UDY OF THE DISTRIBUTION OF HYDROCYANIC ACID GAS IN GREENHOUSE FUMIGATION

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AGRICULTURAL

Experiment Stations

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NEW JERSEY

AGRICULTURAL EXPERIMENT STATIONS

BULLETIN 355

September 1, 1921

A STUDY OF THE DISTRIBUTION OF HYDROCYANIC ACID GAS IN GREENHOUSE FUMIGATION*

WILLIAM H. WOOD KOMP, M. Sc.

Introduction

One of the most puzzling features in greenhouse fumigation is the variation in amount of cyanide which in practice is used to produce insect-killing strength of hydrocyanic acid gas. This large variation may be accounted for in a number of ways: the influence of temperature and moisture conditions; the influence of condensation of moisture; and the tightness of the greenhouse. Of these factors the last is apparently the most important. The operation of these three factors is such as to render the accurate gradation of dosage in greenhouses impossible, as the amount of cyanide which will produce insect-killing strength of hydrocyanic acid gas in one house will be entirely different in another house of the same cubical contents. This fact has led to the great differences in recommendations for dosage in various publications dealing with the subject of hydrocyanic acid gas fumigation, and is the reason for the empirical methods used by the greenhouse man in actual practice.

To overcome this element of uncertainty, it was proposed to construct a machine by which the exact concentration of HCN† in any part of the greenhouse being fumigated could be accurately determined, and to carry out a series of experiments to determine the part of the house from which the gas should be drawn to give an index of killing strength throughout the house, and also to endeavor by means of minimum dosage experiments

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* This study was undertaken and carried out in the season of 1916-17 under the direction of Dr. Thomas J. Headlee, Entomologist of the New Jersey Agricultural Experiment Stations. The author takes this opportunity of acknowledging the constant encouragement received by him.

† HCN—Hydrocyanic acid gas.

with various forms of aphids to determine the relation of the actual amount of HCN gas present in greenhouse fumigation to the amount necessary to kill the insects.

The first step in solving the problem was the finding of an indicator to denote quantitatively the presence of the gas. The ideal indicator for this purpose is one which, on the addition of a definite concentration of HCN, would indicate clearly by a color change or a precipitation that this concentration had been reached, thus obviating the necessity for a titration of the HCN. Tests were made with many substances, but all failed to reach this ideal. Among the substances used were Fehling's solution, which reacted by changing from deep blue to colorless, but lacked delicacy; the Prussian blue reaction which was sufficiently delicate, but was too complicated for practical use; phenolphthalein, which had to be abandoned because of the interference of the CO_2* in the air, necessitating a qualitative separation of the CO_2 and the HCN, an impracticable operation. The iodine test and the silver nitrate test both necessitate the use of titration methods, and as the $AgNO_3$§ test gave a sharper reaction with the minute quantities of HCN which were absorbed in the tests, it was decided to use it as indicator, titrating the HCN absorbed in a dilute solution of NaOH.‡

Description of the Apparatus

The apparatus in its final form consisted of a set of four 8-liter aspirator jars, which draw air from the greenhouse through a 0.01*N* ‖ solution of NaOH in a test-tube by means of a modified Folin-Denis absorption tube. The gas was drawn from various parts of the greenhouse to the absorption tubes through rubber tubing. The absorption of the HCN in the aspirated air was complete, as shown by testing the air for HCN by passing it through silver nitrate solution after it had passed through the absorber.

Operation of the Apparatus

In operation, a liter° of air, as measured by the amount of water run out of the aspirator jar, was drawn through the rubber tube leading from the greenhouse, to clean the tube preparatory to absorbing the HCN in a second liter of air. This second liter of air was run through the absorber, the test-tube containing the solution then being removed, and another put in its place. Before taking a sample of air, a liter of air was drawn through the rubber tubing each time. The absorptions could be repeated indefinitely by filling the aspirator bottles as soon as emptied. The titration of the absorbed HCN gas in the form of NaCN¶ was carried out in the test tubes of the absorbing apparatus, which were numbered and removed after a liter of

* CO_2—carbon dioxide.
§ $AgNO_3$—silver nitrate.
‡ NaOH—sodium hydroxide.
‖ 0.01*N*—1/100th normal.
° Liter—61.023 cu. in., or 1.05671 qt.
¶ NaCN—sodium cyanide.

air had been passed through each, and were replaced by tubes containing fresh NaOH solution. The titrating solution was 0.001*N* silver nitrate.

Description of the Tests

The general plan of all the tests was to take a cross-section of the house under fumigation, so disposing the tubes from the apparatus that air was drawn from a point just beneath the peak of the roof, midway of the length of the house, from a point about a foot above the middle bench of the house, and from two points, one on each side above the side benches. In some cases a cross-section of half the house was made, in which case the points from which air was drawn were beneath the peak of the roof, a point immediately over the middle bench of the house, another over the next bench toward the outer wall, and the last from a point over the outermost bench nearest the wall.

Five greenhouses were used in the fumigation, situated at the College Farm in New Brunswick. Of these, three were of identical dimensions (No. 1, 2 and 4) 25,368 cubic feet, while one other was a small propagator house (1,371 cubic feet), and the other a medium-sized fern greenhouse (4,866 cubic feet). The tests were run in conjunction with fumigations carried out for the purpose of controlling aphis in the large greenhouses, and in cooperation with another series of experiments conducted under the direction of Prof. M. A. Blake on the burning effect of HCN gas upon garden vegetable plants. All the tests were made after dark. The charge was placed and set in the usual manner for greenhouse fumigation, tall earthenware jars being used, which were placed along the middle line of the greenhouse. The commercial cyanide of sodium made by the Roessler-Hasslacher Chemical Company and known as "Cyanegg" was used in all the fumigations. The formula used was 1-1-3.*

Taking for purposes of comparison all the fumigations with a 5/8-ounce charge, No. 1, 2, 3, 4 and 8, it will be noted that the disappearance of the gas is rapid after a certain maximum concentration has been reached. This concentration, which coincides with the time of complete diffusion of the gas throughout the house is reached at from 4 to 6 minutes after the charge is set off, and suffers steady diminution from that time on until it falls below the killing strength. Although the average concentration, as obtained from the sum of the concentrations obtained at the various points in the house from which air was drawn, is high for the first few minutes, it will be seen that if the concentration at the comb is eliminated, the resulting average is

* Formula 1-1-3—1 oz., by weight, of sodium cyanide; 1 oz., by measure, of concentrated sulfuric acid, sp. gr. 1.84; 3 oz., by measure, of water.

low. This is in effect what occurs in the greenhouse, as the great concentration of gas in the comb can have no effect upon the plants or insects upon the benches.

Peculiar effects are sometimes noted in measuring the concentration of the gas. For instance, in many cases the concentration of gas might be highest in the right-hand side of the house, while in others the center of the house would have the greatest concentration. These differences in concentration may be due to air currents in the house, which carry the gas to various parts of the house, or to layers of air of different temperature and moisture, which might retard the diffusion of the gas. This fact might account for the burning of plants in one

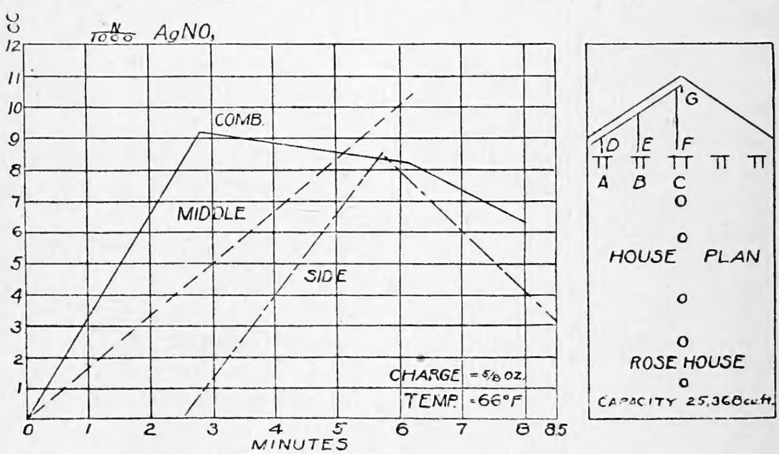

Fig. 1. Graph of Fumigation No. 1

A, outside bench; B, middle bench; C, center bench; D, outside collecting tube; E, middle collecting tube; F, center collecting tube; G, comb collecting tube.

part of the house, while plants of the same kind in another part were unharmed, and also account for the failure to kill the insects in some part of the house.

The distribution of the gas may be even better seen in the figures showing concentrations of gas with higher charges. Thus, in No. 5, 7 and 9 with a 3/4-ounce charge to 1,000 cubic feet the distribution is clearer. Here the uprush of heated gas causes a great massing of the material in the upper part of the house, from whence as it cools it diffuses downward until the concentration becomes approximately equal throughout the house, and from whence it loses in density until it falls below killing strength.

Figures 10 and 11 show very strikingly the variations which may take place in the distribution of gas in the greenhouse.

The fumigations were carried out in the same house, with the same charge—1 ounce of NaCN to 1,000 cubic feet. The result as shown by the graphs are very different. In figure 10 the greatest concentration is found in the comb, within 2 minutes after the charge was liberated. The decrease in concentration is progressive except for the position over the middle bench,

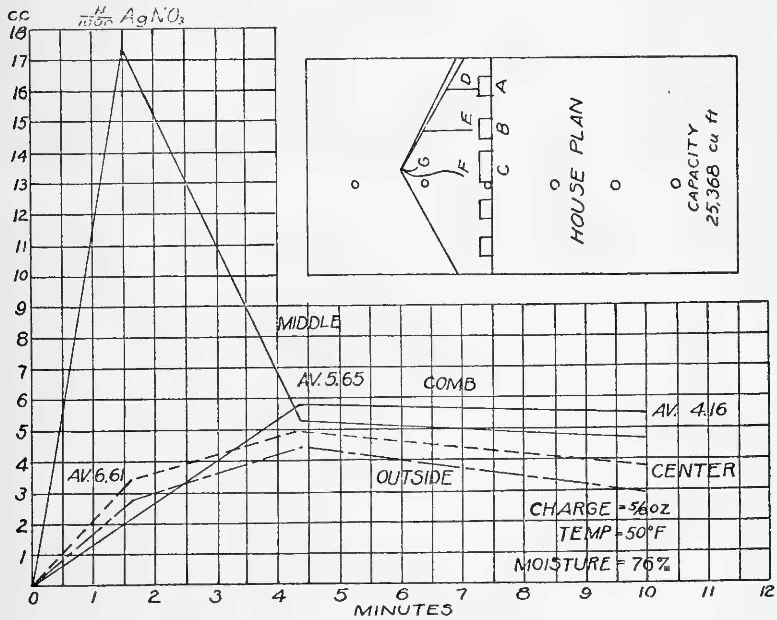

FIG. 2. GRAPH OF FUMIGATION NO. 2

A, outside bench; B, middle bench; E, center bench; D, outside collecting tube; E, middle collecting tube; F, center collecting tube; G, comb collecting tube.

which slowly increases until the end of the fumigation. No. 11, on the other hand, attains maximum concentration in the position over the right greenhouse bench, with only a moderate concentration in the comb, and then decreases rapidly until at the end of the fumigation the concentration throughout the house is approximately equal.

Figures 6, 12 and 15 also show the general course of the fumigation very well, and No. 12 serves to illustrate again the variation which may take place in distribution. In No. 12 and 15 the charge was the same and the house in which the fumigation was done was the same, but in one case the comb had the highest concentration, and in the other the right-hand bench. Figure 6 shows strikingly the reason for failure in some cases to get killing strength of gas in greenhouses under which a trench to accommodate pipes for heating is present. In this fumigation a hole was cut through the concrete floor of the greenhouse and one of the tubes was placed with its opening 1½ feet below the floor, so that it projected down into the trench. The result of the fumigation is seen on the chart. The gas apparently diffused downward and entered the trench, where it attained the highest concentration of any point at the end of the fumigation. This finding would suggest that failure to secure killing with a

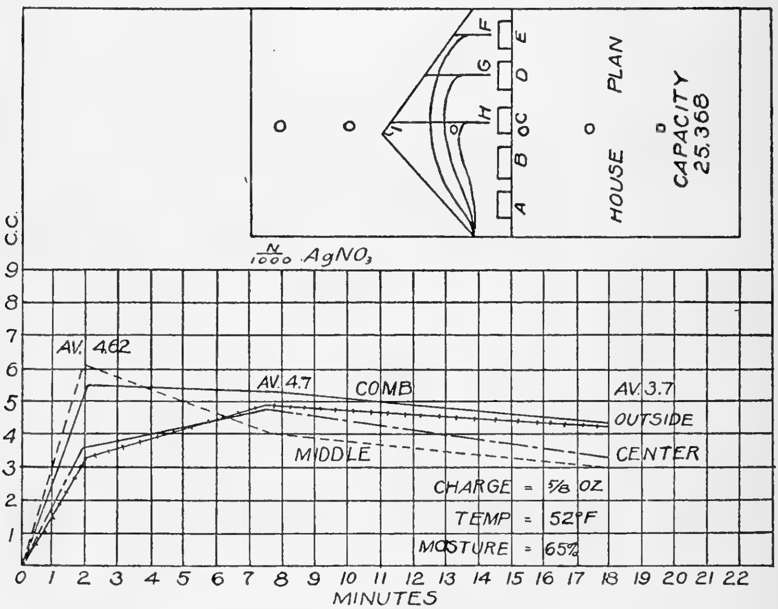

Fig. 3. Graph of Fumigation No. 3

A, outside bench; B, middle bench; C, center bench, D, middle bench; E, outside bench; F, outside collecting tube; G, middle collecting tube; H, center collecting tube; I, comb collecting tube.

charge adequate to get kill in a house of a given cubic capacity may be due to a loss of gas downward into a trench beneath the house. In this case the house was reglazed and tightened before the fumigation so that it was in the best of condition as regards leakage, but nevertheless there was a great loss of gas through the trench beneath.

Figures 13 and 14 showing the results of tests with a 3-ounce and a 6-ounce charge, respectively, show very well the course of the fumigation. The great uprush of heated gas and the subsequent diminution of concentration as the fumigation proceeds is well shown. Likewise, a peculiarity of the house in which these fumigations were made is well brought out. It will be noted in every case in which fumigations are carried out in the so-called fern greenhouse that the concentration of gas tended to be the greatest on the right-hand side of the house. The cause of this peculiar distribution is not known, but it is surmised that temperature may have had something to do with it, as the right-hand side of the house was shaded during the day, and hence would be cooler. The bearing of such a distribution

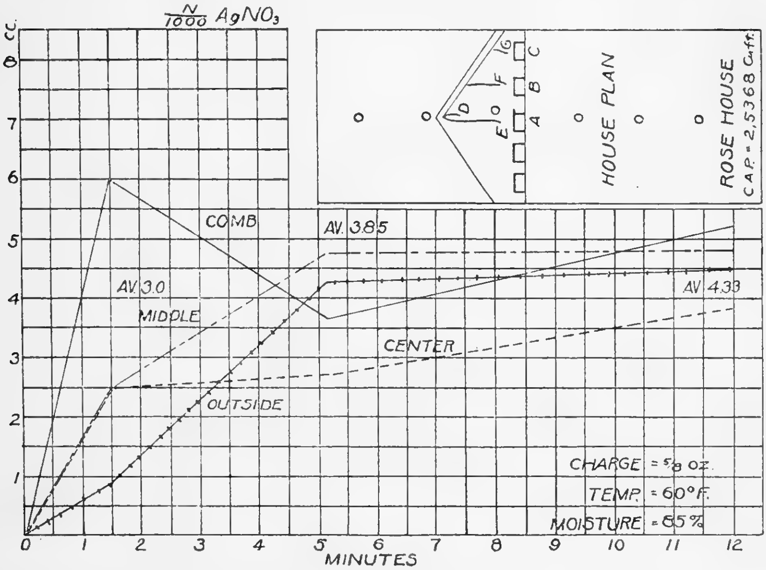

FIG. 4. GRAPH OF FUMIGATION NO. 4

A, center bench; B, middle bench; C, outside bench; D, comb collecting tube; E, center collecting tube; F, middle collecting tube; G, outside collecting tube.

of gas as is shown in this greenhouse upon the burning of the plants and the killing of the insect is obvious.

Distribution in Relation to Dosage

In considering distribution in relation to dosage many peculiar things are noted. In the first place, the great concentration of gas in the comb is not so noticeable with the smaller charges as it is with the larger. A comparison of figures 3 and 4 with figures 13 and 14 will show this plainly. This difference is probably due to the difference in amount of the chemicals taking part in the reaction. More heat is generated with the greater charge and thus the gas would tend to expand and rise more quickly than if less heated.

The same charge used in the same house may give very different results. Taking for comparison figures 12 and 15, both made in the fern house with a charge of 1½ ounces, it is seen that at 1½ minutes after the fumigation started the average concentration throughout the house was 11.5 for No. 15, while for No. 12 it was only 9.06. This discrepancy continues, as 16 minutes later the concentration in No. 15 was 10.26, while in No. 12 it was 7.55. Only at the end of the fumigation was

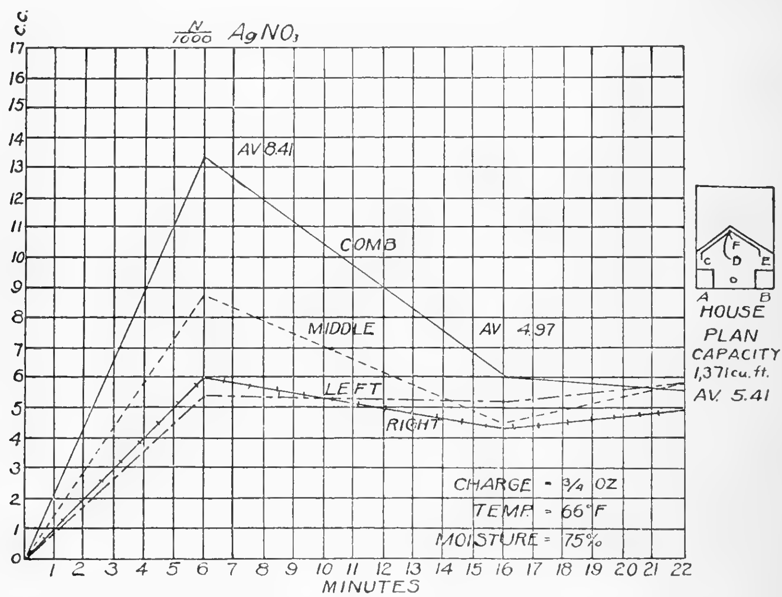

FIG. 5. GRAPH OF FUMIGATION NO. 5

A, left bench; B, right bench; C, left collecting tube; D, middle collecting tube; E, right collecting tube; F, comb collecting tube.

there a close approximation, as at 35 minutes after the fumigation started the concentration in No. 15 was 6.29, while in No. 12 it was 6.89. Likewise, in two fumigations in which double the amount of cyanide was used in one case as was used in the other, the resulting concentrations of gas were not in the same proportion. Figures 13 and 14 show this relation. Using the average concentrations throughout the house in both cases, No. 13 showed an average of 31.5, 1½ minutes after the fumigation started, while No. 14 showed less than this, 27.12 with a charge twice as great. Seventeen minutes later the average for the 3-ounce charge was 19.43 while for the 6-ounce charge it was 23.64; 35 minutes later the average concentration for the whole house was 13.33 for No. 13, and 24.19 for No. 14. This shows clearly that some factor besides the amount of cyanide used will affect the concentration of gas obtained in the greenhouse, and also will tend to explain the contradictory results often obtained in scheduling greenhouses for fumigation.

It seems that the larger the greenhouse the less rapid should be the loss of gas through leakage and consequent diminution in killing strength. This is to be expected, since the volume

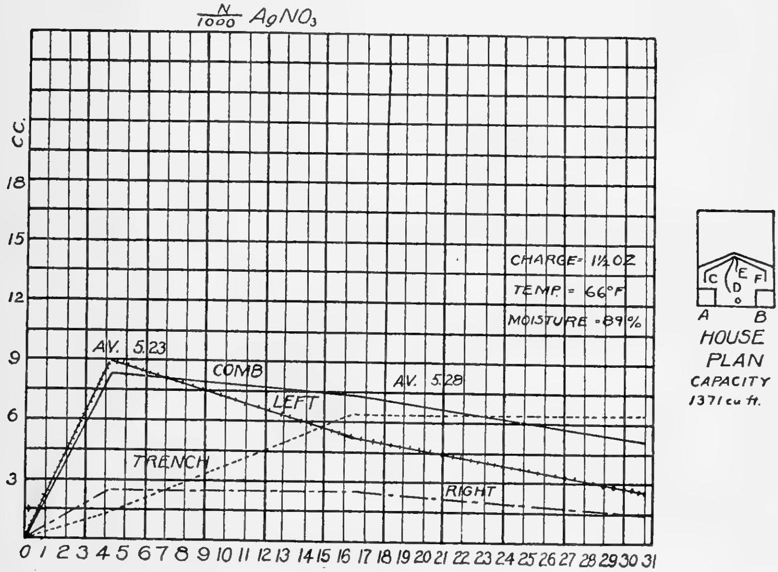

FIG. 6. GRAPH OF FUMIGATION NO. 6

A, left bench; B, right bench; C, left collecting tube; D, center collecting tube; E, comb collecting tube; F, right collecting tube.

of a solid increases as the cube of its diameter, while the surface increases as the square. Thus the larger the greenhouse, the less in proportion to its capacity is its surface, with consequently less loss through leakage. No definite statement can be made as to the correctness of this view, from the data collected. A comparison of two fumigations made with the same relative charge, ¾-ounce per 1,000 cubic feet, in two different houses shows a closely equivalent concentration at approximately equal periods of time after the fumigation started. (See figures 7 and 9.) Judging from these results the balance is in favor of the small house (No. 7), and this is increased if the results of the fumigation charted on No. 5 are considered. In this case the concentrations are uniformly higher than those of No. 7, which was done in the same house, and with the same charge, and likewise higher than those found in No. 9, which was done in the fern greenhouse, which had a capacity of 3½ times the small propagator house in which No. 5 and 7 were performed. Thus it seems that the tightness or looseness of the individual house plays a much more important part in determining the rate of loss and consequent concentration, than does the size of the house. In other words, a small house tightly glazed will retain

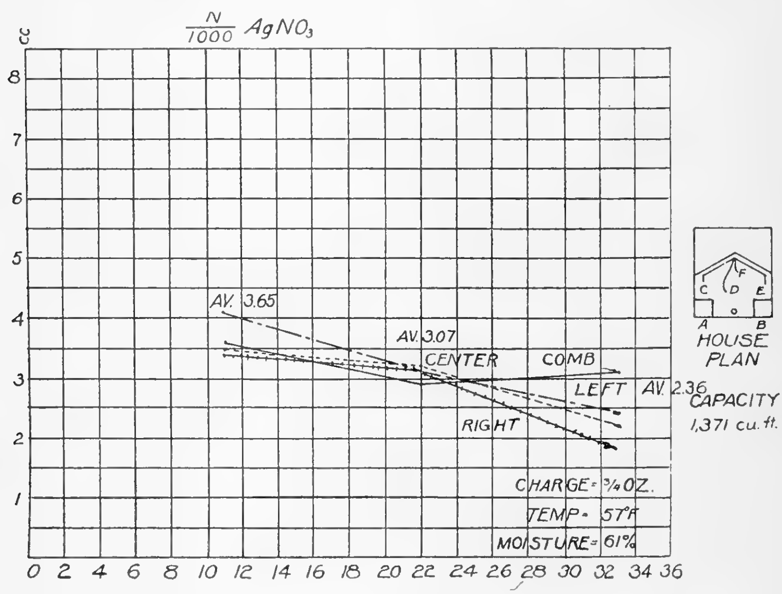

FIG. 7. GRAPH OF FUMIGATION NO. 7

A, left bench; B, right bench; C, left collecting tube; D, center collecting tube; E, right collecting tube; F, comb collecting tube.

a greater portion of the gas generated in a fumigation than a large house which may have less surface in proportion but which is less tight.

Distribution in Relation to Temperature

So far as can be judged from the results of the tests made on the fumigations, temperature has little effect upon the distribution of HCN gas in greenhouse fumigation. Comparing the average concentrations obtained from two fumigations, at the same relative times from the beginning of the fumigation, it is found that with a ⅝-ounce charge in a greenhouse of 25,000 cubic feet capacity, for a difference of 10°F. the concentrations are uniformly lower at the higher temperature. In this case the temperatures were 50° and 60°F. (See figures 2 and 4).

Using the average concentrations for two fumigations with a 1.5-ounce charge, it is found that with a difference of 11°F. the concentrations are lower at the higher temperature. (See figures 12 and 15.) But in two tests made with a ¾-ounce charge and a difference of 9°F. one showed a slight reduction in concentration at the higher temperature, while the other showed a large increase at the higher temperature. While on the whole, the

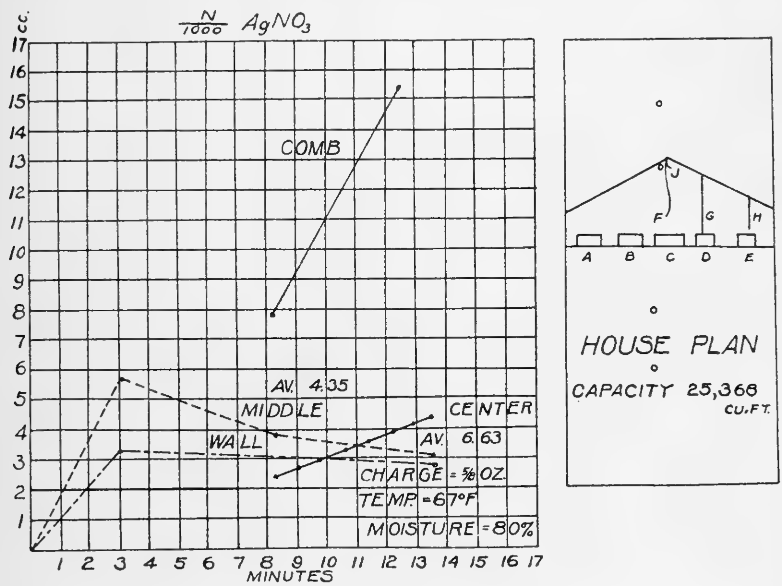

FIG. 8. GRAPH OF FUMIGATION NO. 8

A, wall bench; B, middle bench; C, center bench; D, middle bench; E, wall bench; F, center collecting tube; G, middle collecting tube; H, wall collecting tube; J, comb collecting tube.

greater number of tests show that the concentrations tend to be higher when the temperature is low, the discrepancies noted, together with the small number of tests made, render it impossible to judge accurately of the effect of temperature.

Distribution in Relation to Humidity

Taking the results of two tests made in a greenhouse of 25,000 cubic feet capacity, with approximately equal temperature (50°-52°F.) a difference of 11 per cent relative humidity was accompanied by an increase of concentration with the higher humidity. However, another test in which the time interval was 8 minutes less from the beginning of the fumigation than in the tests described above, the concentration was only slightly higher, with a difference of 20 per cent. relative humidity. Likewise, the results of a comparison of two tests made in the same greenhouse, having a capacity of 3,341 cubic feet, and with the same charge, but with rather a large temperature difference (60°-71°F.) and with a difference in relative humidity of 21 per cent, showed a greater concentration of HCN at the higher humidity and the lower temperature.

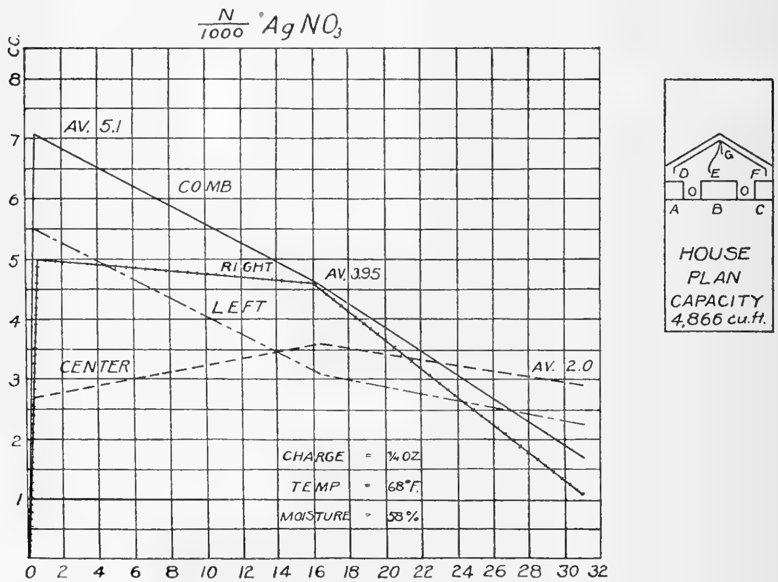

Fig. 9. Graph of Fumigation No. 9

A, left bench; B, center bench; C, right bench; D, left collecting tube; E, center collecting tube; F, right collecting tube; G, comb collecting tube.

Determination of Minimum Dosage for Plant Lice

In order to correlate the findings in regard to concentration of HCN gas in the greenhouse with the actual dosage necessary to kill aphis, a series of experiments was carried out to test for minimum dosage, in relation to temperature and moisture.

Glass flasks, with a capacity of 1 liter, were used in which to liberate the gas. The correct amount of HCN gas was formed in each by diluting a 95.3 per cent solution of liquid HCN with water, so that one drop of water would contain in solution a definite quantity of HCN. By evaporating this drop in the flask by the aid of gentle heat, an atmosphere of HCN of the desired concentration was produced in the flask. The aphids were placed in small glass tubes about 2 inches long and 3/8 inch in internal diameter. Cheesecloth was fastened over the ends of the tubes to prevent their escape. When the desired atmosphere of HCN had been produced, the tubes containing the aphids were dropped into the flasks quickly, and placed in incubators and in the ice-box, so that the insects were subjected to various temperatures.

There were four temperatures tried, 40°F.; room temperature, or about 65°F.; 80°F. and 100°F. Two periods of time also were tried, 15 minutes and 30 minutes.

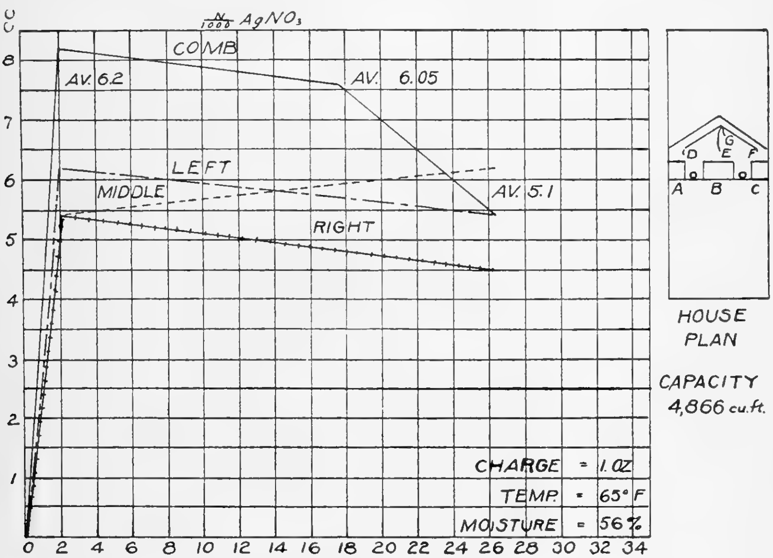

Fig. 10. Graph of Fumigation No. 10

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E, middle collecting tube; F, right collecting tube; G, comb collecting tube.

After the aphids had been removed from the flasks at the end of the desired period of time, the flasks were connected to an absorption apparatus similar to the one used in the greenhouse experiments, and the HCN in the flask absorbed in the same way, and titrated with 0.001*N* silver nitrate. In this way an absolute check could be kept on the amount of HCN in the atmosphere in the flasks.

Using this method, it was found that as would be expected, the effectiveness of the fumigation was much increased by an increase of temperature. The results of a number of tests for minimum dosage are given in table 1.

From this table it will be seen that the minimum dose for plant lice experimented with **(Macrosiphum rosae)**, lies in the neighborhood of 0.00015 to 0.0002 gm. HCN per liter, depending upon the temperature. Checks at 40°F. and at 100°F. were run in all the experiments with minimum dosage, and in no case was any mortality observed.

TABLE I

Effect of Different Dosages of Hydrocyanic Acid Gas on Plant Lice

Amount of HCN per liter	Temperature	Time	Result
gm.	°F.	Minutes	
0.0004	40	15	All Dead
	60	15	All Dead
	80	15	All Dead
	100	15	All Dead
0.0004	40	30	All Dead
	60	30	All Dead
	80	30	All Dead
	100	30	All Dead
0.0002	40	15	All Dead
	60	15	All Dead
	80	15	All Dead
	100	15	All Dead
0.0002	40	30	All Dead
	60	30	All Dead
	80	30	All Dead
	100	30	All Dead
0.00015	40	15	All Alive
	60	15	All Alive
	80	15	50% Dead
	100	15	All Dead
0.00015	40	30	50% Dead
	60	30	50% Dead
	80	30	All Dead
	100	30	All Dead
0.0001	40	15	All Alive
	60	15	All Alive
	80	15	All Alive
	100	15	All Alive
0.0001	40	30	All Alive
	60	30	All Alive
	80	30	All Alive
	100	30	All Alive

The failure to kill the aphids at a low temperature will explain the rather high percentages of HCN gas necessary to produce killing effects in greenhouses, where as a matter of safety to the plants during fumigation the temperature is kept low. But it will not explain the great difference in the amount of gas necessary to kill in these minimum dosage experiments, and the amount found necessary to produce killing in greenhouses.

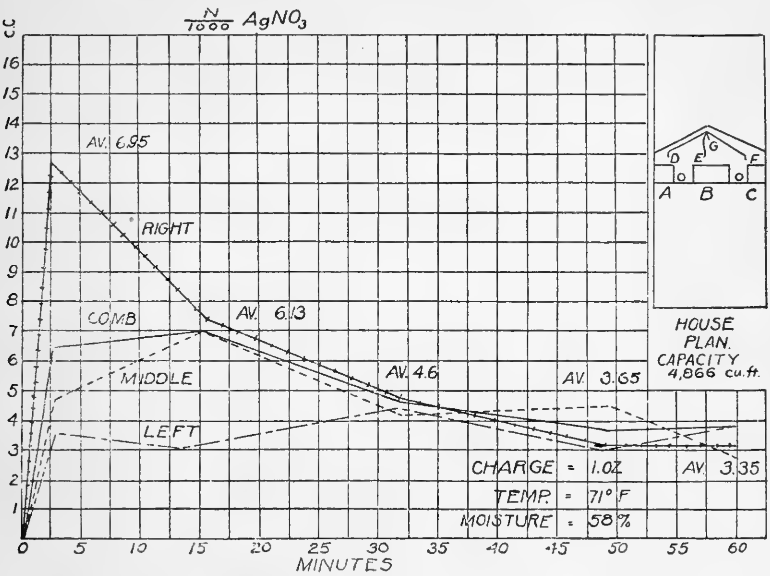

FIG. 11. GRAPH OF FUMIGATION NO. 11

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E. middle collecting tube; F, right collecting tube; G, comb collecting tube.

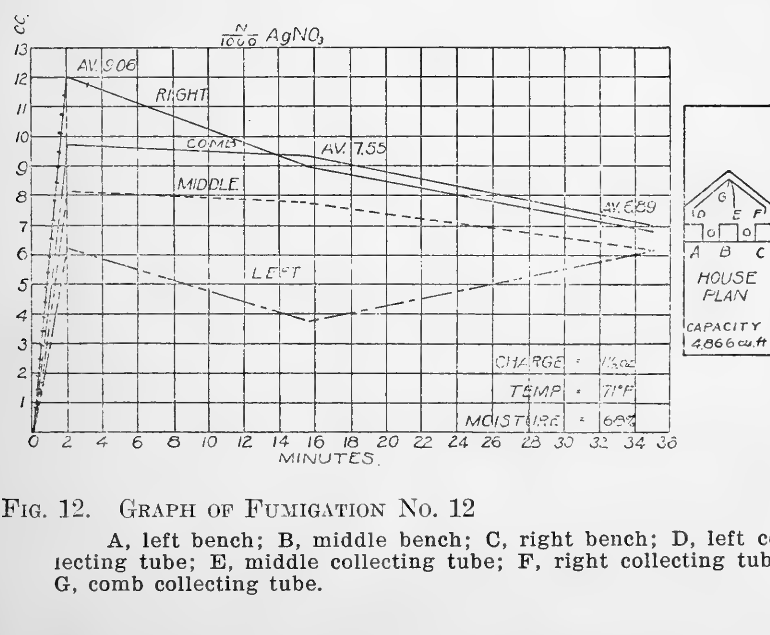

FIG. 12. GRAPH OF FUMIGATION NO. 12

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E, middle collecting tube; F, right collecting tube; G, comb collecting tube.

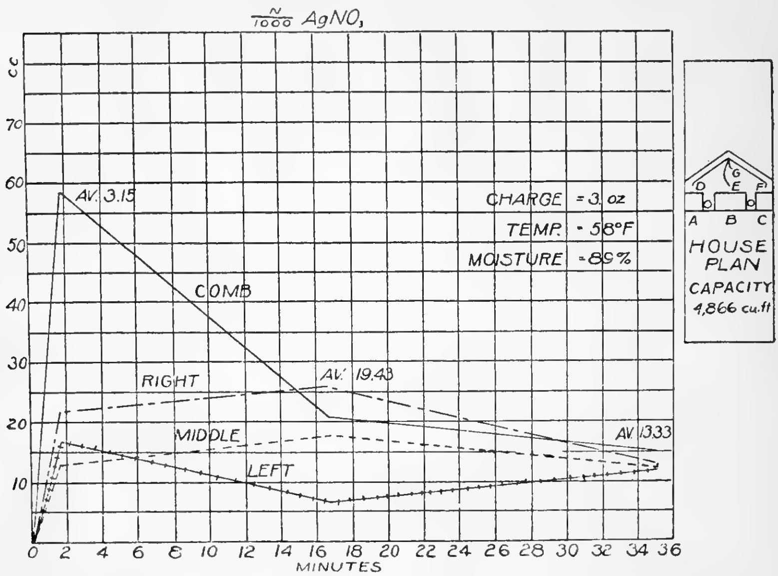

FIG. 13. GRAPH OF FUMIGATION NO. 13

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E, middle collecting tube; F, right collecting tube; G, comb collecting tube.

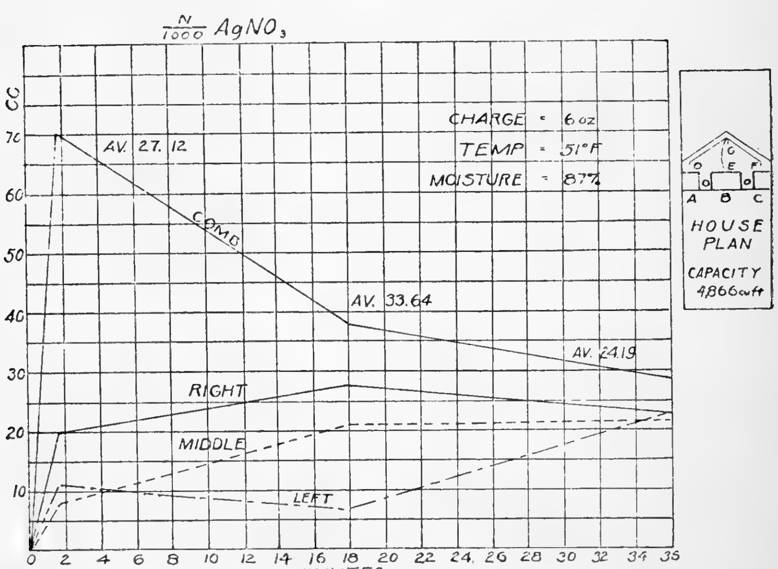

FIG. 14. GRAPH OF FUMIGATION NO. 14

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E, middle collecting tube; F, right collecting tube; G, comb collecting tube.

Our experiments show that in greenhouses under fumigation for rose-aphis, at a rate of 5/8-ounce to 1,000 cubic feet, a concentration of 0.000278 gm. HCN per liter is often reached and frequently exceeded over the greenhouse benches. Contrasting this with the amount of gas found necessary to kill in the controlled minimum dosage experiments, it is found that at least twice as much gas is present as should be necessary to effect a kill. If the total amount of HCN gas were available from the sodium cyanide at the rate of 5/8-ounce per 1,000 cubic feet, a concentration of 0.000312 gm. per liter would be obtained. Such complete evolution of gas is never obtained under ordinary conditions, 1 or 2 per cent or more of gas being lost in the solution or decomposed by the heat of the reaction. Nevertheless, a very

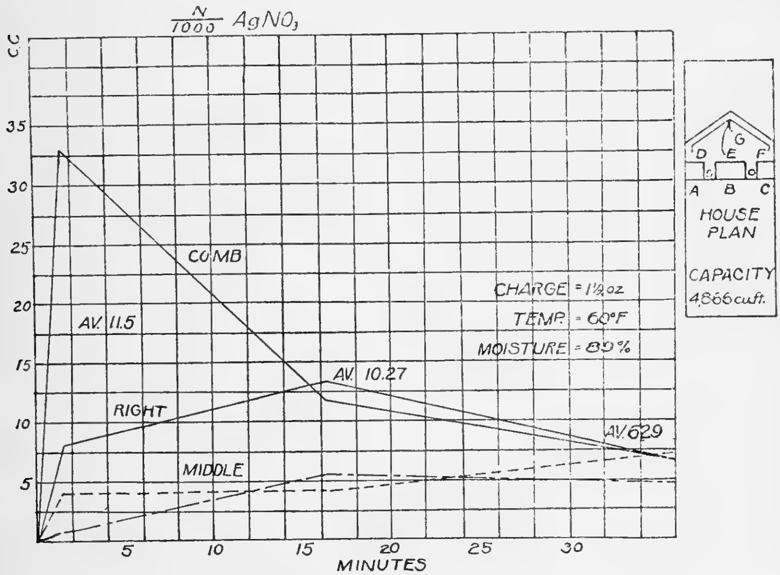

FIG. 15. GRAPH OF FUMIGATION NO. 15

A, left bench; B, middle bench; C, right bench; D, left collecting tube; E, middle collecting tube; F, right collecting tube; G, comb collecting tube.

large proportion, 90 per cent or over, is actually present and is available for fumigation. The discrepancy between the amount observed to be sufficient to kill in the minimum dosage experiments and that necessary to kill in greenhouses seems to be due to the manner of distribution of the gas, as well as the loss of gas due to leakage. Thus almost the entire amount of gas generated is ineffective during the first few minutes of the fumigation, as the sharp uprush of gas carries most of it to the comb of the house, where of course it is not available for use against insects. Later as it diffuses downward it reaches the plants on the benches and begins to affect the insects on the plants, it is partially absorbed by condensed moisture on the glazing and on the leaves of the plants, and also it diffuses out through the small cracks in the glazing, around the doors, and in the ventilators. The interval between the liberation of the gas by the addition of the cyanide to the acid and water, and the time at which complete diffusion has taken place is rather large when compared with the total time of fumigation for aphis in greenhouses, which may be only 15 to 20 minutes. Any method of liberating the HCN in a more diffused manner, so that the gas would reach all parts of the house at approximately the same concentration at the same time, would tend to lessen the danger of burning the plants, shorten the time of fumigation, and give more complete killing results upon the insects.

The fact that insects were killed in much less time, and at much less concentrations of HCN when they were enclosed in tight flasks where the concentration was approximately equal throughout the flask, and in which they were not subjected to air-currents which would tend to vary the concentration of gas, shows that a comparatively quiet atmosphere in which the concentration does not vary, is more effective in which to conduct a fumigation. This line opens up the thought that greenhouses which are regularly fumigated might be piped, as for a sprinkler system, so that definite charges of HCN might be led into them from a generator. This method would have the advantage of simplicity, and would give a much more even distribution of gas than is possible with the use of jars, as is the usual method. However, a more even distribution of gas could be attained by the use of a larger number of jars, scattered through the house, each provided with a deflector of some sort which would prevent the sudden uprush of heated gas, and allow a more gradual diffusion of it through the house.

Any method which will allow for a more even distribution of gas in the house will result in a saving in the cost of fumigation, as less cyanide would be needed to get the same results, because the large amount of gas, concentrated in the comb of the house, and useless there against the insects fumigated, would be eliminated. The efficiency of the fumigation would thereby be increased, the time shortened, and the danger to the plants materially lessened.