of growing flue cured tobacco that was conducted by Underwood[28].

2. In determining the moisture content by heating at 140°C. at 24 hour, the nicotine content was reduced from 2.76% to 0.285%. Heating at 105°C. at 24 hours reduced to nicotine content from 2.76% to 0.74%.

3. The percentage of original charge to the grinding mill that passed through the 200 mesh screen after 5 hours grinding was 53.60%, 47.00%, and 46.60% for rods, 1/2" pebbles, and 1" pebbles respectively. At the end of 10 hours grinding the percentage through the 200 mesh screen was 66.60% and 55.20% for rods and 1/2" pebbles respectively.

4. Approximately 1% of the total charge to the Lancaster Countercurrent Batch Mixer passed through the 200 mesh screen at the end of a preliminary test, indicating that the equipment was unsuitable for grinding tobacco.

5. The tobacco leaf became soft and limp after treatment with HCl gas so that it could not be ground.

6. At the end of 1, 2, 3 and 4 hours of grinding with rods, the nicotine content of tobacco was reduced from 1.59% to 1.31%, 1.28%, 1.24% and 1.03%, respectively.

7. Grinding with rods for 10 hours reduced the nicotine content of tobacco from 1.51% to 0.95%. Grinding with 1/2" pebbles for 10 hours reduced the nicotine content to 1.40%.

8. The treatment of leaf tobacco with HCl gas for 1, 2, and 3 hours and mixing with lime gave a loss of

15.89%, 14.57%, and 13.91%, respectively, of the total nicotine content. Treatment of ground (200 mesh) tobacco for 1 and 2 hours and mixing with lime gave a loss of 11.58% and 10.53%, respectively, of the total nicotine content.

9. Treatment with only HCl gas and mixing with lime gave a loss of 15.89% of the total nicotine content of leaf tobacco. Treatment of the ground (200 mesh) gave a loss of 11.58% of the total nicotine content.

10. Treatment of tobacco containing 45.04% moisture with HCl and mixing with lime gave a loss of 10.58% of the total nicotine content. This was no increase in percentage of total nicotine lost by vaporization in tobacco containing 18.50%.

11. Treatment of the tobacco with NH_3 for 1 hour, subsequent treatment with HCl gas for 1 hour, and mixing with lime gave a loss of 22.86% of the total nicotine content. The maximum percentage of total nicotine content lost by treatment with only HCl gas and mixing with lime was 15.89%.

12. The treatment of <u>N. rustica</u> with NH_3 for 1 hour, subsequent treatment with HCl gas for 1 hour, and mixing with lime gave a loss by vaporization of 53.18% of the total nicotine content.

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VIII. ACKNOWLEDGMENTS

The author wishes to express his sincere appreciation to for his helpful suggestions, constructive criticism, and encouragment during the course of the investigation.

To the author is indebted for several pertinent suggestions and advice.

Graditude is expressed to for growing the N. rustica used in this investigation.

is cited for the time and effort spent in securing the flue cured tobacco used in the majority of the experimental work.

gave assistance in the construction of part of the equipment.