

## Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.



# UNITED STATES DEPARTMENT OF AGRICULTURE



DEPARTMENT BULLETIN No. 1312



Washington, D. C.

January 27, 1925

## LOSS OF NICOTINE FROM NICOTINE DUSTS DURING STORAGE

By C. C. McDONNELL, *Chief*, and H. D. YOUNG, *Assistant Chemist, Insecticide and Fungicide Laboratory, Miscellaneous Division, Bureau of Chemistry*

### CONTENTS

	Page		Page
Purpose of investigation.....	1	Volatility of nicotine sulphate solution and	
Review of literature.....	2	free nicotine solution.....	13
Outline of experiments.....	2	Summary.....	14
Experimental results.....	3	Literature cited.....	14

### PURPOSE OF INVESTIGATION

"Nicotine dusts," since their introduction in 1917 by Smith of California (6)<sup>1</sup>, have come into extensive use for the control of certain insect pests. Campbell (1) estimated that about 2,000 tons of such preparations were produced in 1923. These dusts consist of finely powdered fuller's earth, kaolin, hydrated lime, dolomitic lime, calcium carbonate, gypsum, sulphur or other absorbent material, with which nicotine sulphate or free nicotine solution is incorporated. One serious objection to such products from the standpoint of the consumer, as well as from that of the manufacturer, is that they are not stable and must be used comparatively soon after they have been made. The nicotine is lost more or less rapidly, depending principally upon the form of nicotine used, the chemical and physical nature of the absorbent, and the type of container.

Under the Federal insecticide and fungicide act, all such preparations entering interstate commerce must be labeled to show the percentage of nicotine present and the total percentage of inert ingredients, or, in lieu of this, the name and percentage of each inert ingredient present. The first form of statement is the more feasible and the one generally adopted. Owing to the loss of nicotine from these preparations as now manufactured and packed, it is difficult for the manufacturer to market a product that will remain stable. The consumer, therefore, may unknowingly use a product that has lost its strength to such an extent as to be worthless.

The investigation here reported was undertaken in January, 1922 (Annual Report of the Insecticide and Fungicide Board for 1922, p. 6), for the purpose of obtaining definite information on these

<sup>1</sup> Italic numbers in parentheses refer to the Literature Cited, page 14.

points and to devise methods of preparation and packing which would make possible a more uniform product. The relation of killing power to the rate and extent of the liberation of nicotine when applied to the plants, a very important point, is not considered here.

### REVIEW OF LITERATURE

Smith (6) describes the introduction of nicotine dusts and gives the history of their early manufacture. He mentions the reaction of lime with nicotine sulphate to form free nicotine. In his discussion of fillers and carriers, he comments on kaolin (which he ranks very high), hydrated lime, quicklime, talc, sulphur, tobacco dust, gypsum, kieselguhr, and lime carbonate. His statement that "lime carbonate" has no effect on nicotine sulphate does not agree with the findings of others. De Ong (2) suggests that the results obtained by Smith may have been due to the kind of carbonate of lime he used.

Headlee and Rudolfs (3) and Rudolfs (5) published data on the liberation of nicotine from various types of dusts. Most of these results are included in their later publication (4), which gives also a report of their thorough studies on the volatilization of nicotine, both under field conditions and in the laboratory, by passing air under definitely controlled conditions through the nicotine dusts, and on the relation of the rate of volatilization to insect toxicity.

De Ong (2) presents data showing the difference in toxicity of non-volatile nicotine sulphate and volatile nicotine. He states that the toxicity of a dust varies in proportion to the change from the salt to the free alkaloid.

Thatcher and Streeter (7) conducted a thorough study on the loss of nicotine from nicotine dusts. Their investigation included work on the loss of nicotine from nicotine sulphate dusts during storage very similar to that done in the Bureau of Chemistry. Although the two studies were carried on at the same time, each was conducted entirely independently of the other. Thatcher and Streeter divide fillers or carriers for nicotine sulphate into three groups: (a) Colloidal substances, like kieselguhr and kaolin, that tend to prevent volatilization; (b) crystalline substances, such as sulphate and gypsum, that are inert; and (c) all the common hydroxides and carbonates which are "active;" that is, change the nicotine sulphate into the more volatile free nicotine. In studying the effect of storage on nicotine dusts, they found significant losses from the calcium carbonate and calcium hydroxide dusts, especially the carbonate dust, even in sealed containers. They suggested that this might be due to some chemical change in the nicotine.

Campbell (1) presents a historical review of nicotine dusting, which includes a complete bibliography.

### OUTLINE OF EXPERIMENTS

In preparing the mixtures used in the investigation here reported, two commercial nicotine solutions, a 40 per cent solution of nicotine sulphate and a 40 per cent solution of free nicotine, were used. Kieselguhr, kaolin, talcum, calcium hydroxide, calcium carbonate, and plaster of Paris were the carriers or absorbents. Grades of the usual commercial purity and degree of fineness were used, so that the results might be comparable with those obtained in commercial practice.

The dust mixtures were made up as follows: A quantity of the nicotine solution sufficient to produce a dust of the highest nicotine content employed in the tests was added to the carrier. After putting this mixture through a suitable mixing and sifting machine, a portion was reserved for the highest strength, and two other portions were diluted with the carrier and again put through the sifter and mixer. The mixtures were then analyzed and packed in the following types of containers: Pasteboard boxes made of two thicknesses of cardboard, with a slip cover of the same material; heavy cylindrical paraffined boxes, having close-fitting covers of the same material, a type much used commercially for semiliquid materials; glass jars with screw top and rubber ring; and small canvas bags. They were stored at room temperature.

The samples were analyzed at definite intervals covering a period of a year or longer. In the earlier part of the work all analyses were made by the official silicotungstic-acid method (Official and Tentative Methods of Analysis, Association of Official Agricultural Chemists (1920), p. 65). As the number of samples increased and the time involved in the steam distillations became great, experiments were made to determine whether or not the nicotine could be washed out of the dusts and precipitated directly without undue loss in accuracy. It was found that by direct extraction with water practically the same results were obtained as by steam distillation. This procedure therefore was adopted for the later work.

### EXPERIMENTAL RESULTS

The results of the analyses of the nicotine dusts are given in Tables 1 to 10, inclusive.

Three strengths of nicotine sulphate-kieselguhr dust (Table 1) were used in the first series reported.

TABLE 1.—*Nicotine in nicotine sulphate-kieselguhr dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Mar. 18.....	7.17	7.17	7.17	7.17	5.12	5.12	5.12	5.12	2.24	2.24	2.24	2.24
Apr. 3.....	7.29	7.56	7.54	-----	5.26	5.23	5.09	-----	2.25	2.28	2.26	2.25
May 3.....	7.49	7.50	7.13	7.14	5.20	5.28	5.14	5.05	2.28	2.34	-----	-----
June 3.....	7.35	7.40	7.09	7.32	5.20	5.39	5.11	5.30	2.30	2.31	2.24	2.28
Sept. 3.....	-----	-----	-----	-----	-----	5.01	5.01	5.14	2.25	2.38	2.25	2.34
Nov. 3.....	6.84	-----	6.80	6.64	4.84	4.86	4.76	4.93	2.08	2.10	2.14	2.23
1923												
Nov. 3.....	5.90	-----	6.18	5.78	3.89	4.03	4.46	3.89	1.85	1.92	1.90	1.74

The results in Table 1 show that the rate of liberation of nicotine from kieselguhr is slow. No loss had occurred at the end of three months.<sup>2</sup> Even in the canvas bag, the change in nicotine content

<sup>2</sup> The higher strengths show, in some cases, an increase in the nicotine content after three months, which may be accounted for, partially at least, by the loss of moisture from the samples and consequent relative concentration of nicotine in the samples.

in the strongest mixtures was only from 7.17 to 5.78 per cent after 20 months. In other words, 80.6 per cent of the nicotine originally present remained at the end of 20 months, a loss of only 1 per cent per month.

Two strengths only were made up with kaolin, because it is such a poor absorbent that even the 5 per cent strength was too damp for use as a dust.

TABLE 2.—*Nicotine in nicotine sulphate-kaolin dust*

Date of analysis	Strength 2				Strength 3			
	Paste-board box	Par-affined box	Glass jar	Canvas bag	Paste-board box	Par-affined box	Glass jar	Canvas bag
1922	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
April 3.....	5.25	5.25	5.25	5.25	2.49	2.49	2.49	2.49
May 3.....	4.92	5.17	5.23	5.02	2.48	2.50	2.50	2.50
June 3.....	5.03	5.08	5.07	5.06	2.44	2.46	2.45	2.47
September 3.....	4.92	5.00	5.08	4.93	2.33	2.43	2.38	2.37
December 3.....	4.80	4.83	-----	4.81	2.31	2.39	2.40	2.39
1923								
November 3.....	4.71	4.75	4.92	4.57	2.11	2.17	2.23	2.03

The kaolin dusts also showed practically no loss after storage for two months. (Table 2.) At the end of 19 months the loss from the stronger dust packed in the canvas bag was 13 per cent of the total quantity originally present; the loss from the same dust packed in the glass jar was 6 per cent.

As talcum proved to be the poorest absorbent of the materials tested, a dust of the lowest concentration only was prepared with it.

TABLE 3.—*Nicotine in nicotine sulphate-talcum dust*

Date of analysis	Strength 3		
	Paste-board box	Par-affined box	Glass jar
1922	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Feb. 20.....	1.79	1.79	1.79
Mar. 20.....	1.71	1.67	1.79
Apr. 20.....	1.65	1.67	1.78
June 20.....	1.41	1.47	1.61
Sept. 20.....	1.18	1.34	1.64
1923			
Feb. 20.....	1.10	1.16	1.54
July 20.....	1.00	1.10	1.45
Dec. 20.....	.94	1.00	1.33

Talcum dust lost nicotine more rapidly than either the kieselguhr or kaolin dust. (Table 3.) The maximum loss was 47 per cent of the quantity originally present after 22 months.

As calcium hydroxide, calcium carbonate, and plaster of Paris were very active in liberating nicotine, they will be considered together. (Tables 4, 5, and 6.)

TABLE 4.—*Nicotine in nicotine sulphate-calcium hydroxide dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Apr. 12.....	7.04	7.04	7.04	7.04	5.04	5.04	5.04	5.04	2.41	2.41	2.41	2.41
May 12.....	6.58	7.20	7.22	6.50	4.95	5.32	5.24	4.86	2.34	2.36	2.52	1.88
June 12.....	6.24	6.92	7.24	6.00	4.40	4.82	5.18	4.56	2.09	2.11	2.48	1.91
Sept. 12.....	5.08	6.03	7.07	3.06	3.76	4.13	5.32	2.99	1.54	1.81	2.50	1.14
1923												
Feb. 12.....	3.69	-----	6.75	2.76	2.43	3.80	5.42	1.73	1.13	1.55	2.45	.81
Dec. 12.....	1.78	3.26	6.40	1.81	1.37	2.60	4.93	1.06	.81	1.00	1.99	.67

TABLE 5.—*Nicotine in nicotine sulphate-calcium carbonate dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Apr. 8.....	7.22	7.22	7.22	7.22	4.99	4.99	4.99	4.99	2.33	2.33	2.33	2.33
June 8.....	5.86	6.36	6.53	4.02	3.46	4.06	4.56	3.22	1.31	1.61	1.98	1.08
Sept. 10.....	4.32	5.54	6.31	2.45	2.22	3.15	4.26	1.42	.82	1.01	1.56	.60
1923												
Feb. 8.....	2.08	-----	6.49	1.90	1.90	3.19	4.64	1.29	.97	1.16	1.79	.96
Nov. 8.....	1.85	-----	6.27	1.02	1.16	1.66	4.08	.75	.70	.75	1.63	.52

TABLE 6.—*Nicotine in nicotine sulphate-plaster of Paris dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag	Paste-board box	Parafined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Apr. 16.....	6.70	6.70	6.70	6.70	4.65	4.65	4.65	4.65	2.02	2.02	2.02	2.02
May 16.....	6.12	6.50	6.31	5.98	4.22	4.28	-----	4.08	1.66	1.82	1.82	1.55
June 16.....	5.88	6.09	6.40	5.03	3.70	4.08	4.38	-----	1.22	1.56	1.85	.98
Sept. 16.....	5.18	5.56	6.78	4.88	2.84	3.05	4.57	2.51	.69	1.23	1.81	.59
1923												
Feb. 16.....	3.77	5.88	6.49	4.08	2.17	3.08	4.41	2.12	.63	.76	1.67	.42
Nov. 16.....	3.67	-----	6.30	3.78	2.24	2.31	4.11	1.99	.49	.56	1.36	.39

Both calcium hydroxide and calcium carbonate react with nicotine sulphate, freeing the alkaloid, which is volatile. Analysis of the plaster of Paris showed that it was alkaline enough (equivalent to 1.5 per cent calcium carbonate) to react with the nicotine sulphate in the weaker dusts, but not alkaline enough to react completely with the nicotine sulphate in the stronger dusts. For this reason, dusts of this material in different strengths do not behave alike. The strongest, containing originally 6.7 per cent nicotine, lost, from the canvas bag, 44 per cent of its nicotine content after 19 months, while the sample with 2.02 per cent nicotine lost during the same period over 80 per cent of its nicotine. The maximum loss from

the calcium hydroxide dust was 80 per cent and the minimum loss from the dust of highest nicotine content, packed in the glass container, was less than 10 per cent during storage for 20 months. The calcium carbonate dust lost more than any of the other nicotine sulphate dusts, the loss ranging from a maximum of 86 per cent to a minimum of 13 per cent during the 19-month storage period.

Tables 7, 8, 9, and 10 give the results of analyses of dusts prepared and packed in the same manner as the dusts already considered, except that free nicotine solution was substituted for nicotine sulphate solution. These results cover a maximum period of storage of 12 months except in the case of free nicotine-kieselguhr dust, which ran for 19 months. Kaolin and talcum, being poor absorbents, were not used.

TABLE 7.—*Nicotine in free nicotine-kieselguhr dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
May 28.....	8.06	8.06	8.06	8.06	5.56	5.56	5.56	5.56	3.94	3.94	3.94	3.94
June 28.....	7.57	7.51	7.99	6.90	5.05	5.30	5.41	5.30	2.81	3.44	3.88	3.17
Nov. 28.....	4.54	5.30	7.20	3.73	4.00	4.52	5.00	3.78	2.26	2.36	3.72	1.77
1923												
Feb. 15.....	3.86	4.65	6.99	3.42	3.39	4.07	4.72	3.26	1.96	2.28	3.66	1.42
Dec. 15.....	2.87	3.71	6.39	2.66	2.83	3.33	4.18	2.54	1.34	1.49	3.09	.65

TABLE 8.—*Nicotine in free nicotine-calcium carbonate dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Nov. 20.....	7.43	7.43	7.43	7.43	4.70	4.70	4.70	4.70	2.05	2.05	2.05	2.05
1923												
Jan. 20.....	6.50	6.55	7.00	5.04	3.40	3.42	4.48	2.58	1.55	1.51	1.95	1.35
Feb. 20.....	4.27	4.14	6.87	4.09	2.51	2.82	4.39	1.99	1.23	1.30	1.83	1.04
May 20.....	4.00		6.55	3.11	2.02	2.06	4.02	1.55	1.05	1.08	1.70	.95
Nov. 20.....	2.97	3.54	6.26	1.94	1.40	1.58	3.56	.98	.64	.43	1.55	.51

TABLE 9.—*Nicotine in free nicotine-plaster of Paris dust*

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag
1922	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Dec. 8.....	6.18	6.18	6.18	6.18	3.80	3.80	3.80	3.80	1.62	1.62	1.62	1.62
1923												
Feb. 8.....	5.49	6.05	6.12	3.52	3.65	3.74	3.77	3.01	1.38	1.50	1.51	.68
Apr. 8.....	4.50	5.18	6.04	3.11	2.80	3.20	3.70	2.32	1.09	1.11	1.38	.74
July 8.....	3.08	4.41	5.91	2.18	1.70	2.48	3.51	1.33	.68	.73	1.26	.51
Dec. 8.....	1.72	3.55	5.38	.35	.78	1.84	3.12	.14	.22	.22	1.03	.13



TABLE 10.—Nicotine in free nicotine-calcium hydroxide dust

Date of analysis	Strength 1				Strength 2				Strength 3			
	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag	Paste-board box	Paraf-fined box	Glass jar	Can-vas bag
1922												
Dec. 28.....	Per ct. 7.80	Per ct. 7.80	Per ct. 7.80	Per ct. 7.80	Per ct. 5.46	Per ct. 5.46	Per ct. 5.46	Per ct. 5.46	Per ct. 3.31	Per ct. 3.31	Per ct. 3.31	Per ct. 3.31
1923												
Feb. 28.....	6.39	7.45	7.75	3.31	4.59	5.30	5.45	2.85	2.77	3.10	3.24	2.29
Apr. 28.....	5.42	6.43	7.60	2.83	3.80	4.48	5.20	2.40	2.41	2.88	3.10	1.80
Sept. 28.....	3.10	5.23	7.47	2.11	2.04	3.63	4.92	1.53	1.23	2.23	2.95	1.10
Dec. 30.....	2.00	4.52	7.18	1.79	1.26	3.24	4.74	.72	.68	1.59	2.72	.92

The results obtained by calculating the percentage of nicotine lost from each sample and averaging for the three strengths are shown in Figures 1 to 10. The figures, therefore, show the percentage of nicotine lost from the samples, while the tables show the percentage of nicotine in the samples at the time of analysis.

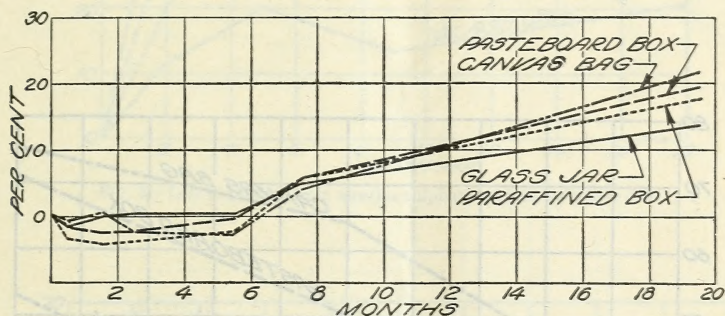


FIG. 1.—Loss of nicotine from nicotine sulphate-kieselguhr dust

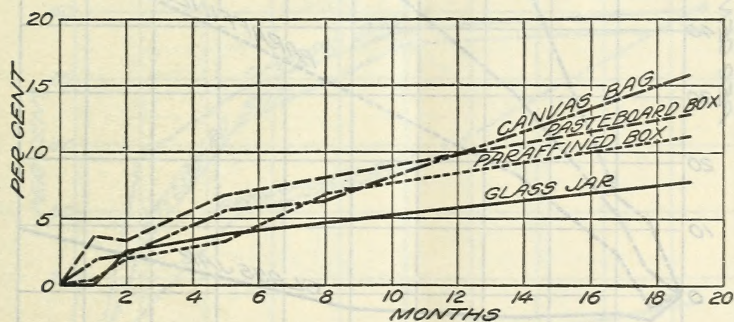


FIG. 2.—Loss of nicotine from nicotine sulphate-kaolin dust

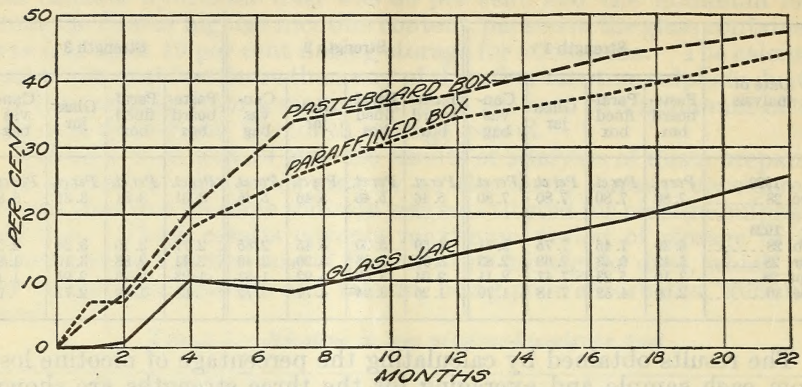


FIG. 3.—Loss of nicotine from nicotine sulphate-talcum dust

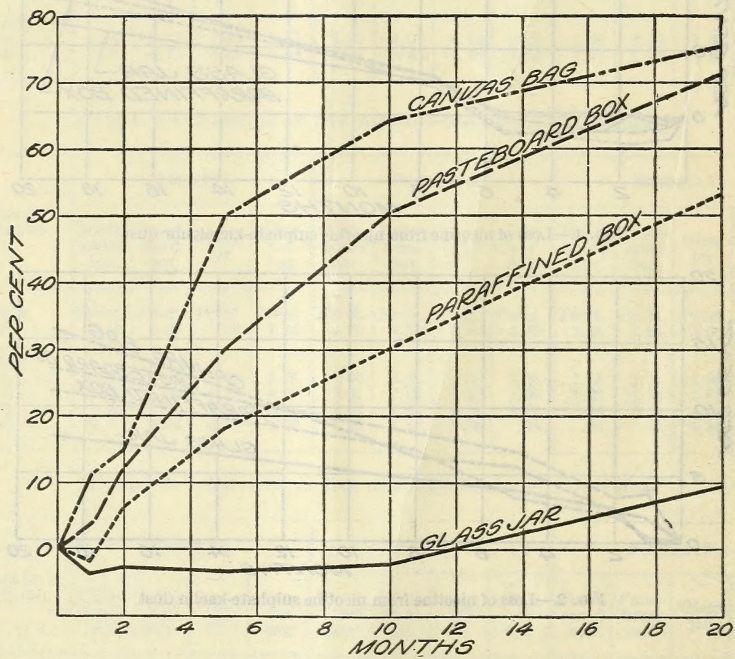


FIG. 4.—Loss of nicotine from nicotine sulphate-calcium hydroxide dust

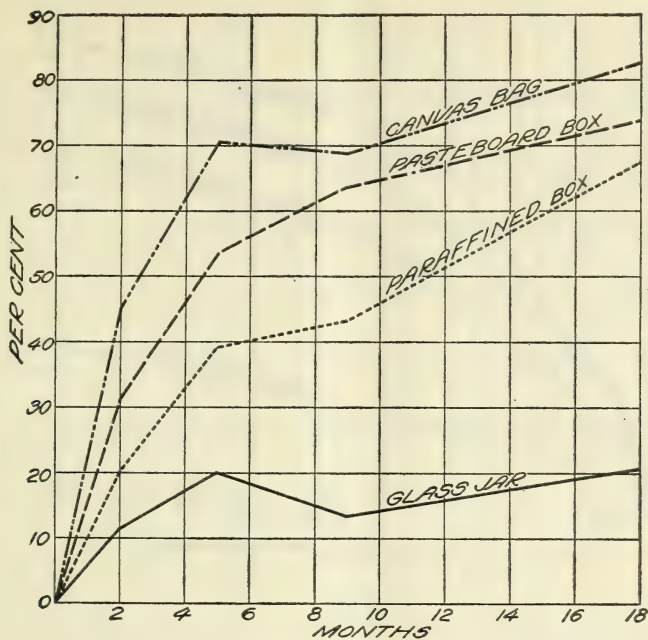


FIG. 5.—Loss of nicotine from nicotine sulphate-calcium carbonate dust

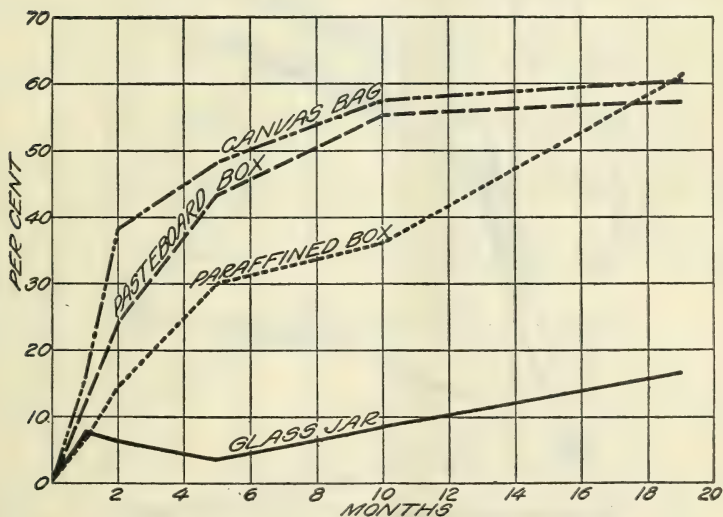


FIG. 6.—Loss of nicotine from nicotine sulphate-plaster of Paris dust

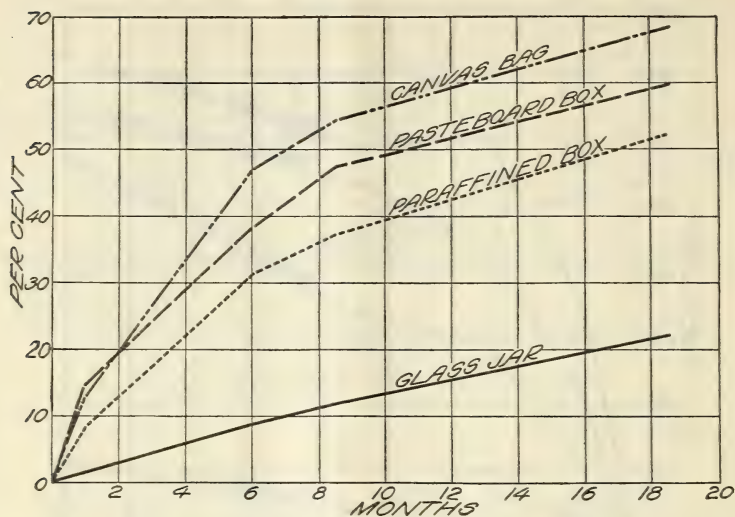


FIG. 7.—Loss of nicotine from free nicotine-kieselguhr dust

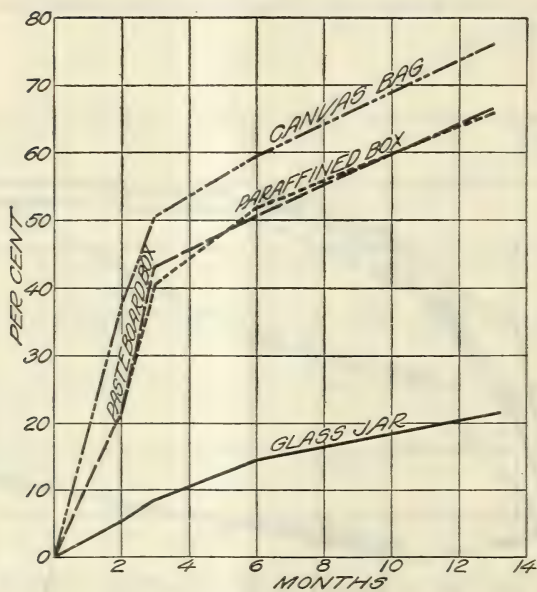


FIG. 8.—Loss of nicotine from free nicotine-calcium carbonate dust

The dusts prepared from free nicotine solution showed a greater loss in all cases than those prepared from nicotine sulphate. After 19 months, the maximum loss from the kieselguhr dust was 68 per cent and the minimum loss was 22 per cent of the quantity originally present. The greatest loss was from the plaster of Paris dust in canvas, which after 12 months reached 94 per cent of the quantity added. The most striking feature of these samples is the high initial rate of loss, except in the case of the samples packed in the glass jars, from which the loss was small during the first two or three months. During the first two months, the maximum losses were more than 50 per cent for the calcium hydroxide dust, 45 per cent for the calcium carbonate dust, and 42 per cent for the plaster of Paris dust.

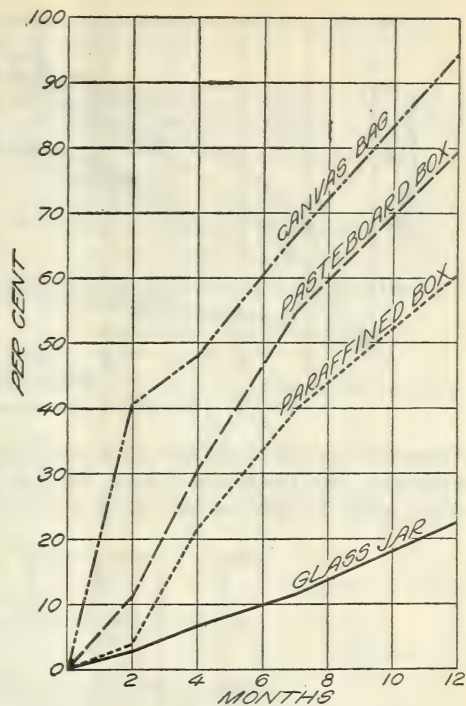


FIG. 9.—Loss of nicotine from free nicotine-plaster of Paris dust

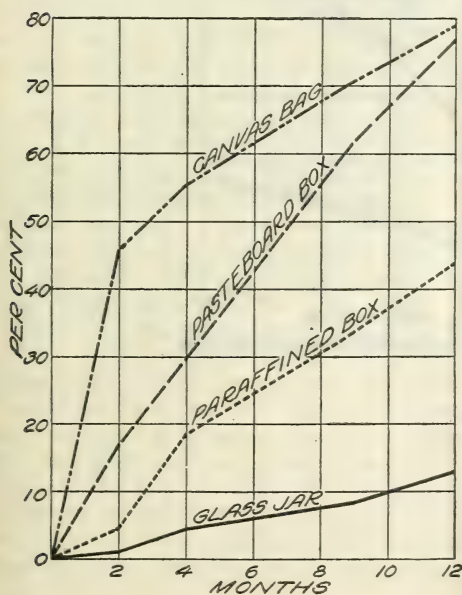


FIG. 10.—Loss of nicotine from free nicotine-calcium hydroxide dust

All the results obtained by using nicotine sulphate as the source of nicotine are summarized in Figure 11, which shows the effect of one variable only, the container, over a period of 20 months, for the six carriers and the three strengths. The loss from the glass jars is small. The other three containers show large losses and do not differ much.

Similar data for the free nicotine preparations, covering the four carriers used, are shown in Figure 12. As might be expected, the differences between the several types of containers are greater than was the case with nicotine sulphate. For the 12 months during which all of the samples were under observation the paraffined box

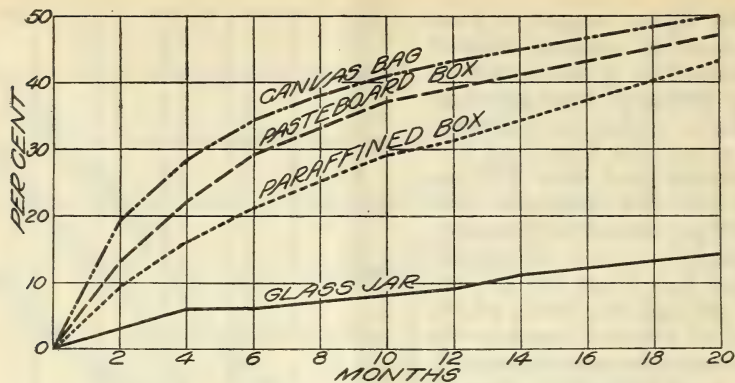


FIG. 11.—Average loss of nicotine from all nicotine sulphate dusts

showed a loss of 52 per cent, as compared with 31 per cent for nicotine sulphate; the pasteboard box, 67 per cent as compared with 39 per cent; and the canvas bag, 77 per cent as compared with 43 per cent.

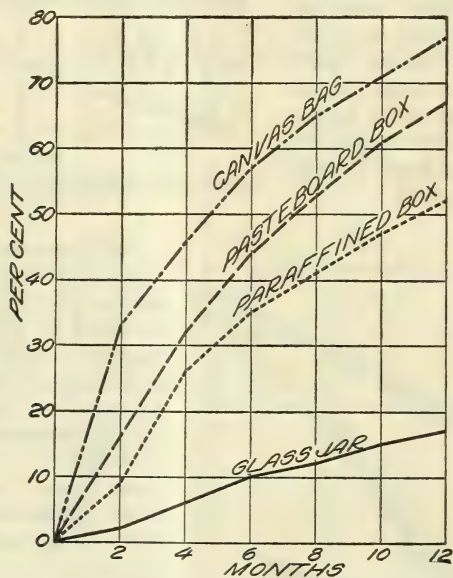


FIG. 12.—Average loss of nicotine from all free nicotine dusts

### VOLATILITY OF NICOTINE SULPHATE SOLUTION AND FREE NICOTINE SOLUTION

Nicotine is ordinarily incorporated in dusts as a solution of nicotine sulphate. As a few dusts made with free nicotine solution are on the market, however, the volatility of the two types of nicotine used, independent of any carrier, was determined.

Solutions of nicotine sulphate and of free nicotine were allowed to stand exposed in 500-cubic centimeter beakers at room temperature for several months. They were analyzed from time to time, record being kept of the weight of the samples and the quantity withdrawn for analysis.

Nicotine sulphate solutions of two strengths were used. Of one solution, 195.2 grams, containing 41.3 per cent (80.7 grams) of nicotine, was taken. At the end of three weeks there remained, after correcting for the material used in analysis, 79.3 grams of nicotine, or a loss of 1.4 grams. (This is probably within the limits of error.) The concentration of nicotine in the solution had increased to a little more than 45 per cent, owing to the evaporation of water. No further change occurred in the concentration of the solution. Of the other solution, 229.1 grams, containing 21 per cent (48.1 grams) of nicotine, was used. During the five months no loss of nicotine occurred, but at the end of the period its concentration was 49 per cent of nicotine.

Two strengths of free nicotine solutions, one containing originally 41.5 per cent and the other 20.8 per cent of nicotine, were exposed during the same period and in a similar manner. Both of these solutions reached a concentration of 89 per cent nicotine, but meanwhile they lost 10 and 17 per cent, respectively, of their nicotine content. These results are shown in Figure 13.

The experimental results with nicotine sulphate solutions confirm those with nicotine sulphate absorbed in an inert carrier, such as kieselguhr, the loss of nicotine from which did not exceed 1 per cent per month. A greater loss than this indicates a reaction between the nicotine sulphate and the carrier, by which the nicotine sulphate is broken up and free alkaloid is formed. This occurs in the presence of calcium hydroxide or calcium carbonate, which react to form calcium sulphate and free nicotine, nicotine dioxide being evolved when calcium carbonate is used.

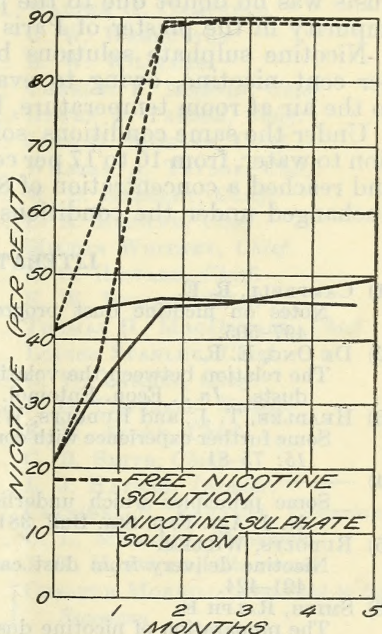


FIG. 13.—Changes in strength of nicotine sulphate and free nicotine solutions on standing in open vessels

## SUMMARY

The rate of loss of nicotine from nicotine sulphate dusts prepared with kaolin, kieselguhr, talcum, plaster of Paris, calcium hydroxide, and calcium carbonate follows the order in which the carriers are given.

Dusts made with free nicotine solution lose their nicotine much more rapidly than those made with nicotine sulphate. The effect of the carriers, however, is in about the same order in both cases.

Canvas bags, pasteboard boxes, and paraffined boxes are unsatisfactory containers for nicotine dusts. Air-tight metal or glass containers only should be used for packing them commercially.

The rapid loss of nicotine from plaster of Paris-nicotine sulphate dusts was no doubt due to the presence of calcium carbonate as an impurity in the plaster of Paris used.

Nicotine sulphate solutions became concentrated to about 45.5 per cent nicotine, owing to evaporation when they were exposed to the air at room temperature, but with little or no loss of nicotine.

Under the same conditions, solutions of free nicotine lost, in addition to water, from 10 to 17 per cent of the nicotine originally present, and reached a concentration of 89 per cent nicotine, which remained unchanged under the conditions of the test.

## LITERATURE CITED

- (1) CAMPBELL, R. E.  
Notes on nicotine dust progress. *In J. Econ. Entomol.* (1923), 16: 497-505.
- (2) DE ONG, E. R.  
The relation between the volatility and toxicity of nicotine in sprays and dusts. *In J. Econ. Entomol.* (1923), 16: 486-493.
- (3) HEADLEE, T. J., and RUDOLFS, WILLEM.  
Some further experience with contact dusts. *In J. Econ. Entomol.* (1922), 15: 75-81.
- (4) ———  
Some principles which underlie the making and use of nicotine dust. *N. J. Agr. Exp. Sta. Bull.* 381 (1923), 47 pp.
- (5) RUDOLFS, WILLEM.  
Nicotine delivery from dust carriers. *In J. Econ. Entomol.* (1922), 15: 421-424.
- (6) SMITH, RALPH E.  
The preparation of nicotine dust as an insecticide. *Calif. Agr. Exp. Sta. Bull.* 336 (1921), 14 pp.
- (7) THATCHER, R. W., and STREETER, LEON R.  
Factors which affect the volatility of nicotine from insecticide dusts. *N. Y. Agr. Exp. Sta. Bull.* 501 (1923), 34 pp.



**ORGANIZATION OF THE  
UNITED STATES DEPARTMENT OF AGRICULTURE**

December 1, 1924

---

<i>Secretary of Agriculture</i> .....	HOWARD M. GORE.
<i>Assistant Secretary</i> .....	
<i>Director of Scientific Work</i> .....	E. D. BALL.
<i>Director of Regulatory Work</i> .....	WALTER G. CAMPBELL.
<i>Director of Extension Work</i> .....	C. W. WARBURTON.
<i>Solicitor</i> .....	R. W. WILLIAMS.
<i>Weather Bureau</i> .....	CHARLES F. MARVIN, <i>Chief</i> .
<i>Bureau of Agricultural Economics</i> .....	HENRY C. TAYLOR, <i>Chief</i> .
<i>Bureau of Animal Industry</i> .....	JOHN R. MOHLER, <i>Chief</i> .
<i>Bureau of Plant Industry</i> .....	WILLIAM A. TAYLOR, <i>Chief</i> .
<i>Forest Service</i> .....	W. B. GREELEY, <i>Chief</i> .
<i>Bureau of Chemistry</i> .....	C. A. BROWNE, <i>Chief</i> .
<i>Bureau of Soils</i> .....	MILTON WHITNEY, <i>Chief</i> .
<i>Bureau of Entomology</i> .....	L. O. HOWARD, <i>Chief</i> .
<i>Bureau of Biological Survey</i> .....	E. W. NELSON, <i>Chief</i> .
<i>Bureau of Public Roads</i> .....	THOMAS H. MACDONALD, <i>Chief</i> .
<i>Bureau of Home Economics</i> .....	LOUISE STANLEY, <i>Chief</i> .
<i>Bureau of Dairying</i> .....	C. W. LARSON, <i>Chief</i> .
<i>Fixed Nitrogen Research Laboratory</i> .....	F. G. COTTRELL, <i>Director</i> .
<i>Office of Experiment Stations</i> .....	E. W. ALLEN, <i>Chief</i> .
<i>Office of Cooperative Extension Work</i> .....	C. B. SMITH, <i>Chief</i> .
<i>Office of Publications</i> .....	L. J. HAYNES, <i>Director</i> .
<i>Library</i> .....	CLARIBEL R. BARNETT, <i>Librarian</i> .
<i>Federal Horticultural Board</i> .....	C. L. MARLATT, <i>Chairman</i> .
<i>Insecticide and Fungicide Board</i> .....	J. K. HAYWOOD, <i>Chairman</i> .
<i>Packers and Stockyards Administration</i> .....	} CHESTER MORRILL, <i>Assistant to the</i> <i>Secretary</i> .
<i>Grain Futures Administration</i> .....	

---

This bulletin is a contribution from

<i>Bureau of Chemistry</i> .....	C. A. BROWNE, <i>Chief</i> .
<i>Miscellaneous Division</i> .....	J. K. HAYWOOD, <i>Chief</i> .



