

FILE NAME: Owens Illinois (OWILL)

DATE: 1952 Jan 30

DOC#: OWILL052

DOCUMENT DESCRIPTION: Confidential Report - Investigation Concerning the Capacity of Inhaled Kaylo Dust to Injure the Lung with Cover Letter

DCS/5

THE TRUDEAU FOUNDATION
FOR
THE CLINICAL AND EXPERIMENTAL STUDY OF PULMONARY DISEASE

SARANAC LAKE, N.Y.

THE SARANAC LABORATORY
THE TRUDEAU FOUNDATION
THE TRUDEAU SCHOOL

AT TRUDEAU, N.Y.

THE DEPARTMENT OF PHYSIOLOGY
THE DEPARTMENT OF BIOCHEMISTRY
THE DEPARTMENT OF RADIOLOGY
THE TRUDEAU LABORATORY

February 7, 1952

Mr. W. C. Hazard
Industrial Relations Division
Owens-Illinois Glass Company
Toledo 1, Ohio

Dear Bill:

Herewith is the final report of our studies concerning The Capacity of Inhaled Kaylo Dust to Injure the Lung. We are enclosing four copies for distribution by you. One copy has been sent to Doctor Shook for his information and we are retaining a copy for our files.

The results of the investigations with animals show that Kaylo dust is capable of producing a peribronchiolar fibrosis typical of asbestosis. The dust also has a slightly unfavorable influence upon a tuberculous infection. Although extrapolation from animal to human experience is difficult, nevertheless the results of the study indicate that every precaution should be taken to protect workers against inhaling the dust. Therefore, control measures should be directed to reducing the amount of atmospheric dust, especially at those points of operation where dust is generated. Our report of May 29, 1951 concerning the industrial hygiene survey may be of help to you in this regard.

We hope to publish this study either separately or in combination with similar studies pertaining to other dust. In doing so, however, reference will be made only to hydrous calcium silicate and not to "Kaylo;" thus the interest of your Company will be safeguarded. Of course the final manuscript will be forwarded to you for review before being released to the publisher. Your comment in this regard would assist us greatly in preparing the manuscript.

In submitting this final report, may I express to you and others of the Company our sincere appreciation for having had the privilege of collaborating with the Owens-Illinois Glass Company in the study of Kaylo. The collaboration has always been most pleasant and stimulating.

My every best wish,

Sincerely yours,

Arthur J. Vorwald, M.D.
Director

AVJ:LB

Encs. (4)

CC: C. F. Shook, M.D.

Grateful & Salutatory

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CONFIDENTIAL

Final Report

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Urban
on 1/30/52*

INVESTIGATION CONCERNING THE CAPACITY OF INHALED KAYLO DUST
TO INJURE THE LUNG



to

Owens-Illinois Glass Company

Toledo, Ohio

by

The Saranac Laboratory

Saranac Lake, New York

January 30, 1952

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INVESTIGATION CONCERNING THE CAPACITY OF INHALED KAYLO DUST
TO INJURE THE LUNG

I. INTRODUCTION

In 1943 the Owens-Illinois Glass Company had developed to the pilot plant stage a new industrial product, known as Kaylo, which could be used as a thermal insulating material and for other applications. According to information supplied by the company, this product was prepared by heating under steam pressure a suspension of calcium hydroxide, silica, and asbestos to form a hydrated calcium silicate. Studies at The Saranac Laboratory showed that although the principal component of Kaylo was the hydrated calcium silicate, some unchanged raw materials, including asbestos and free silica in the form of quartz or diatomaceous earth, were also present in the final product. Before Kaylo was used commercially on a large scale, it was important to determine whether or not any dust liberated from this product during manufacture, handling or fabrication would be likely to constitute a respiratory hazard to exposed individuals. Therefore, a comprehensive investigation was undertaken in which the effect of inhaled Kaylo dust on animals was studied. The investigation was started in February 1945 and was carried on for more than five years. It explored the nature of the pulmonary tissue reaction to inhaled Kaylo dust in normal animals and in animals harboring a tuberculous infection. In this final report of the study the principal findings given in previous interim reports will be reviewed briefly and the conclusions derived from the entire experimental program will be stated.

II. DUSTING MATERIAL USED

The material used for generating atmospheric dust in the experimental dust room was finely-divided Kaylo obtained from the dust collectors of the Berlin, New Jersey plant, in which Kaylo is manufactured. The material was received in separate shipments, as follows:

Lot #1 - Received in January 1945.
#2 " " February 1947.
#3 " " January 1949.

A. Composition: The results of chemical and petrographic analysis of a representative sample of the material as received at The Saranac Laboratory, are given in table 1. It will be noted that the only asbestos mineral revealed by the petrographic examination of this sample is chrysotile. Since Kaylo is a commercial product, it is possible that minor alterations in composition may occur from time to time in accordance with changes in the raw materials used. For example, the quartz value for lot #1 of Kaylo was less than 1 per cent but the quartz content of lots #2 and #3 was between 2 to 5 per cent. In the final shipment of Kaylo, received in January 1949 and first used in this experiment in September 1949, the asbestos component contained the mineral amosite as well as chrysotile. The fibers of amosite, in comparison with those of chrysotile, are less flexible and have a higher iron content. However, since experiments with animals have shown that both amosite and chrysotile are capable of causing asbestosis, it is unlikely that the substitution of the one mineral for the other in Kaylo would cause a significant change in the toxic properties of the final product.

B. X-Ray Diffraction studies: The X-ray diffraction pattern of samples of two different shipments of Kaylo are shown in fig. 1. The prominent lines present are probably those of a hydrous calcium silicate, which is the principal component of Kaylo. Comparison of patterns a and b discloses that the quartz line is stronger in b than in a, indicating that the Kaylo received in January 1949 has a higher quartz content than has the earlier product, received in January 1946. Faint lines representing calcite and chrysotile are visible in both patterns. Diffraction lines produced by asbestos are also present in pattern b.

C. Size-frequency studies: The size-distribution of Kaylo dust is shown by the graph in fig. 2. The lines of the graph represent the parent material in the dust machine and two samples of atmospheric dust collected in the dust room on different days. A magnification of 1200X was used in making the size-frequency counts. It will be noted that about 83 per cent of the particles in the parent material visible at that magnification are smaller than 5 microns in size; the corresponding values for the samples of atmospheric dust are 90 and 95 per cent. This difference in the two samples of atmospheric dust, which were derived from the same parent material, may have been due to the day-to-day variation in humidity inside the dust room.

III. REVIEW OF PREVIOUS REPORTS

During the course of the investigation with Kaylo dust five interim reports were issued.

The first report, dated May 13, 1946, revealed that in guinea pigs

exposed for 14 months to Kaylo dust the tissue reaction consisted only of microscopic focal collections of dust cells. The associated tuberculous infection had not been altered enough to be of significance.

In the second report, issued on October 30, 1947, it was noted that there was no evidence suggesting that Kaylo dust when inhaled into the lungs produces pulmonary fibrosis in rats exposed for 18 months or in guinea pigs exposed for 30 months. These findings seemed to show Kaylo dust to be a biologically inert material. In the experiment dealing with the effect of the dust on a tuberculous infection the results were inconclusive, indicating that the experiment should be repeated.

The third report, dated October 30, 1948, presented the final observations on the effect of inhaled Kaylo dust on normal uninfected guinea pigs. Contrary to previous tentative impressions, based upon an exposure of 30 months, which suggested Kaylo dust to be inert, the study of animals exposed for 36 months disclosed that the material is not inert but is capable, on prolonged inhalation, of producing asbestosis in guinea pigs. However, neither silicosis nor the diffuse fibrosis which is caused by inhaled diatomaceous earth developed in the exposed animals.

The fourth report, dated April 30, 1949, reviewed the results of the first 8 months of a new experiment in which the effect of inhaled Kaylo dust on a tuberculous infection in guinea pigs was studied. No definitely adverse effect of the dust on the infection during this 8-months period was noted.

The fifth report, dated January 1, 1950, supplemented the information given in the fourth report by extending the observations to infected animals

that had been exposed to Kaylo dust for periods up to 14 months. The findings at that time suggested that Kaylo dust had a slightly adverse influence on the course of a tuberculous infection in guinea pigs but that the effect was by no means as pronounced as that of quartz dust.

IV. INHALATION EXPERIMENT WITH KAYLO DUST

As a continuation of the previous interim reports and as a summary for this final report, a review of the entire program of study is reported.

In the inhalation experiment (Saranac Laboratory No. 1012, 1100, 1104) groups of guinea pigs, rats, and hamsters were placed in a cubical dust room, 3 feet in dimension, in which an atmospheric suspension of Kaylo dust was maintained. The room was operated for more than five years, from February 1945 to June 1950. About 450 animals were used in the experiment and some of them lived in the dust room for as long as 3 years.

A. Conditions of exposure

The atmospheric suspension of Kaylo dust was created by the action of a paddle that rotated inside a hopper, located in a corner of the dust room, which contained the finely-divided Kaylo product. The dust cloud generated in this manner floated out into the room where it was maintained for 8 hours on five days of the week and for 4 hours on Saturdays. At regular intervals during the experiment a few animals were killed and the tissues examined grossly and microscopically to determine the nature and the extent of the dust reaction. The tissue was also analyzed chemically to estimate the amount of Kaylo dust retained in the lungs of animals exposed for definite periods of time.

Dust counts of atmospheric samples collected in the dust room with the midge impinger were made regularly. The concentration of Kaylo dust to which the uninfected animals were exposed was generally within the range of 100 to 125 million particles per cubic foot of air and the overall average was 115 million. In the experiment with infected animals the concentration was higher, the average being 205 million.

B. Exposure of uninfected animals

Three species - guinea pig, rat, hamster - were employed in this phase of the investigation. A summary of the findings is given in table 2.

(1) Guinea pigs. At the beginning of the experiment 100 uninfected guinea pigs were placed in the dust room and 6 months later 25 more were added to replace animals that had died in an epidemic of acute pneumonia. Since this pneumonia occurred at that time also in animals exposed to another dust in an adjacent room, the unfortunate epidemic was considered accidental. Epidemics of this kind, which occurred occasionally in experiments at that time, have since been controlled. Groups of guinea pigs, usually 2 to 4 in number, were killed at 2, 4, 6, 8, 12, 18, 21, 24, 27, 30, 33, and 36 months after the beginning of their exposure to Kaylo dust so that the course of the pulmonary reaction could be followed.

In guinea pigs exposed for periods up to 30 months there was only a mild tissue response to the inhaled Kaylo dust and the reaction was limited to the accumulation of clumps of dust-containing macrophages in groups of adjoining air spaces (fig. 3). The intervening alveolar walls were only slightly thickened by accumulated lymphocytes. Occasional multinucleated

ghost cells were visible but there was no excessive proliferation of histiocytes or deposition of connective tissue. A few asbestos bodies were seen (fig. 4). Microscopic study of the tracheobronchial lymph nodes, which became enlarged but remained soft, revealed clusters of inactive dust cells similar to those in the lungs but without asbestos bodies. It is pertinent to point out here that animals exposed to asbestos dust for a similar 30-months period exhibit a severe tissue reaction with the production of fibrosis (fig. 5). For comparison, a lung section of a normal guinea pig is shown (fig. 6).

The 3 animals exposed to Kaylo dust for more than 30 months (3 animals for 33 months and 6 animals for 35 months) showed not only the lesions like those of the 30-months animals but also exhibited a true fibrosis of a type characteristic of the response of guinea pigs to inhaled asbestos fibers. This reaction is a rather diffuse type of fibrosis which forms principally about the bronchioles, the small ducts from which the terminal air spaces, or alveoli, arise. A comparison of fig. 7 and fig. 5 reveals the similarity of the tissue reaction produced in the lungs by Kaylo dust and by asbestos dust. The lesions in the tracheobronchial lymph nodes showed no change between 30 months and 35 months.

(2) Rats. This species was employed because typical nodular fibrosis produced by inhaled quartz dust develops more rapidly in rats than in animals of other species. Forty rats were used and some of them were exposed to Kaylo dust for as long as 18 months. Pneumonia was not a problem during the first year of the experiment but among the rats exposed for 15 to 18 months 16 animals died with terminal bronchiolitis and lobular

phenomena. Since laboratory rats older than one year, even those not exposed to dust, are commonly subject to this type of lesion, the high mortality rate is not considered significant. In order to follow the progression of the tissue reaction to the inhaled Kaylo dust rats were killed in groups of 4 after 6, 9, 12, 15, and 18 months of exposure. It was revealed (fig. 8) that throughout the experiment the reaction to the dust consisted of focal collections, in the alveoli, of macrophages which contained particles and spicules of dust. The only change observed as the exposure period became longer was an increase in the number of the alveolar foci. Similar dust-filled macrophages were seen in the tracheobronchial lymph nodes. In no animal was there any tissue reaction typical of silicosis. The peribronchiolar fibrosis of asbestosis was not discovered, though occasional asbestosis bodies were observed. In view of other experiments with pure long-fibre asbestos dust, typical peribronchiolar fibrosis would not be expected in 18 months exposure to Kaylo dust.

Reaction in hamsters

(3) *Hamsters.* Twenty hamsters were placed in the dust room and after 6, 9, 12, and 18 months of exposure 2 animals were killed at each period for study; at the 6-months period 3 animals that died from other causes were examined. Histologic examination revealed that the Kaylo dust accumulated very slowly in the lungs. No fibrosis was seen except in one hamster, which had been exposed for 18 months. In that animal there was a trace of peribronchiolar fibrosis so slight that it could be definitely recognized only at high magnification. Asbestosis bodies in small numbers and at the usual sites were present.

Experiment on tuberculous animals

There are three types of experiment which are valuable for determining the effect of inhaled dust on the development of tuberculosis in experimental animals. In the simultaneous infection experiment normal animals are infected with attenuated tubercle bacilli by the inhalation method and then are immediately transferred to the dust room. Thus, the tuberculous disease and the reaction to the inhaled dust will develop simultaneously. In the reactivation experiment the animals, after being infected with the tubercle bacilli, are allowed to live in a clean atmosphere for several months before being exposed to dust. During this period the tuberculous disease generally regresses and some healing takes place. Following exposure of the animals to some dusts the lesions may continue to regress or, with other dusts, the tuberculous process may become active and spread. The predisposition experiment is one in which animals are exposed to dust for several months, are then infected, and are immediately returned to the dust room, where their exposure to dust is continued. In this way the effect of a previous dust exposure on the early course of a tuberculous infection can be studied.

For investigating the effect of inhaled Kaylo dust on the course of tuberculous infection a particular attenuated strain of human-type tubercle bacilli, known as the R₁ strain, was employed. This strain has a low degree of virulence for guinea pigs and, in the normal animal, produces a self limited disease which will progress for 2 to 3 months and subsequently heal. In an animal which has inhaled quartz dust or certain other toxic dusts, however, the disease usually progresses continuously and ultimately causes the death of the animal. Since experience has indicated that tubercle

bacilli of the R_1 strain have about the same degree of virulence for the guinea pig as tubercle bacilli of the human strain have for man, it is possible to obtain some correlation between the influence of various dusts on the R_1 tuberculous infection in guinea pigs and the effect of these dusts on a tuberculous disease in industrial workers.

For infecting the animals with tubercle bacilli an inhalation technic was used. A glass tube was fitted over the animal's nose and into this tube a suspension containing a known number of bacilli of the R_1 strain was sprayed with a DeVilbiss atomizer. As the animal breathed, he inhaled the atmospheric suspension of bacilli into the lungs, where they became localized.

(1) Simultaneous infection: guinea pigs infected at the time their exposure to Kaylo dust was started. In the first experiment in which animals were infected with tubercle bacilli at the time that their exposure to Kaylo dust was started (table 3), 25 guinea pigs were used but an episode of pneumonia during the first 10 months reduced to 16 the number of animals available for study. Two of these died at 14 months, apparently from progressive tuberculosis, and 2 others died from undetermined causes, but not from tuberculosis. In the remaining 12 animals the course of the tuberculous disease was followed by killing 1 or 2 animals at 1, 6, 9, 12, 15, 18, and 24 months after infection. In 11 of the 12 animals the lesions were isolated, encapsulated, and regressing; in the remaining animal, which was killed at 15 months, the lesions were widespread throughout the lung but fibrosis had been produced, which indicated a tendency to heal. In an attempt to develop a strain of guinea pig resistant to the infection that had caused the pneumonia, some of the animals were used for breeding during

this experiment. The 2 animals that died from progressive tuberculosis (at 14 months) and the single animal, killed at 15 months, that exhibited widespread lesions were all used as breeders during the experiment but 4 other animals in the experiment, which were also employed for breeding, failed to show extensive disease. Owing to the element of uncertainty introduced by this factor of breeding, and also because of the high mortality from pneumonia, it was considered impossible to establish, from this experiment, definite conclusions about the effect of inhaled Kayle dust upon tuberculosis, and therefore the experiment was repeated.

The simultaneous infection experiment was conducted a second time (table 3) with 30 guinea pigs, instead of 25, and animals were killed in pairs for study at 2, 4, 6, 8, 12, 15, and 24 months after infection. Again the incidence of pneumonia was unusually high, 6 of the animals dying from that cause during the first 8 months of exposure and 2 more during the following 16 months. Of the remaining 22 guinea pigs 4 animals revealed, at 15, 23, and 24 months after infection, a spreading tuberculous disease, of minimal extent (fig. 2), which was confirmed by microscopic examination. Three other animals also had a spreading tuberculosis but in them the disease had extended to the spleen and other organs. These animals were considered exceptions and in the past have been classified as "sports". The term "sport" is given to a guinea pig in which the inhalation infection with the attenuated bacilli is not confined principally to the lungs and tracheobronchial lymph nodes but extends also to other organs and produces in them tuberculous changes of sufficient extent to be recognized in the gross. Since "sports" represent a departure from the normal pattern of tissue reaction to attenuated

tubercle bacilli, whether the infection is combined with dust exposure or not, these animals must be excluded in assaying the effect of an inhaled dust upon a tuberculous infection. It is believed that "sports" are animals whose native resistance to the attenuated R_1 organism is unusually low. Support for this belief is given by experience with quartz dust, a definitely hazardous material, which reactivates an inhalation infection produced by attenuated R_1 bacilli but ordinarily is not associated with tuberculous extension to organs other than the lungs and tracheobronchial lymph nodes.

The control non-dusted group (table 3) of guinea pigs for this experiment was infected at the same time as the experimental group of animals subsequently dusted. The same culture of organisms was used for all animals. The tuberculous infection followed its usual course in the control group and resolved in all of the animals except one, a "sport", in which the disease spread.

Although a progressive tuberculous disease was noted in 4 guinea pigs of the second experiment, the degree of extension of the disease in them was slight. The remaining 23 animals, 8 of which died of intercurrent pneumonia, showed no spread and the infection followed the natural course of regression and healing as in the non-dusted control animals. It is believed, therefore, that the inhaled Kayle dust had no significantly adverse effect on the tuberculosis.

- (2) Reactivation infection: guinea pigs, after being infected, lived in clean air for several months before being exposed to Kayle dust.

In the reactivation experiment (table 4) 23 guinea pigs were infected with the attenuated bacilli and allowed to live in a clean atmosphere. Animals

of this group were transferred to the dust room at 2 months (10 animals), 4 months (3 animals), and 6 months (3 animals) after infection. In order to follow the course of the tissue reaction a pair of animals of the first sub-group (2 months in clean air) was killed after only 2 months of dust exposure and, in addition, animals from all groups were killed in pairs after 4, 8, and 12 months of exposure to the dust. Examination of the tissue of the dusted animals failed to reveal a significant reactivation of the tuberculous disease by the inhaled Kaylo dust. Only one animal showed evidence of spreading pulmonary tuberculosis, minimal in extent (fig. 19). That animal was 1 of 3 that had lived in clean air for 2 months after infection and then had been exposed to the dust for 12 months.

In the control group (table 4) the tuberculous disease behaved in the normal manner and healed, with the exception that one animal (a "sport") developed a spreading disease and died from tuberculosis 6 months after infection.

(3) Predisposition infection: Guinea pigs were infected 3 months after their exposure to Kaylo dust was started. An experiment of this type is a severe test because a dust that is only very slightly toxic may produce tissue changes which, though minor in character, may be sufficient to alter profoundly the development of a fresh tuberculous infection. In this experiment (table 5), which was designed to study the effect of Kaylo dust accumulated in the lung upon a newly-developing tuberculous disease, 30 guinea pigs were exposed to the dust for 3 months and then were infected with attenuated tubercle bacilli. The dust exposure was immediately resumed and carried on for another 21 months.

During the course of this experiment 5 animals were killed at the end of the preliminary 3 months dusting period. They showed only an occasional clump of dust phagocytes within the alveolar spaces. Of the dusted and infected animals, 7 died from intercurrent pneumonia, which eliminates them for purposes of analysis; 1 animal died at 14 months after infection from spreading tuberculosis; and the remaining 17 animals were killed in pairs at 2, 4, 6, 8, 10, 12, and 16 months and 3 were killed at 21 months after infection. Of the latter group of 17 animals, 1 showed many spreading tubercles, 10 revealed scattered spreading tubercles and 6 exhibited healed tubercles. These findings indicate that a tuberculous infection in guinea pigs that originates several months after a prolonged exposure of the animals to Kayle dust was initiated may be unfavorably influenced by the inhaled dust. This influence, however, is minimal in degree.

The tuberculous infection in the control animals (table 5) in general regressed and healed. Of the control animals there was, as usual, 1 "spert animal", killed 10 months after infection, which revealed a few scattered isolated tubercles that were of the spreading type.

F. Analysis of tissue of exposed animals

Chemical analysis of the lungs of uninfected guinea pigs that had inhaled Kayle dust for periods up to 36 months yielded the data reported in table 6. It will be noted that as the period of exposure became longer the ash values gradually increased, thus showing that mineral matter was accumulating in the lungs. There was a pronounced increase in the silica component up to about 30 months and then a slight decrease. The reason for

the dust, which has been observed in a number of other dusts, is not entirely clear but it seems to be due in part to dilution of the dust with the inorganic components stabilized as a result of the increased disease reaction which developed, as already mentioned, in the guinea pigs between 30 and 36 months of exposure to Kaylo dust. Since only 3 animals were analyzed for each exposure period, individual values occasionally were out of line but the general trend was clearly indicated.

W. REVIEW OF EXPERIMENTAL FINDINGS

The findings derived from the various inhalation experiments will be reviewed briefly and some points of significance mentioned.

A. Findings for guinea pigs inhaling Kaylo dust

The results of the inhalation experiments given Kaylo dust, when inhaled into the lungs of guinea pigs for a prolonged period (30 to 36 months), is capable of producing the peribronchial fibrosis characteristic of the disease asbestosis. No fibrosis was found by the inhaled dust in rats or in hamsters, with the exception that fibrosis was barely demonstrable in a single hamster, exposed for 18 months. In animals exposed for 30 months or less the lesions showed no fibrosis but certain aspects of the lesions, although masked by the reaction to the inert components in Kaylo dust, were compatible with an early stage in the development of asbestosis, a disease in which there is a predilection of the fibers to localize in the terminal bronchioles. This behavior is unlike that of particulate material, which is initially deposited chiefly in the alveoli rather than in the bronchi.

the bronchioles. Since the major portion of Kaylo dust is particulate, a great deal of it lodged in the alveoli and the reaction overshadowed the developing peribronchiolar response to the asbestos fibers of Kaylo. It was noted that in the region of the bronchioles of animals which were exposed for less than 30 months there was evidence of more dust cell activity than is usual in the reaction to an inert dust. Little significance was attached to this observation at first, however, because there was no indication of fibrosis. The early reaction to Kaylo dust is somewhat different from that to asbestos dust, since the particulate components modify the type of lesion. In silicosis modification of the lesions is a well-recognized condition which follows the inhalation of a mixed dust containing quartz and certain inert material, such as iron oxide.

The rate of pneumonia among the animals exposed to Kaylo dust was unusually high. Since the pneumonia rate has been elevated also in several inhalation experiments conducted with asbestos dust, it is possible that the asbestos component of Kaylo may have an unfavorable influence on the incidence of pneumonia. Verification of this possibility must await further study.

While the experimental investigation proved that Kaylo dust is capable of producing asbestosis, the results showed that the dust will not cause silicosis in animals of the three species tested. In none of the animals, even those exposed for the full 3-year period, was there any focus of the hyaline nodular fibrosis characteristic of silicosis. A study of the exposed animals disclosed also that Kaylo dust did not produce the diffuse fibrosis which follows the inhalation of diatomaceous earth. In regard to the hydrated

calcium silicate which is the major constituent of Kaylo it may be said that although there is no specific information about the effect of that compound on pulmonary tissue, there is much clinical and experimental evidence to suggest that it is biologically inert. Furthermore, in the inhalation experiment with Kaylo dust there was no deleterious effect noted which could be attributed to the calcium silicate component of the inhaled material. This observation is supported by our studies concerning similar calcium silicates in cement.

3. Findings for tuberculous animals inhaling Kaylo dust

The inhalation experiments in which tuberculous guinea pigs were exposed to Kaylo dust indicate that when the animals were infected with attenuated tubercle bacilli at the time exposure to the dust was started, or were infected prior to that time, the inhaled Kaylo dust had little or no unfavorable effect of significance on the tuberculous infection. In most of the exposed animals the tuberculous disease healed in a normal manner and in the few animals that exhibited a spreading type of lesion the aggravation of the disease by the dust was minimal.

When the animals inhaled Kaylo dust for several months before being infected, the subsequent accumulation of the dusts in the lungs seemed to have a slightly unfavorable influence on the usual course of the tuberculous disease.

VI. SUMMARY

A. Kaylo dust, when inhaled for a prolonged period, is capable of producing

in the lungs of guinea pigs, but not of rats, the peribronchiolar fibrosis typical of asbestosis.

B. The inhalation of Kaylo dust will not cause in guinea pigs, rats, or hamsters either silicosis or the diffuse fibrosis which sometimes follows the inhalation of diatomaceous earth.

C. The inhalation of Kaylo dust does not significantly influence the usual course of tuberculosis in guinea pigs infected with attenuated tubercle bacilli, provided the infection is given to the animals before they are first exposed to the dust. In guinea pigs that have inhaled Kaylo dust for several months before being infected, a continuation of the exposure appears to have a slightly unfavorable effect on the disease.

Table 1

ANALYSIS OF KAYLO

(Analysis of lot #2, received in January 1947)

Chemical analysis

SiO ₂	43.30%
Fe ₂ O ₃	1.62
Al ₂ O ₃	1.97
TiO ₂	0.12
MnO	0.09
CaO	24.84
MgO	7.10
Na ₂ O	0.17
K ₂ O	0.15
Loss below 105°C	4.52
" above 105°C	15.12
	<u>92.74</u>

Petrographic and X-ray diffraction analysis

The material consisted principally of clay-like masses which produced a definite X-ray diffraction pattern, presumably that of a hydrous calcium silicate. Quartz particles up to 50 microns in size were present, but they were not numerous and by weight probably made up less than 1 per cent of the sample. About 5 per cent of the material was calcite and approximately 15 to 20 per cent was chrysotile or serpentine, which was largely fibrous. The fibers and bundles present were usually several millimeters in length and were seldom shorter than 0.3 millimeter.

Table 3

SUMMARY OF INSTALLATION EXPERIMENTS WITH VARIOUS DOSES

Exposure of Uninfected Animals

Nature of Experiment	Animals used	Maximum duration of exposure to Tylo dust	Results
Uninfected animals exposed continuously to Tylo dust until death.	125 g. pigs	36 months	Peribronchiolar fibrosis, like that of asbestosis, produced after 36 months of exposure. At 30 months of exposure reaction was limited to accumulation of clumps of dust-containing macrophages, without fibrosis. A few asbestosis bodies were seen. Tracheobronchial lymph nodes became enlarged but remained soft and without fibrosis.
	40 rats	18	Focal collections of dust-containing macrophages in the alveoli and in the tracheobronchial lymph nodes. Increase in number of focal collections was the only change observed as exposure period became longer. No silicosis or asbestosis.
	30 hamsters	10	Lesions occurred very slowly. No fibrosis except in one animal which showed very slight fibrous reaction after 10 months of exposure. Asbestosis bodies in small numbers and at nasal sites.

Table 1

OUTCOME OF INHALATION EXPERIMENTS WITH KAYLO DUST

Exposure of Infected Animals
 (Simultaneous infection phase)

Nature of experiment	Animals used	Maximum duration of exposure to Kaylo dust	Results
Animals infected with tubercle bacilli at beginning of exposure to Kaylo dust	25 g. pigs	24 months	Results inconclusive; adverse influence of Kaylo dust on a tuberculous infection not definitely established. Progressive disease in 5 animals that had been used as breeders.
Controls to infection; no exposure to dust	25 g. pigs	0	Infection followed usual course; no spreading disease. No pneumonia.
Animals infected with tubercle bacilli at beginning of exposure to Kaylo dust. (Repetition of previous experiment)	27 g. pigs	24	Little or no significantly adverse effect of inhaled Kaylo dust on a tuberculous infection was demonstrated. Confirmed spreads, all of slight degree, in 4 of 27 exposed animals; the other 3 exposed animals were "cures".
Controls to infection; no exposure to dust	24 g. pigs	0	Infection followed usual course; no spreading disease except in 1 "cure".

All guinea pigs were infected with tubercle bacilli of the low virulent E₁ strain.

Table 4

SUMMARY OF INHALATION EXPERIMENT WITH KAYLO DUST

Exposure of Infected Animals

(Reactivation phase)

Nature of experiment	Animals used	Maximum duration of exposure to Kaylo dust	Results
Animals infected with tubercle bacilli* and then left in clean air for the following periods before being exposed to dust: 2 months 6 months 6 months	10 guinea pigs 8 8	months 12 12 12	No definite evidence of reactivation of the tuberculous infection except in 1 animal, exposed to Kaylo dust for 12 months, that had a slight spread of the disease. No definite evidence of reactivation. No definite evidence of reactivation.
Controls to infection; no exposure to dust	34	0	Infection followed its usual course and resolved in all animals except one, a "spore", in which the disease spread to other organs and caused the death of the animal 6 months after infection.

*The guinea pigs were infected with tubercle bacilli of the low-virulent R₁ strain.

Table 5

SUMMARY OF INHALATION EXPERIMENT WITH KAYLO DUST

Exposure of Animals That Were Infected 3 Months After Their
Exposure to Kaylo Dust Was Started

Nature of experiment	Animals used	Maximum duration of exposure to Kaylo dust	Results
Animals exposed to Kaylo dust for 3 months, then infected with tubercle bacilli*, and the exposure to dust continued	30 g. pigs	3 (before infect.) plus 21 (after infect.)	Spreading disease in 1 animal killed 2 months after infection and in 2 animals dying 14 months after infection.
Controls to infection; no exposure to dust	25 g. pigs	0	Infection followed its usual course and resolved, with the exception that 1 animal, killed 10 months after infection, showed scattered isolated spreading tubercles.

*The guinea pigs were infected with tubercle bacilli of the low-virulent R₁ strain.

Table 3

ANALYSES OF LUNGS OF UNINFECTED CUMBER PIGS EXPOSED TO DUST
IN INHALATION EXPERIMENT WITH KAYLO DUST

(Exposure of animals to dust was continuous until death)

Period of exposure to dust (months)		2	4	6	8	10	12	18	21	24	27	30	33	36
		%	%	%	%	%	%	%	%	%	%	%	%	%
Amount of ash (% of dried tissue)		4.42	4.47	4.18	4.59	4.53	4.86	4.38	5.12	5.02	5.15	5.62	5.70	5.63
		4.94	4.42	3.74	4.34	4.21	4.99	4.90	5.25	4.71	4.73	5.55	5.33	5.56 5.15
Mineral components (% of dried tissue)	SiO ₂	0.24	0.22	0.25	0.31	0.46	0.53	0.59	0.54	0.63	0.32	1.10	0.97	0.65
		0.20	0.19	0.20	0.23	0.41	0.51	0.36	0.54	0.60	0.45	1.12	0.59	0.37
	CaO	0.68	0.69	0.68	0.15	0.14	0.14	0.08	0.19	0.15	0.14	0.14	0.15	0.13
		0.08	0.08	0.10	0.11	0.11	0.16	0.10	0.15	0.14	0.12	0.13	0.12	0.11
	MgO	0.07	0.09	0.08	0.03	0.21	0.22	0.23	0.11	0.08	0.19	0.05	0.21	0.10
		0.03	0.03	0.03	0.11	0.14	0.03	0.32	0.09	0.09	0.15	0.05	0.17	0.17
Mineral components (% of ash)	SiO ₂	5.57	4.37	5.33	5.04	10.12	10.60	12.27	12.64	12.79	15.05	19.56	17.16	15.10
		4.06	4.42	5.56	3.49	3.42	10.35	17.35	10.30	12.52	3.55	20.25	11.00	12.45
	CaO	1.35	1.09	2.00	2.75	3.04	2.00	1.23	3.30	4.54	2.37	2.56	2.25	2.89
		1.23	1.09	2.03	2.45	2.20	3.17	2.03	3.30	2.32	2.41	2.23	2.23	2.11
	MgO	1.59	1.09	2.00	1.77	4.50	4.65	5.31	2.08	1.20	5.73	0.92	5.77	3.20
		1.32	1.39	2.10	2.45	3.02	1.30	3.50	1.45	1.72	3.03	0.91	3.25	3.17

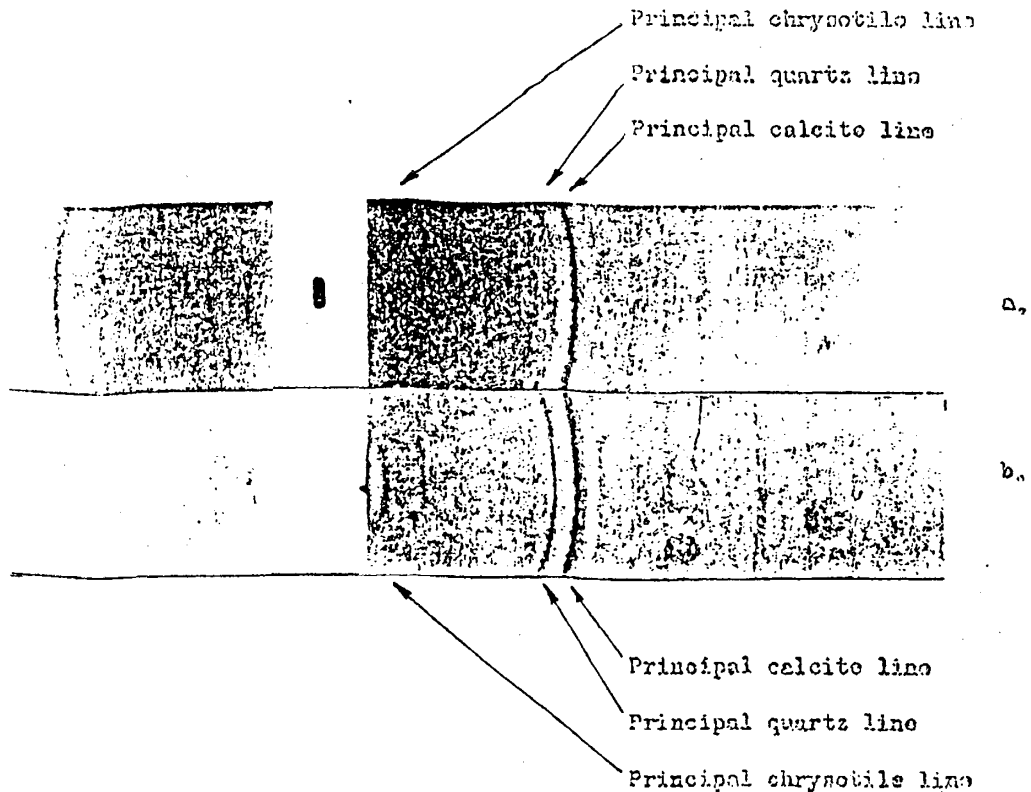


Fig. 1

X-RAY DIFFRACTION PATTERN OF KAYLO

a. Kaylo dust received in January 1945.

b. Kaylo dust received in January 1949.

Both patterns contain lines which represent a moderate amount of chrysotile and of calcite and a small amount of quartz. Faint lines representing what is probably the hydrous calcium silicate component of Kaylo are also present in both patterns. In addition, the original film of pattern b contains a very weak line corresponding to amosite but the line is too faint to appear in the reproduced pattern.

The quartz content indicated by pattern a is less than 1 per cent and by pattern b, about 4 per cent.

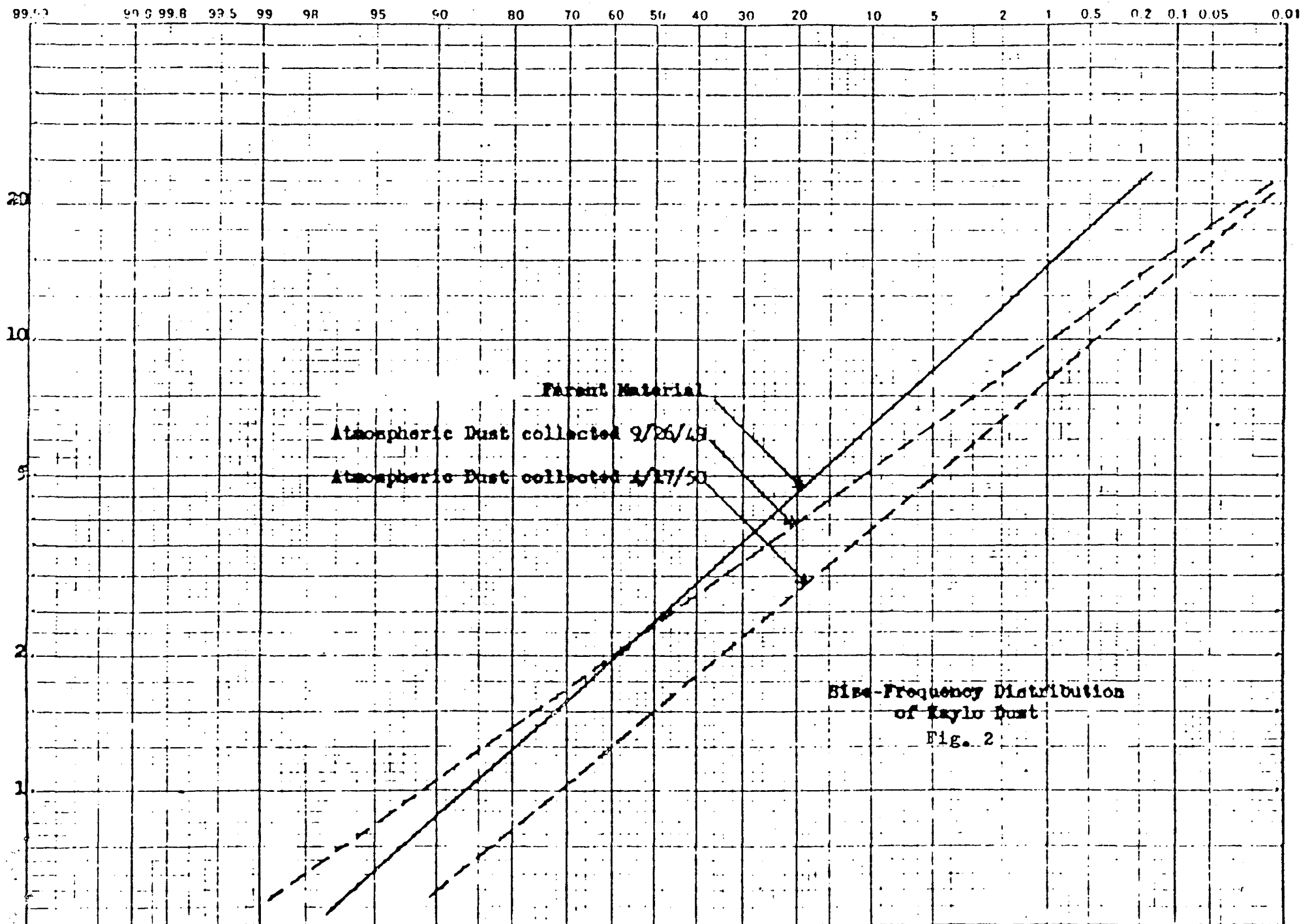




Fig. 3

Fig. 4



Fig. 5

Fig. 3. Lung tissue of a guinea pig exposed for 50 months to Kroylo dust by inhalation and then killed. A cross section of a bronchiole is shown just above and slightly to the left of center of the figure. The other spaces are alveoli. Note the dark masses within some of the smaller spaces, especially to the right of the bronchiole. These masses are macrophages and multinucleated giant cells. Note the thinness of the wall of the bronchiole; it is virtually normal, as is apparent on comparison with the tissue sections of a normal guinea pig (Fig. 5). An asbestosis body is present in this field and is revealed in Fig. 4 at higher magnification. (200 X)

Fig. 4. High magnification of the tissue section shown in Fig. 3. This higher magnification of part of the field of Fig. 3 reveals a typical form of asbestosis body, the dark curved elongated structure to the right of center. (1200 X)

Fig. 5. Lung tissue of a guinea pig exposed for 50 months to King's Blende asbestos dust by inhalation and then killed. The bronchiole in this photomicrograph is just above and to the left of center. Note the thickening of its wall. Strands of fibrous tissue are apparent within the wall of this bronchiole as well as in the adjacent alveolar walls. (200 X)



Fig. 6

Fig. 7

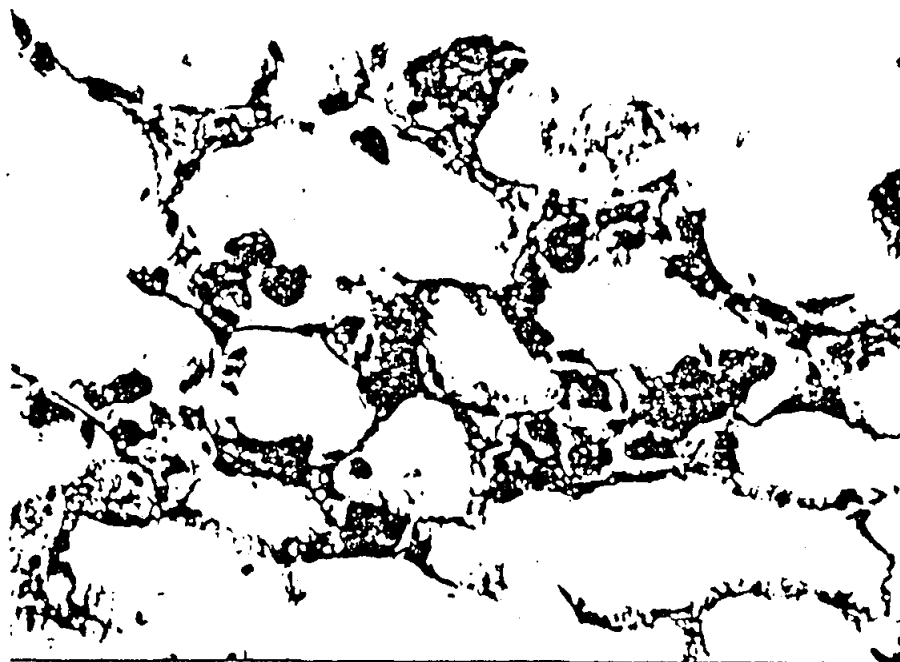


Fig. 8

Fig. 6. Lung tissue of a normal guinea pig. This field shows two bronchioles, one in cross section at the right of center and the other in longitudinal section. The small associated blood vessel is visible above the bronchiole in longitudinal section. Compare the thickness of the wall of these bronchioles with the thickness of wall of the bronchioles in figs. 5, 8, and 7. Note also the empty alveoli and the thin alveolar walls in the normal animal. (200 X)

Fig. 7. Lung tissue of a guinea pig exposed for 56 months to Kaylo dust by inhalation and then killed. In this photomicrograph a bronchiole is just above and to the right of center. Note the thickness of the tissue between the lumen of the bronchiole and the lumen of the adjacent alveoli in comparison with the thin bronchiolar walls in figs. 5 and 6. Note also that the greatly thickened bronchiolar walls of the animal exposed to asbestos dust for 30 months (fig. 5) are similar to those of this animal, exposed to Kaylo dust for 56 months. Fine strands of fibrous tissue are visible near the bronchiole in this photomicrograph but they are more apparent, because they are less dense, in the area at the right of the field where fibrosis has extended to adjacent alveolar walls. (200 X)

Fig. 8. Lung tissue of a rat exposed for 18 months to Kaylo dust by inhalation and then killed. The photomicrograph reveals simple accumulation of cells in alveoli. The cytoplasm of the cells is packed with particles of dust. Note that the alveolar septa, even those of spaces containing numerous dust cells, are thin and delicate. (200 X)



Fig. 9



Fig. 10



Fig. 11

Fig. 9. Lung tissue of a guinea pig infected with attenuated tubercle bacilli and immediately exposed to Kayle dust by inhalation for 24 months, and then killed. The figure is a slightly magnified photograph of the robbing histologic separation of the lungs, including both lungs seen open to base. The cluster of dark foci of irregular outline in the lower center (apical lobe) and at the right (left caudal lobe) are foci of active spreading tuberculous. The widely-distributed minute dark foci are merely lymphoid cell clusters and are unrelated to the infection. The extent of the tuberculous activity is seen to be exceedingly small. (1.5 X)

Fig. 10. Lung tissue of a guinea pig infected with attenuated tubercle bacilli and left in clean air for 2 months, then exposed for 12 months to Kayle dust by inhalation, and killed. The dark zone, irregular in outline, in the left upper quadrant (right cephalic lobe) is an area of active spreading tuberculous. A smaller but similar focus is visible at the lower margin of the left cephalic lobe. Again, as in Fig. 9, the extent of the tuberculous process is very small. (1.5 X)

Fig. 11. Lung tissue of a guinea pig exposed to Kayle dust by inhalation for 3 months, then infected with attenuated tubercle bacilli and exposed to the dust for an additional 16 months, and killed. The numerous poorly-defined dark zones, irregular in outline, are all sites of active, spreading tuberculous lesions. The tuberculous involvement exhibited in this photograph is the most extensive noted in any animal killed during the experiment. The involvement is relatively slight in comparison with the progression which occurs in animals exposed to quartz dust under similar conditions; in fact, all of the animals exposed to quartz dust would have died within 12 months after infection because of almost complete loss of functioning pulmonary tissue. (1.5 X)

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NINTH PROGRESS REPORT

ON

STUDIES OF KAYLO

905

TO

GENERAL-ILLINOIS GLASS COMPANY

C-57334

ROLL	81
CODE	71

May 16, 1947

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TABLES I TO VI

PLATES I TO III

Arthur D. Little, Inc.

CHEMISTS-ENGINEERS



CAMBRIDGE 42, MASSACHUSETTS

KLK:pg

May 16, 1947

Owens-Illinois Glass Co.
Toledo
Ohio

Attention: Mr. U. E. Bowes

Gentlemen: C-57334

We transmit herewith our NINTH PROGRESS REPORT
ON STUDIES OF KAYLO. This report covers the first phase
of our investigation on determining the possibility of
replacing part or all of the asbestos in Kaylo by paper
pulp.

While our previous investigations on the use
of paper pulp were exploratory in nature and concentrated
on the replacement of "spiculated" asbestos, our present
investigation takes into account the present plant prac-
tice and emphasizes replacement of asbestos in a manner
which would necessitate no change in plant procedure
and as little change in batch formulation as possible.

At this time it can be reported that from a
strength point of view Kraft pulp can be used as a
satisfactory substitute for Chrysotile asbestos, if some

I. INTRODUCTION

In previous work as summarized in our EIGHTH PROGRESS REPORT ON THE FUNDAMENTAL STUDY OF MICROGRITE, of March 27, 1946, we established the fact that paper pulp can be used successfully as a substitute for "spiculated" asbestos. The present asbestos shortage greatly increased the interest in this possibility from a practical point of view. Accordingly, the present study was initiated in order to investigate the possibilities of partial or total substitution of asbestos by paper pulp from a technological and economical point of view.

Since from a merely sales point of view there may be a resistance towards the inclusion of an organic material in Kaylo, it is assumed that the use of paper pulp is contemplated as a substitute only when and if the amount of suitable asbestos grades available are insufficient. Accordingly, it was endeavored to develop formulae for the incorporation of paper pulp which will not necessitate any change of the present plant procedure, as well as the least possible change in slurry characteristics and batch formulation.

It is believed that we have achieved this aim for Kraft pulp as a substitution material, even though we are fully aware of the fact that the final test will reside in pilot-plant tests.

In our investigations we have made some interesting observations with respect to the action of clays in Kaylo and we feel, that further study of these phenomena may show up that clays may serve as a suitable material to balance out property variations due to changes in the types and/or amounts of certain raw materials used, such as bestos, diatomaceous earth and quartz.

II. SUMMARY AND CONCLUSIONS

1. Kraft pulp can be used as a partial or total replacement for asbestos when:
 - a. introducing clay into the batch,
 - b. if hinging is not important,
 - c. whereby addition of Gum Karaya has proven to be helpful.
2. A brief economic survey of the availability of cellulosic pulp materials is recommended.
3. The significance of clay addition is discussed and it is felt that clay may be:
 - a. in reducing the asbestos amount in all asbestos mixtures,
 - b. in adjusting variations in raw materials.
4. A proposed future program for this project is outlined.

III. SUBSTITUTION OF ASBESTOS BY PAPER PULP

1. ECONOMIC ASPECTS

In our report of November 26, 1946 on "Availability of Asbestos" the market situation of asbestos has been discussed in some detail. While it is stated there that the shortage of spinning-grade asbestos may not be as severe as of some other grades, it was still felt that the general situation will be tight for years to come. Accordingly, the recommendations of this earlier report emphasized the desirability of a search for substitution products.

Our previous and present findings strongly indicate that certain paper pulps may furnish a suitable substitution material for part or all the asbestos. It is realized that paper stock of all kinds is not too readily available. However, the tonnage necessary for partial or total replacement of asbestos in Kayle is a much smaller percentage of the paper trade, so that local conditions and strong bidding could create a much more favorable position than in the asbestos market.

Still it is strongly recommended that a brief economic survey of the availability of cellulose pulp materials suitable for our purposes be undertaken as soon as possible, especially with respect to new Kraft clippings.

Kraft prices, while on a high level at present, compare favorably with asbestos prices and new Kraft clippings, as used in our present experiments, are quoted at only \$45.00 per ton. Baled, old newsprint is quoted at \$20.00 and wood pulp anywhere from \$60.00 to \$100.00 per ton.

2. THE FUNCTION OF ASBESTOS

While originally a strong emphasis was put on the suspending properties of asbestos, present practice has moved away from this concept and primarily considers its functions as a reinforcing agent.

By and large it is now generally accepted that "spiculated" asbestos is not necessary for the successful preparation of Kaylo and asbestos as used in present batch formulation can be hardly designated as "spiculated." A sample of asbestos slurry provided by Berlin had a freeness of fifty, while "spiculated" asbestos would correspond to a freeness of about 70 or more. The degree of opening up presently used provides satisfactory strength and hinging properties.

When the concept of asbestos as a suspending agent still was emphasized, the "gelatinous" properties of "spiculated" Chrysotile were frequently thought of as a contributing factor. However, even from this point of view it was quite conclusively found that blue African

and Amosite asbestos which do not show any "gelatinous" properties are just as good, if not better than those of Chrysotile. They are possibly somewhat inferior with respect to hinging (see THIRD PROGRESS REPORT ON INVESTIGATION OF EFFECTS OF ADDITIONS OF ASBESTOS TO MICROCRITE, August 16, 1943, C-57316). This has been somewhat borne out by actual plant experience with Amosite in the Berlin pilot plant.

Since, as we understand, 10 lbs./cu.ft. products for boiler lagging and pipe covers will be of secondary importance in the planned large-scale production, while 20 lbs./cu.ft. floor scores and roof slabs will be of primary importance, it is believed that hinging properties will be of less interest than in the earlier investigations. Furthermore, reinforcement and suspending power are probably less critical in the 20 lb. product.

Accordingly, it is believed that it should be quite possible to use various grades of asbestos interchangeably without materially affecting the quality of production. As will be discussed later, appropriate utilization of minor amounts of clay might prove helpful in adjusting the batch properties in changing over from one type of asbestos to another.

Minor additions of clay might also permit a reduction in the total amount of asbestos used and thus help to conserve stocks.

3. SUBSTITUTION OF ASBESTOS BY CELLULOSE PULP

As will be discussed in detail in the following section, the first phase of our investigation has proven quite conclusively that from a strength point of view asbestos can be successfully replaced by paper pulp, whereby our experiments thus far were limited to Kraft pulp as made from new Kraft clippings.

In preparing a batch of similar consistency as now used in actual production and in obtaining satisfactory strength:

- (1) the use of about 10% clay addition (whereby the SiO_2 of the clay is substituted for SiO_2 of the basic batch) was found desirable for successful total substitution of asbestos by paper pulp and,
- (2) the addition of Gum Karaya for dispersion of the fiber was found to be quite helpful and is included in our preferred batch formulation.

Batches made with paper or even paper and asbestos mixtures are not satisfactory with respect to ringing however.

Typical batch compositions are given in Table VI under 3a and 3b.

Our present experience appears to indicate that for partial replacement of asbestos the amount of clay to be added may be a function of the degree of reduction in asbestos addition.

In preparing paper pulps we used a paper beater, but it is assumed that any other method of pulping, whether dry or wet, could be used with equal success.

4. FUTURE PROGRAM

Now that the usefulness of paper pulp as an asbestos substitute has been established and in view of our other observations, we recommend continuation of our program as follows:

- (a) Preparation of samples of our best present formula and of standard composition for comparative tests with respect to the following properties:
 - (1) fire resistance
 - (2) strength vs. heat treatment up to 650°C.
 - (3) strength vs. humidity.
- (b) Addition of clay vs. varying substitution of asbestos in the base formula.
- (c) Exploration of other cellulose pulps in place of Kraft:
 - (1) Newsprint
 - (2) Wood pulp
 - (3) Other available paper pulps.
- (d) Reduction of asbestos in present formulae without substitution vs. addition of clay.
- (e) Consideration of paper pulping machines other than in the Valley paper beater.

IV. HYDROLYSAL PINT

1. PREPARATION OF SAMPLES

In the preparation of samples it was endeavored to duplicate as much as possible plant conditions at Berlin.

The basic batch formula is given in Table VI under 1, and correspond to the basic formula as used in Berlin. Sliced lime, as used in Berlin, however, was replaced by Warner Limb Hydrate, since slaking is believed to be undesirable for laboratory purposes both with respect to handling and reproducibility of results.

At the recommendation of Dr. Emma, a box-type mixer was used as it was believed to reproduce mixing conditions at Berlin better than the Sigma mixer previously used in this laboratory. The box-type mixer is shown in Plate I. Mixing time was one half hour.

It was decided to omit the prehardening step in our experiments, since this is believed to not seriously change the validity of experimental and would introduce another factor complicating reproducibility of results.

The indurating cycle was adjusted according to recommendations by Berlin and the entire cycle, including the blow off rate, is regulated by a program controller and the temperature cycle was recorded as shown in a

typical record chart in Plate II. The autoclave, boiler, program controller, and temperature recorder are shown in Plate III.

The samples were dried as shown in the various tables and conditioned in the laboratory for 24 hours before testing.

The asbestos and paper were beaten on a standard paper beater and the degree of beating checked by the standard freeness test.

2. EVALUATION OF DATA

Close approximation of plant procedure on a laboratory scale introduces considerable difficulties with respect to reproducibility of data. The short mixing time in particular appears to be objectionable in this respect.

Nevertheless, it was decided to adhere to the above outlined procedure rather than to adopt the methods used in the Toledo "Materials Laboratory," since it was believed that in this manner results were more directly applicable to plant procedure.

Ten pound density was chosen for most of our experiments, since it was assumed that differences between paper pulps and asbestos would become more noticeable in the lighter product.

14
65

In order to obviate some of the day to day deviations comparison samples of the standard plant composition were included in all runs, unless comparative values of variations in paper pulp batches were to be investigated only.

In evaluating results average trends were considered rather than comparison of individual batches. In this manner it is felt that, in spite of considerable variation in results from run to run, the findings are quite reliably established.

All strength values were reduced to a theoretical density of 10 lbs./cu.ft. by assuming straight line relationship between strength and density, which appears to be satisfactory for comparison purposes, since densities of the samples varied between 11 lbs./cu.ft. and 12.5 lbs./cu.ft. only.

3. FIRST SERIES, ADDITION OF KRAFT

A freeness of 80 was chosen as the standard freeness for Kraft paper pulp, even though 50 would probably be sufficient. Since the raw material used is new Kraft clippings, it was thought that a higher freeness would warrant more uniformity. If it should become desirable, however, tests with 50 freeness pulps will be carried out.

Table I summarizes results in this series of experiments. On the average the Kraft mixtures (12% Kraft) were 18% lower in strength than the asbestos samples.

Previous experiments had indicated that Kraft in additions of not more than 4% is most beneficial for reinforcing purposes. Such mixtures, however, are too thin for plant practice. Samples A-41 to A-45 in Table I appear to indicate that 10 to 12% Kraft gives better strength than either 8 or 14%. Since, considering the difference in specific gravity, 12% Kraft pulp should have about the same volume as 20% asbestos, and since 12% Kraft gives about the same consistency as 20% asbestos, 12% Kraft addition was chosen as the standard

for all our comparison tests.

The various Kraft asbestos mixtures shown in the Table appear to indicate that the mixtures result in as low values as the all-Kraft pulp samples.

Improved mixing in the Simpson mixer seems to benefit asbestos more than paper pulp (possibly because of the greater freeness chosen), while longer autoclaving apparently benefited them equally well. However, the results shown in Table II, make it apparent that the imduration time influence rather insignificant and does not affect the relative results of this investigation.

Arthur D. Little, Inc.

In connection with this series the effects of Xerocene and Foamex as dispersing agents were tried. While there was some indication that these additions have a slightly beneficial effect, results were not significant enough to warrant further investigation.

4. SECOND SERIES, INFLUENCE OF DRYING TEMPERATURE

The inconsistency of results, and the known properties of cellulosic materials suggested a brief investigation of the influence of drying procedure as summarized in Table II.

The variations in drying procedure did not show any significant results for the Kraft samples and the average difference between asbestos and Kraft samples was even greater than in the first series.

5. THIRD SERIES, MISCELLANEOUS ADDITIONS

Observations of the batch mixtures appeared to indicate that paper pulp has a tendency to agglomerate during mixing in the concentrations used in our batches. The relatively good results previously obtained with batches containing 4% Kraft where this effect would be minimized appear to bear out this conclusion.

Various unsuccessful approaches to obviate this are summarized in Table III.

6. FOURTH SERIES, ADDITION OF GUM KARAYA

Experience in the manufacture of stencil sheets has shown that deacetylated Gum Karaya is outstanding in the dispersion of long-fiber paper pulps. Accordingly, it was thought that it may be beneficial in a similar manner in the preparation of cellulosic pulp Kayle batches.

The use of deacetylated Gum Karaya is patented and the patents are held by the John A. Manning Co., which had been assisted in this development by our Company. It is assumed that, if desirable, satisfactory license arrangements could be obtained. The price of Gum Karaya is from about 30 to 50¢ per pound. Two percent deacetylated Gum Karaya was used in most of our experiments; possibly higher additions may bring about some improvement.

The results of this series are summarized in Table IV. Deacetylated Gum Karaya obviously bring about some improvement and the average figures are given in the last lines of the Table. Especially comparison with the averages from the second and fourth series point out the marked improvement obtained. Nevertheless, the results are still consistently lower than those obtained with asbestos.

strength

17

It should be noted, however, that the two batches using mixtures of 6% asbestos plus 6% Kraft (A-130, A-135) give equally as good results as 20% asbestos alone.

7. FIFTH SERIES, ADDITION OF DIXIE CLAY

Previous experience with the beneficial effect of clay additions suggested the use of clay for the improvement of cellulose pulp batches. The results as summarized in Table V show the highly beneficial effect of this addition. The batch compositions used in this series are given in Table VI, 3.

Samples A-134 through A-137 illustrate a phenomenon, observed several times previously, which is not quite understood and could not be further investigated within the scope of this project. Certain batches seem to improve markedly on redrying of the samples to 120°C. for about one half to one hour.

While this series clearly demonstrates the beneficial action of clay on Kraft batches, it is incomplete in many respects. It fails to show whether Gum Karaya is beneficial in conjunction with clay. It also does not show sufficient variations in clay content and basic formula variation, both with respect to all-Kraft and to mixed Kraft-asbestos batches.

V. THE CLAY PROBLEM

That clay additions may have a beneficial effect on Kaylo has been observed from time to time, both in Berlin and in our laboratories.

In our EIGHTH PROGRESS REPORT (page 12) it was stated that the amount of clay which can be introduced to advantage appears to be dependent on the type of asbestos used and on the amount of asbestos dust introduced by it. We received information from Berlin that clay addition was beneficial with certain shipments of Dicalite. Five percent clay was used as a standard addition in most of our previous cellulosic pulp batches so that no comparison data were available. Aluminum hydroxide, clay, and Portland cement were found beneficial in previous experiments in our laboratories. Most of this previous work was made with Celite, which is purer than Dicalite, and usually with asbestos additions of less than 15%.

In twenty pound materials we have been able to introduce considerably higher amounts of clay obtaining very strong bodies without excessive shrinkage.

The present investigation again highlights the fact that clay can be highly beneficial under certain circumstances. The scant results seem to indicate (see Table V in conjunction with Table VI), when comparing

batch A-133 and A-135 with batch A-134, that introduction of clay by substituting its silica content for silica of the base composition is preferable to substituting total clay for silica.

The evidence thus far collected seems to indicate that asbestos and clay (or aluminum hydroxide?) have similar action on the culspore formation. Possibly we deal with absorption reactions which might have a retarding effect on the lime. In any case, the effect of aluminum compounds and its possible similarity to the chemico-physical action of asbestos appear to be of sufficient importance to warrant more systematic exploration.

In this respect cellulosic pulp batches lend themselves well for research, since the possible influence of asbestos, and variations thereof from batch to batch can be eliminated *rep 15*.

The following variables appear to be of immediate interest:

- (a) Variation of clay content with a given C/M ratio ($M = \text{silica raw material} + \text{SiO}_2 \text{ in clay}$).
- (b) Variation of C/M ratio for a given clay content.
- (c) Influence of type of aluminum raw material, such as hydrated alumina, Kaolin, different types of ball clays.

In addition thereto, the influence of asbestos from a chemical point of view should be studied by using asbestos dust in a similar manner as outlined for clay under (a) and (b) above.

From a practical point of view the following interesting implications can be foreseen:

- (1) Asbestos content could be radically reduced when introducing clay in a balanced manner.
- (2) Clay could be used as a compensating factor for lot to lot variations of asbestos and diatomaceous earth shipments.
- (3) Clay might allow reduction of diatomaceous earth content.
- (4) Clay could influence the chemical stability of Kaylo.

TABLE I*

First Series

Kraft Pulp Addition, Dried at 110°C.SampleLbs./sq.in.

Average 20% Asbestos

54

Average 12% Kraft

56

Variation of Kraft Addition

A-41 6% Kraft

48

A-42 8%

53

A-43 10%

52

A-44 12%

52

A-45 14%

49

Kraft Asbestos Mixtures

A-50 20% Asbestos

70

A-44 4% Kraft + 10% Asbestos

64

A-47 10% Kraft + 8% Asbestos

54

A-46 6% Kraft + 4% Asbestos

47

A-66** 20% Asbestos

66

A-70 7% Kraft

48

A-66 6% Kraft beaten with 6% asbestos
to 65 fineness

53

A-67 6% Kraft + 1% Asbestos

50

Mixing Procedure and Autoclaving5 Hours Autoclave7 Hours AutoclaveSimpsonBoxSimpsonBox

20% Asbestos

65

58

77

65

12% Kraft

48

46

53

47

12% Kraft

48*

*(twofold mixing time)

* Basic batch compositions - see Table VI.

** In this and the following tables separate runs are separated by a solid line.

TABLE XI
Second Series
Influence of Drying Temperature

Sample No.	Composition	Heat Treatment	Lbs./sq. in.	Avg.
A-85-A	20% Asbestos	105°C.	63	64
A-85-A			67	
A-85-B			59	
A-86-B			67	
A-82-A	12% Kraft	105°C.	45	50
A-81-A			47	
A-82-B		150°C.	55	
A-81-B			53	
A-83-B	"	51		
A-83-C	"	105°C. up to 150°C.	53	
A-86-A*	20% Asbestos	160°C.	75	70
A-87-A*			65	
A-86-B*	"	room temp. to 160°C.	50	
A-87-B*	"		54	
A-83-A*	12% Kraft	160°C.	50	53
A-83-A*			56	
A-83-A*		"	52	
A-83-B*		"	52	
A-83-B*	"	room temp. to 160°C.	52	
A-89-B*	"	" " " "	50	
A-107	20% Asbestos	120°C.	85	
A-108	12% Kraft	"	44	
A-95-D	12% Kraft	160°C. to 105 moisture, conditioned.	44	
A-97-A	"	160°C. to 105 moisture, conditioned.	49	
A-96-B	"	160°C., tested immediately	40	
A-97-B	"	160°C., tested immediately	47	
A-100-B	"	160°C., tested immediately	58	
A-100-C	"	160°C.-105°C., tested immediately	52	
A-98-C	"	160°C.-105°C., tested immediately	61	
A-99-C	"	160°C.-105°C., tested immediately	48	
A-98-D	"	like C, but conditioned	43	
A-99-D	"	"	59	

* 7 hours induration

TABLE III

Third Series

Miscellaneous Additions, Dried at 120°C.

<u>Sample</u>	<u>Composition</u>	<u>lbs./ sq.in.</u>
A-110	20% Asbestos	79
A-111	12% Kraft increased Jasper X	53
A-112		59
A-115	20% Asbestos	72
A-113	12% Kraft + Methocel for dispersion	46
A-116		52
A-116	12% Kraft .5% C/M ratio	52
A-117	12% Kraft .65 C/M ratio	52
A-119	20% Asbestos	64
A-121	12% Kraft	57
A-120	12% Kraft beaten with lime	53
A-122	12% Kraft beaten to 50 frequencies	54
	+ Methocel	
A-118	12% Kraft + oxalic acid	56

why? precipitate formed as Carbazate

TABLE IV

Fourth Series

Gum Karaya Addition, Dried at 120°C.

Sample	Composition	lbs./ sq. in.	
A-110	20% Asbestos	79	
A-112-A	12% Kraft + Gum Karaya	68	
A-123	20% Asbestos	53	
A-125	12% Kraft + Gum Karaya	55	
A-126	12% Kraft + Gum Karaya + Kerosene	62	
A-132	20% Asbestos	70	
A-129	12% Kraft + Gum Karaya	62	
A-130	6% Asbestos + 6% Kraft + Gum Karaya	70	
(A-131	10% Kraft + Gum Karaya, 20 lbs./ cu. ft.	170)	
A-135-A	20% Asbestos	61	
A-133-A	12% Kraft + Gum Karaya	55	
A-134-A	12% Kraft + Gum Karaya + Kerosene	46	
A-136-A	12% Kraft	48	
A-141	20% Asbestos	74	
A-144	12% Kraft + Gum Karaya + Kerosene	64	
A-145	6% Asbestos + 6% Kraft + Gum Karaya	79	
			Second and Fourth Series
Average	20% Asbestos	65	66
Average	12% Kraft	53	51
Difference		-18%	-23%
Average	20% Asbestos	67	
Average	12% Kraft + Gum Karaya	59	
Difference		-11%	

TABLE V

Fifth Series

Addition of Dixie Clay, Dried at 120°C.

Lbs./
sq.in.

Sample

Composition

79

A-110 20% Asbestos
A-108 12% Kraft, .6 C/A ratio,
6% clay

79

A-109 12% Kraft, .6 C/A ratio,
6% clay

67

58

A-123 20% Asbestos
A-124 20% Asbestos, .6 C/A ratio,
6% clay

58

A-127 12% Kraft, .6 C/A ratio,
6% clay

62

Asbestos
Revised

77

A-137 20% Asbestos
A-133 12% Kraft, .6 C/A ratio,
6% clay, Gun Koroys

92

A-134 12% Kraft, .67 C/A ratio,
10% clay, Gun Koroys

74

A-135 12% Kraft, .6 C/A ratio,
10% clay, Gun Koroys

82

A-136 12% Kraft, .6 C/A ratio,
25% cla., Gun Koroys

97

48

A-141	20% Asbestos	72
A-142	12% Kraft, .6 C/M ratio, 6% clay, Gum Laraya	84
A-144	12% Kraft, Gum Laraya	84
A-145	6% Asbestos + 6% Kraft, Gum Laraya	79
A-146	6% Asbestos, .6 C/M ratio, 6% clay, Gum Laraya	81

Average	20% Asbestos	72
Average	12% Kraft, .6 C/M ratio, 6% clay	73