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FLUORINE COMPOUNDS USEFUL IN THE CONTROL OF INSECTS

By

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Laboratory apparatus for determining the relative effectiveness of various insecticides on roaches.

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CONTENTS

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INTRODUCTION

Attention was called by the senior author in 1924 to the insecticidal properties of the fluorine compounds against crop insects. Since then a marked development in the use of sodium fluosilicate and cryolite against insects has taken place. Among the inorganics, the fluorine compounds have proved to be the best available arsenical substitutes. Much has been learned concerning their toxicity, plant tolerance, effect on higher animals, and specific uses against various crop pests. As several of the early publications of the Tennessee Station on fluorine compounds are out of print, it is the purpose of this bulletin to bring together the major results of the experimental work since 1924 with the various fluorine compounds, with special reference to sodium fluoride, sodium fluosilicate, and cryolite.

HISTORICAL

Perhaps the earliest note on the insecticidal properties of fluorine compounds occurs in British patent No. 8236, granted to C. H. Higbee in 1896 (29), entitled "An improved composition or material for destroying insects." Higbee considered fluorine compounds to be effective against such insects as the Colorado potato beetle and roaches. For roaches, finely pulverized sodium fluoride and sodium silico fluoride were advocated.

Nineteen years elapsed before the value of sodium fluoride was more fully recognized. In 1915, it was recommended by Marlatt (55) as the most simple means of ridding premises of roaches. In the next few years other workers (5) established the value of sodium fluoride for the control of ants and chicken lice and as a wood preservative against termites.

A severe outbreak of the cotton worm and cotton boll weevil resulted in an acute shortage of arsenic in 1923. This situation clearly showed the need for an arsenical substitute and stimulated the search for new insecticides. In 1924, Marcovitch (40, 41) reported that sodium fluosilicate and cryolite were capable of destroying the cotton boll weevil and the Mexican bean beetle, opening up a new field for fluorine insecticides against insects affecting field crops. Favorable results were obtained also against the potato beetle, flea beetles, cucumber beetles, and hornworms. A large number of other fluorine compounds have been tested since, but cryolite has proved the most valuable, primarily because of its safety for foliage. In 1926, the value of sodium fluosilicate in poison baits for cutworms and grasshoppers was reported (42). Further data were obtained in 1928 on the value of sodium fluosilicate in baits (43).

In 1931, Newcomer (62) presented evidence that either cryolite or barium fluosilicate was equal to lead arsenate in the control of the codling moth in the Pacific Northwest. Large amounts of cryolite are employed now on apples in the state of Washington.

The development of fluorine insecticides was hindered by the lack of a good method of analysis. In 1933, Willard and Winter (94) announced a distillation method that was sensitive and accurate enough for the determination of spray residues. Thorium nitrate is used for titrating this fluorine.

SODIUM FLUORIDE

Properties.—Sodium fluoride is a stable neutral salt of the element fluorine. It is readily soluble, 1 gram dissolving in 25 cubic centimeters of water at 18°C. It does not occur free in nature, but is made from calcium fluoride, or fluorspar, as follows:

$$CaF_2 + H_2SO_4 \rightarrow CaSO_4 + 2HF$$

The resulting hydrogen fluoride is volatile, and tests conducted by Marcovitch (54) show that it is toxic to insects and has possibilities as a fumigant. The resulting hydrofluoric acid is neutralized with sodium carbonate as follows:

$$2HF + Na_2CO_3 = 2NaF + H_2O + CO_2$$

Uses.—Sodium fluoride finds its greatest use in the control of household insects, such as roaches and ants, and chicken lice. Its tendency to burn foliage rules it out for use on crops. Sodium fluoride may find a place, however, as an herbicide for the control of lawn weeds (52). It is used also in making enamelware and as a wood preservative.

Toxicity.—Sodium fluoride, being very soluble, is poisonous to man. Care should be taken to have all containers plainly labeled so as to keep it out of food supplies. Numerous fatalities have resulted from its being swallowed by mistake (39, 77, 78). In 1940, eleven men died from eating pancakes made with sodium fluoride mistaken for baking powder. From 5 to 10 grams, or 1 to 2 teaspoonfuls, generally is considered to be a fatal dose for humans. A roach powder, to be foolproof, should contain as small an amount of sodium fluoride as possible, preferably not over 10 percent.

Antidotes.—Sodium fluoride appears to act mainly by inactivating calcium through the formation of the insoluble calcium fluoride. Any form of calcium, therefore, is an antidote. Recent work shows that aluminum sulfate may act more effectively as an antidote through the formation of cryolite, which is relatively non-toxic to higher animals (53).

INSECTS CONTROLLED BY SODIUM FLUORIDE

COCKROACHES

Cockroaches were largely responsible for the first use of fluorine compounds as insecticides in 1896 (29). Sodium fluoride did not come into general use until 1915. Since then it has become the standard remedy for roaches, especially the German roach (*Blattela germanica*).

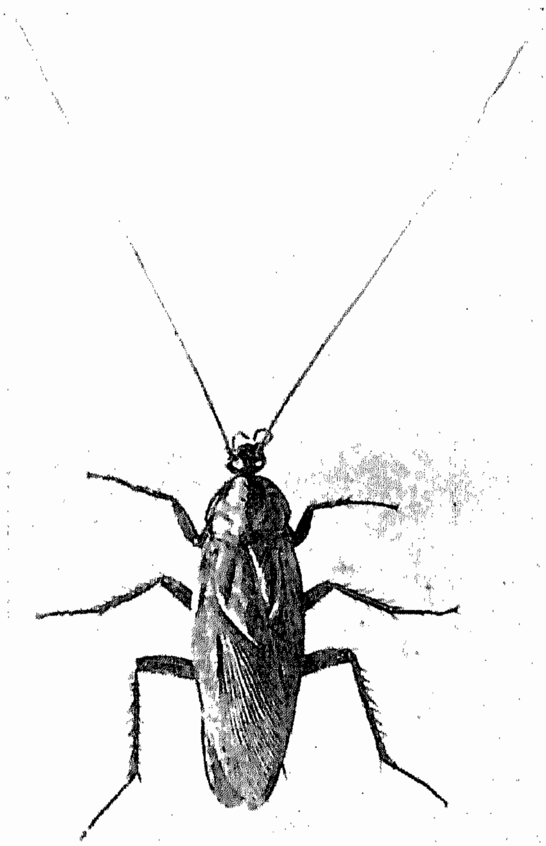

Fig. 1—The American cockroach. Natural size.

Because roaches have the habit of cleaning themselves, it was generally thought that some of the powder is swallowed and thus acts as a stomach poison (75). Recent work by Sweetman and Laudani (90) and others indicates that sodium fluoride acts primarily as a contact insecticide, and when a combination of sodium fluoride and pyrethrum is used, the contact effect greatly predominates. This is due to the fact that the pyrethrum produces paralysis so rapidly that the roach is unable to clean itself, and thus the chances of swallowing the powder are greatly reduced. The combination of pyrethrum and sodium fluoride is especially effective because they act synergistically. A multifold effect is produced when the two materials are combined. For example, 100 mg. of pyrethrum blown into testing chamber will kill 4 percent of female roaches in 24 hours; the same amount of sodium fluoride will kill 12 per cent; whereas 50 mg. of each combined gave a kill of 64 percent in 24 hours.

The pyrethrum produces a rapid paralysis, so that the roach is unable to clean itself of the offending powder. In addition, it causes the roach to salivate, with moist droplets appearing over the abdomen and legs. The moisture thus produced appears to dissolve the fluoride, which then penetrates the chitin and causes death. The combination of sodium fluoride and pyrethrum will give a rapid and desirable effect—one being water-soluble and entering through the pore canals of the chitin, and the other being fat-soluble and entering through fatty tissue.

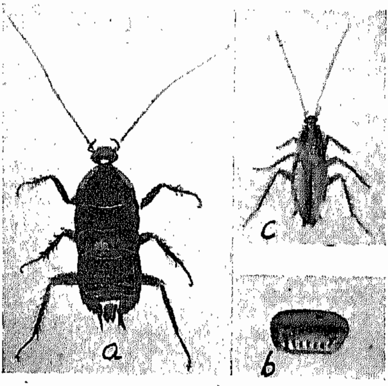

Fig. 2.—a. Oriental roach; b. egg pod of oriental roach. c. German roach. Natural Size.

For roach control, sodium fluoride is much more effective than either sodium fluosilicate or cryolite. Many pest-control operators use a combination of 75 percent sodium fluoride and 25 percent pyrethrum.

Since sodium fluoride is poisonous, a large number of diluents were tried to reduce the fluorine content. The outstanding material discovered is an acid dextrin. The following formula[1] was adopted finally as most effective:

Sodium fluoride	10	percent
Pyrethrum	10	percent
Dextrin	80	percent

For best results, this mixture should be used as a contact insecticide and applied with a small duster or bellows. An electric power duster with an extension rod gives good results. One should aim

[1]Patent pending.

to blow the powder into the roaches' hiding places and to hit as many as possible with the powder. No cracks or holes in the kitchen, about the sink or elsewhere, should be missed. The pyrethrum excites the roaches and causes them to come out of hiding. In this way they show which places are most severely infested.

Fig. 3—The handy type of duster used in roach control.

One thorough treatment is sufficient to clean up an infestation of roaches, and assure freedom from their depredations for a year or longer. Where they reappear, a second treatment should be applied.

POULTRY LICE

Since the publication, in 1917, of Bishopp and Wood's bulletin (5) on the use of sodium fluoride for lice on poultry, this material has become a standard remedy. It is best applied in the dust form to various parts of the body by the so-called "pinch" method. The fowl may be held and a pinch of the chemical applied next to the skin on the back, another pinch in the fluff around the vent, one under each wing, and one on the breast, the head, and the neck. If care is taken to make the application thorough, 100-percent control may be obtained within 4 days, and the fowls should remain free of lice for at least a year. A pound of the powder is enough to treat 100 birds.

In summertime the dipping method may be used. Three-fourths to 1 ounce of the sodium fluoride is dissolved in 1 gallon of warm water. A tub filled to within 6 inches of the top is suitable. The fowls should be dipped in the solution from 20 to 30 seconds. The head is left out at first, then just before the bird is released the head is ducked once.

CATTLE LICE

Cattle are attacked by several species of lice, such as the short-nosed cattle louse (*Haematopinus eurysternus*) and the long-nosed cattle louse (*Linognathus vituli*). Tests made against the latter species with the above roach powder, containing 10 percent sodium fluoride, 10 percent pyrethrum, and 80 percent dextrin, gave excellent results.

FIREBRAT

A dust of sodium fluoride was recommended for the firebrat (*Thermobia domestica*) as early as 1915 (56), while Wakeland and Waters (92) showed that a bait also was effective. Later a sodium fluoride bait was found more rapid in its action than arsenic trioxide (83). Seiferle et al. (73) tested various fluorine compounds and determined that sodium fluoride is more effective than either sodium fluosilicate or cryolite.

According to Snipes et al. (83), a satisfactory bait may be made up as follows:

Sodium fluoride	7	parts	by	weight
Ground rolled oats	86	"	"	"
Powdered sugar	5	"	"	"
Salt	2	"	"	"

All the ingredients should be thoroughly ground and mixed. The bait is placed in shallow cardboard boxes, a teaspoonful each, and the boxes placed where the insects are likely to find them.

SILVERFISH

Since the silverfish (*Lepisma saccharina*) craves starch, it may be controlled by a bait consisting of 8 parts wheat flour and 1 part sodium fluoride (3). The mixture should be scattered about shelves, drawers, cracks and other places frequented by this insect.

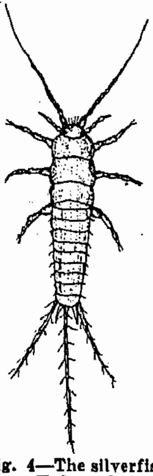

Fig. 4—The silverfish. Enlarged.

SODIUM FLUOSILICATE

Properties.—Sodium fluosilicate, or "silicofluoride", is a white, granular, non-volatile, odorless powder with an acid reaction. At room temperature it dissolves in water at the rate of 1 part to 154. It is insoluble in the common organic solvents.

In water, hydrolysis is thought to take place according to the following equation:

$$Na_2SiF_6 + 2H_2O = 2NaF + 4HF + SiO_2$$

Being an acid salt, it may be titrated with sodium hydroxide as follows:

$$Na_2SiF_6 + 4NaOH = 6NaF + Si(OH)_4$$

Twenty-one cubic centimeters of normal NaOH, or nearly 1 gram of the solid, is required to neutralize 1 gram of sodium fluosilicate. According to the above equation, 1000 cc. of normal NaOH is equivalent to 47.07 grams of Na_2SiF_6.

A saturated solution of sodium fluosilicate in water shows a pH of 3.6. At a dilution of 1-2000, further hydrolysis takes place, with a rise in acidity to a pH of 3. Upon dilution to 1-20,000 the pH remains at 3, indicating a high degree of buffering. Even at the high dilution of 1-80,000, considerable acidity is indicated, with a pH of 3.8.

Source.—Sodium fluosilicate is obtained as a by-product in the manufacture of phosphate fertilizer. Phosphate rock is made up largely of a double compound of calcium phosphate and calcium fluoride. In the manufacture of superphosphate by the acid method, the following reactions take place:

$$CaF_2 + H_2SO_4 = 2HF + CaSO_4$$
$$4HF + SiO_2 = SiF_4 + 2H_2O$$
$$3SiF_4 + 2H_2O = 2H_2SiF_6 + SiO_2$$

Formerly the fumes of silicon tetrafluoride were allowed to escape. Now this is prohibited because of injury to surrounding vegetation. Some of the manufacturers recover the fluorine gases and make the sodium salt as follows:

$$H_2SiF_6 + 2NaCl = Na_2SiF_6 + 2HCl$$

Over 2,000,000 tons of phosphate rock are mined annually in the United States. The average phosphate rock contains about 3 percent fluorine, so that it is possible to recover over 50,000 tons of sodium fluosilicate. At the present time, however, less than 5 percent is recovered. Before the war several million pounds were imported, mostly from Denmark and Holland. With imports shut off, a larger amount is being produced in this country. According to the Department of Commerce, about 5,000,000 pounds were consumed in 1925, of which about 2,000,000 pounds were used in laundries as a sour and 3,000,000 pounds in the ceramic and other trades. In 1937 about 5600 tons were produced in this country. As the insecticidal properties of sodium fluosilicate become better known, it is expected that large amounts will be required. In the future, the insecticide industry may provide the greatest outlet for this material.

TOXICITY TO INSECTS

Sodium fluosilicate was the first of the fluorine compounds to be used against crop insects, such as the Mexican bean beetle. It is much safer on foliage than sodium fluoride, but it is not foolproof, and has been replaced by cryolite. Sodium fluosilicate now is used also as a mothproofing agent. Minaeff and Wright (57) found that wool possesses a strong affinity for the fluosilicate, somewhat akin to that of a dye.

Because of its acid reaction, sodium fluosilicate shows to best advantage when dissolved in water. The larvae of the *Culicidae* are easily raised and are well adapted for testing the toxicity of solutions. At .01 molar (1-532) sodium fluosilicate will produce mortality in one hour and is more toxic than sodium arsenate. Even as low a concentration as 1-80,000 is fatal to mosquito larvae of *Culex quinquefasciatus* after 24 hours (43). In water, sodium fluosilicate is about ten times as toxic as sodium fluoride to mosquito larvae. This probably is due to the HF liberated by hydrolysis.

Against roaches, sodium fluoride is more effective. This is due to the fact that the sodium fluosilicate possesses a complex ion, SiF_6, in which the fluorine is tied up with few fluorine ions available in the absence of moisture. Against roaches, sodium fluosilicate is used in the dry form and consequently will not give as good control as the sodium fluoride through contact action. Formerly it was thought that a roach in cleaning itself swallowed some of the poison, but recent work shows that the contact action predominates (90).

When the fluosilicate is swallowed, it usually is more toxic than any of the fluorides, since it contains 60 percent fluorine. For this reason the sodium fluosilicate finds its greatest uses in the form of poison baits for such insects as cutworms, grasshoppers, crickets, and various weevils.

In 1928, the red-legged grasshopper (*Melanoplus femurrubrum*) was used to determine the toxicity of baits against grasshoppers (43), as shown in table 1.

Table 1—*Summary of tests of poison baits with grasshoppers, at Knoxville,* 1928.

Material	Strength of bait	Percent dead	
		24 hrs.	48 hrs.
Sodium fluosilicate	1-56	41	100
Sodium arsenite	1-56	38	89
Paris green	1-50	50	84
Arsenic trioxide	1-50	40	100

The figures indicate that sodium fluosilicate is somewhat more toxic than sodium arsenite or paris green. In Colorado, Langford (36) found sodium fluosilicate more toxic than sodium fluoride or the arsenicals.

The early work with baits laid the foundation for further experimentation, and at the present time it appears that sodium fluosilicate may be the most extensively used insecticide for baits against Mormon cricket and grasshoppers. The following is taken from correspondence with Dr. J. R. Parker, of the Bureau of Entomology and Plant Quarantine:

"Through our experimental work we have developed a bait which has proven very successful against Mormon crickets. This consists of either pure bran or a mixture of one part millrun bran and three parts sawdust with four pounds of sodium fluosilicate to 100 pounds of carrier as the killing agent. The failure of sodium arsenite baits against Mormon crickets is apparently due to the fact that even small quantities of sodium arsenite are decidedly repellent to crickets. This bait has been used very successfully in actual control programs this year and where grasshoppers were present has given good control of both insects. Our baiting work with grasshoppers has also indicated that sodium fluosilicate baits give as high kills as arsenical baits and in most cases kill more quickly. Scattered general observations point to the fact that baits made with sodium fluosilicate are less toxic to livestock than arsenical baits, but we have no definite information on what the relative toxicity may be."

Two hundred and forty thousand pounds of sodium fluosilicate was used in Canada as a grasshopper bait in 1938, and nearly that much against the Mormon cricket in the United States in 1941. As sodium fluosilicate is a by-product, it is subject to considerable fluctuation both in price and availability. In 1939, prices of less than 4 cents a pound were quoted for the material in carlots. With imports shut off by the war, the price jumped to 15 cents. Its high price and scarcity hinder the extension of the use of the fluosilicate in baits for the time being.

Had sodium fluosilicate been available in quantity in 1939, the grasshopper campaign would have required 3,000 tons, together with 180,000 tons of bait.

INSECTS CONTROLLED BY SODIUM FLUOSILICATE

GRASSHOPPERS

Since 1926, extensive experiments by several workers have established the effectiveness of sodium fluosilicate in baits for the control of grasshoppers (25, 79). Its principal advantages over arsenic are that it is (a) less repellent to insects, (b) less toxic to livestock and higher animals, and (c) more toxic to insects.

According to Shotwell (79), attractants are of little value in baits and add unnecessarily to their cost. Although wheat bran is superior to other carriers, 50 percent sawdust may be substituted for the bran with good results. The following quantities of ingredients are recommended:

Sodium fluosilicate	4 pounds
Wheat bran	100 pounds
Water	10-15 gallons

For best results, the fluosilicate is thoroughly mixed with the bran. Sufficient water is added to make a wet mash, which will spread readily without lumping. The exact amount of water needed will depend on the moisture content of the bran or sawdust. The bait should be scattered in such a way as to cause it to fall apart in individual flakes. About 12 pounds per acre on dry basis is required. Since hoppers begin feeding in the morning, better results are secured by spreading the bait in the early morning hours.

MORMON CRICKET

From 1927 to 1935, the Mormon cricket (*Anabrus simplex*) was controlled by a dust of sodium arsenite. Liquid sodium arsenite, even in small quantities, acted as a repellent when used in baits. Cowan and Shipman (17) have developed a bait using sodium fluosilicate as the toxic agent.

A bait consisting of the following formula is recommended:

Standard bran	100 pounds
Sodium fluosilicate	3 pounds
Water	10-15 gallons

The bait should be spread, while the crickets are migrating, at the rate of 10 pounds (dry weight) per acre, either with mechanical spreaders or by hand. When spread at the recommended quantities there is no danger whatever to livestock or game birds.

CUTWORMS

The use of arsenical baits for the control of cutworms is well known. With the advent of fluorine compounds several workers have established the effectiveness of sodium fluosilicate and sodium fluoride (13, 43, 88).

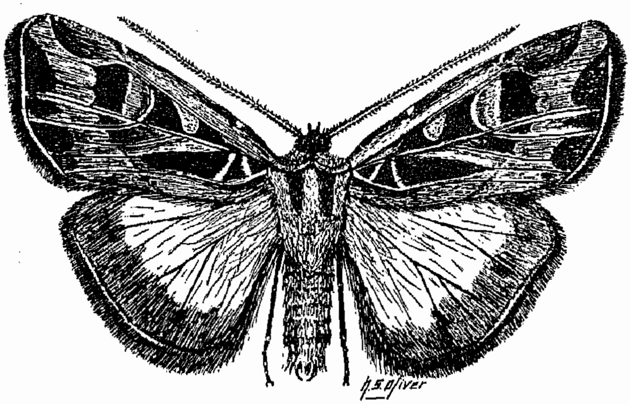

Fig. 5—Cutworm moth (*Feltia ducens*).
Eggs laid during the fall months soon hatch into small worms that overwinter.

Control of cutworms may be obtained by using a bait made up as follows:

Wheat bran or cottonseed meal	25 pounds
Sodium fluosilicate or sodium fluoride	1 pound
Water	3 gallons

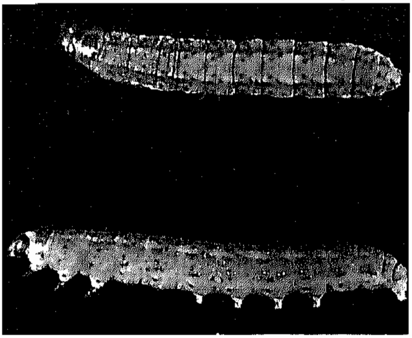

Fig. 6—Larvae of the cutworm.

The first two ingredients are mixed dry and the water added until a wet mash is formed. In the garden, small amounts should be scattered about the individual plants. Under field conditions 15-20 pounds per acre should be evenly distributed late in the evening or at dusk.

EUROPEAN EARWIG

Fulton (24) was the first to recommend sodium fluoride in baits for the control of the European Earwig *(Forficula auricularia)*. More extensive experiments by Crumb, Eide, and Bonn (18) showed that sodium fluosilicate is more effective and quicker-acting than sodium fluoride. They recommend the following bait:

Wheat bran	12 pounds
Sodium fluosilicate	1 pound
Fish oil	1 quart

The bran and fluosilicate are first mixed dry and then the fish oil worked in thoroughly. The bait is scattered thin over the entire area where control is sought, as along board fences, walks, and foundations of buildings, and about trees and woodpiles. The above quantity is sufficient to treat from 5000 to 8000 square feet.

STRAWBERRY ROOT WEEVIL

It appears that weevils of the genus *Brachyrinus* are attracted to baits. Downes (20) found that the strawberry root weevil *(Brachyrinus ovatus)* may be controlled by a bait of the following formula:

Apple pomace	95 percent
Sodium fluosilicate	5 percent

The sodium fluosilicate gave better results than magnesium arsenate or sodium fluoride. On the Pacific Coast, where the weevil is more injurious, two applications should be made—the first in April and the second in June. A small handful of the bait is dropped down among the crowns of the plants, at the rate of 50 to 100 pounds per acre. A commercial preparation known as "Go-West" is made up in the same way as the above formula.

ALFALFA SNOUT BEETLE

The alfalfa snout beetle *(Brachyrhinus ligustici)*, a parthenogentic, flightless weevil of European origin, has recently become a serious pest in New York State.

Palm et al. (64) found fruit baits attractive to this insect and have developed the following formula:

Raisins	50 pounds
Shorts (standard wheat middlings)	50 pounds
Sodium fluosilicate	5 pounds

Sodium fluoride also was effective, but paris green and cryolite were ineffective. The raisins are soaked in water for several hours. The excess water is then drained off and the shorts and insecticide added, after which the mixture is put through a food chopper twice. The first application is made during the last week in April. Two others are made at weekly intervals at the rate of 1 bushel of bait to the acre.

VEGETABLE WEEVIL

Laboratory tests on the vegetable weevil *(Listroderes obliquus)* by Cockerham and Deen (16) showed that the following bait is effective:

Sodium fluoride	1 pound
Wheat bran	15 pounds
Finely chopped turnips	8 pounds
Water to moisten	

Tests by Lewis (37) in California indicated that the dried-apple-pulp bait with sodium fluosilicate gave good results when broadcast at the rate of 75 pounds per acre. A dust of sodium fluosilicate or barium fluosilicate also was used successfully.

STRAWBERRY CROWN BORER

The strawberry crown borer *(Tyloderma fragariae)* is another flightless weevil that appears to be attracted to baits. Tests conducted by Ritcher (70) gave good results with the dried-apple bait coated with 3.5 percent sodium fluosilicate, sold under the name of "Go-West". Kills of from 87.5 to 100 percent were obtained.

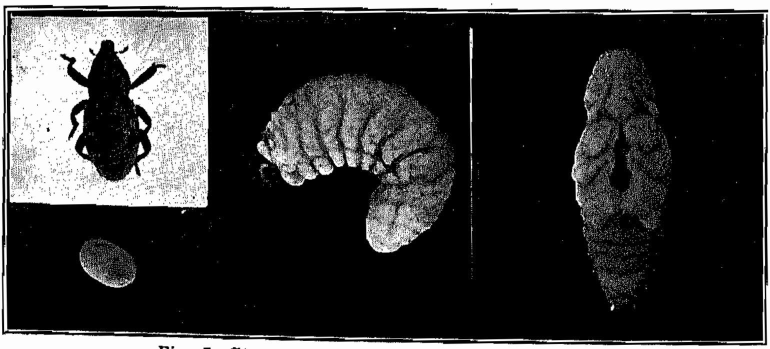

Fig. 7—Stages of the strawberry crown borer.

GREENHOUSE STONE CRICKET

Bue and Munro (10) report satisfactory control against the greenhouse stone cricket *(Tachycines asynamorus)* with the following bait:

Ground beef hamburger	90 percent
Sodium fluosilicate	10 percent

This bait resulted in 80-percent control within 4 days. Arsenicals were tried, but were found to be repellent to the crickets.

CLOTHES MOTH

As early as 1925 patents were taken out on the use of sodium fluosilicate as an ingredient of mothproofing compositions. There is a well-known mothproofing liquid on the market known as "Larvex", the active ingredient of which is sodium fluosilicate.

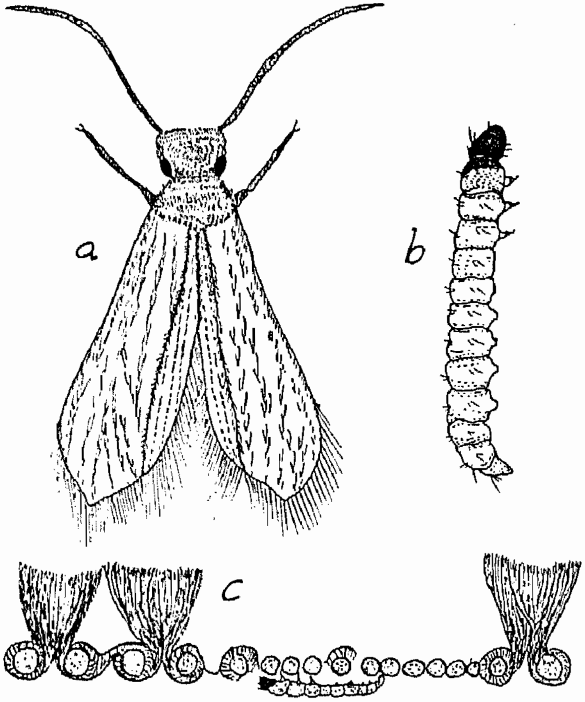

Fig. 8—The clothes moth.

a, b. Adult and larva (enlarged); c. diagramatic section of mohair covering illustrating feeding of worms from beneath.

As a mothproofing agent, sodium fluosilicate is inodorous, adheres to the fibers treated, does not dust off, is toxic to the larvae, and is reasonable in price. For use it is dissolved in warm water at the rate of 1 ounce or 2 tablespoonfuls in a gallon of water.

Where the fluosilicate is not available, sodium fluoride may be substituted. For best results, the material treated should be immersed in the solution or heavily sprayed. A light spraying on the surface will not reach the fibers within, where the larvae frequently feed. If the fabric is soaked thoroughly a deposit of sodium fluosilicate remains after the water has evaporated. The fabric thus becomes impregnated with the insecticide and will remain mothproof for a long time. Mothproofing with a solution of sodium fluosilicate is especially adapted for rugs, carpets, and mohair of upholstered furniture. We have obtained excellent results by treating rugs that were heavily infested with the clothes moth.

Fig. 9—Spraying a mohair-covered chair with a saturated solution of sodium fluosilicate as a permanent treatment for moths.

HOUSE FLY

Sodium fluosilicate is toxic to both larvae and adults of the house fly *(Musca domestica)*. A saturated solution (1-154), or about 1 teaspoonful dissolved in a pint of water, is readily imbibed by the adult flies, especially when no other food or water is about. The solution may be exposed in shallow dishes near windows where flies congregate. The flies succumb within 30 minutes after taking a drink of the solution.

Manure in which flies breed may be treated with sodium fluosilicate to destroy the larvae (45). If it is sprinkled with a 1-600 solution, no breeding of flies will take place. Such fluosilicate-treated manure will not be detrimental to soil, for the Tennessee Station has applied as much as 300 pounds of sodium fluosilicate per acre without injury to plant growth.

According to Sanders (71), flies are unable to complete their development in manure treated with fluosilicate. To quote: "I found that where I had sprayed a small pile of manure with ammonium silicofluoride that the larvae burrowing into the ground to pupate, did not complete pupation . . . . The small residue of silicofluoride in the soil was preventing pupation and giving effective fly control." Later, Sanders treated the soil only, using 1 pound of powder per square yard of soil, and again obtained a decrease in the number of flies.

COWPEA CURCULIO

A major insect pest in the Gulf States is the cowpea curculio *(Chalcodermus aeneus)*. Edible varieties of peas may become so "wormy" as to be unfit for use.

According to Arant (2), sodium fluosilicate was found to be the most satisfactory material for control. Dusting should begin before the first pods appear and be repeated at weekly intervals. Generally 6 applications, at the rate of 8 pounds to the acre, will be found necessary. The value of the peas protected may be estimated conservatively at $25.00 an acre.

SNOWY TREE CRICKET

The snowy tree cricket *(Oecanthus niveus)* is an important pest of raspberries in California. Tests by Smith (81) showed that a dust composed of 70 percent sodium fluosilicate and 30 percent diatomaceous earth applied at the rate of 50 pounds to the acre gave good control. A mixture of bran, water, molasses, amyl acetate, and sodium fluosilicate proved to be a satisfactory bait.

COMMON BLACK FIELD CRICKET

The common field cricket (*Gryllus assimilis*) has become a serious pest of certain field and garden crops in North and South Dakota. It is especially injurious to the developing seed of alfalfa, grain, flax, and sweet clover.

The most effective method for destroying the black cricket has been found to be a bait made with sodium fluosilicate. Severin (74) recommends the following formula:

Coarse bran	100 pounds
Sodium fluosilicate	5 "
Cane molasses, low grade, such as blackstrap	1½ gallons
Water	10-12 "

The mash should be broadcast over the infested fields at the rate of 20 pounds to the acre in the late afternoon.

BEAN WEEVIL

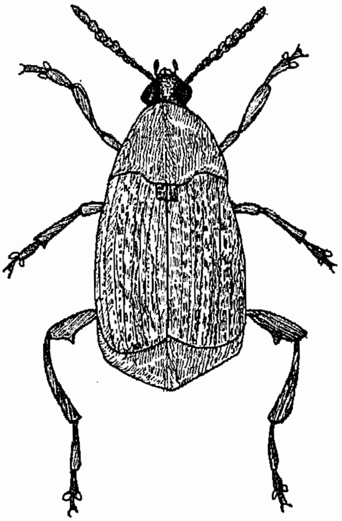

Fig. 10—Adult of the bean weevil. Enlarged 15 times

Stored beans are subject to attack by the bean weevil *(Mylabris obtectus)*, while cowpeas in storage suffer from the cowpea weevil *(M. quadrimaculatus)*. Experimental work in Tennessee (46) showed that protection could be had by thorough mixing of beans or cowpeas with sodium fluosilicate at the rate of 1 ounce to a bushel of seed. The sodium fluosilicate is advised only when the beans or cowpeas are to be used for seed purposes. When they are to be used for food, lime should be used at the rate of 1 pound to 1 bushel.

BARIUM FLUOSILICATE

Properties.—Barium fluosilicate is the least soluble of the fluosilicates, 1 part dissolving in 3750 parts of water. For this reason it is comparatively safe on most types of foliage. Like all fluosilicates, the barium salt is acid; one grain requiring 110 cc. of .1 normal NaOH for neutralization. A saturated solution shows a pH of 3.4.

Barium fluosilicate may be made from by-product fluosilicic acid or sodium fluosilicate. It is sold under the trade name of "Dutox". The insecticide so designated contains 72 percent barium fluosilicate, 8 percent cryolite, and 20 percent inert material. In general, barium fluosilicate shows no particular advantages over cryolite, so that in most instances the latter material is used. Because of the acid reaction of the barium fluosilicate, slight foliage injury sometimes appears. Damage to valves on spray pumps has been reported.

CRYOLITE

Properties and Uses.—Cryolite (Na_3AlF_6) occurs in Greenland as a native combined double fluoride of aluminum and sodium. It is more soluble than the barium fluosilicate, yet safer on foliage. The natural cryolite dissolves at the rate of 1 gram in 2,800 cc. of water, while the synthetic form requires only 1,639 cc. of water.

When placed in water, cryolite appears even more soluble than these figures would indicate. This is because the refractive index of cryolite is nearly the same as that of water.

Cryolite is soluble in dilute acids and akalis. This fact is taken advantage of by washing apples in dilute hydrochloric acid for the removal of spray residues. Cryolite reacts readily with lime, liberating soluble sodium fluoride and sodium aluminate. Since these materials cause foliage injury, cryolite should not be used with lime on plants. The reaction with lime takes place as follows:

$$Al_2F_6.6NaF + 6Ca(OH)_2 = 3Na_2O.Al_2O_3 + 6CaF_2 + 6H_2O$$

More than 10,000 tons is shipped annually for use in the manufacture of aluminum, the fused mineral being used as a bath and solvent in the electrolysis of alumina. Cryolite is used also in the manufacture of opaque white glass, in the enameling of ironware, and as a flux in making white Portland cement. During 1942 about 6 million pounds of cryolite was used for insecticidal purposes.

The natural cryolite when mined is freed from impurities and then ground. For insecticidal purposes the cryolite is put through a special pulverizing process and air-floated. The resultant product is a fine powder composed of particles 5 microns or less in diameter and neutral in reaction, and contains at least 90 percent cryolite. The form on the market today represents a considerable improvement in physical properties over the early cryolites, but the density still is greater than that of synthetic cryolite.

Synthetic cryolite (Na_3AlF_6) is made by several patented processes. Cryolite may be considered also as a sodium aluminum fluoride or a fluoaluminate of an alkali metal. Potassium fluoaluminate (K_3AlF_6) can be made, but it has no advantages over the sodium fluoaluminate.

Cryolite may be made from Al_2O_3 plus an alkali such as NaOH and hydrofluoric acid.

Howard (32) used the following method: $Al_2F_6 + 6NH_4F + 6NaCl = Al_2F_6.6NaF + 6NH_4Cl$.

A synthetic cryolite is made now by recovering the fused cryolite from the carbonaceous waste of the discarded pot linings of electrolytic cells used in the production of aluminum by the Hall process (58). The cryolite is dissolved in sodium hydroxide or sodium aluminate and sodium fluoride and precipitated with any acidic material, such as carbonic acid, at a temperature of about 60° F. This tends to give light, fluffy material that has good adherence on foliage and high susceptibility in water. This insecticide occupies about 80 cubic inches to the pound, and is sold under the trade name of "Alorco" cryolite. Aqueous suspensions of this insecticide are slightly alkaline, whereas most other synthetic forms are acid. Alorco cryolite is practically free from soluble sodium fluoride, and contains about 86 percent sodium fluoaluminate, with the balance mostly as aluminum hydroxide.

Effects on plants.—Repeated tests in Tennessee and elsewhere have shown that one of the marked advantages of cryolite is its safety for foliage. Even the most tender foliage, such as peach, apple, tobacco, or smartweed, is unharmed. Smartweed is especially sensitive to fluorine injury, but is not affected by cryolite.

Spray materials, in the course of years, may accumulate in the soil and injure plants by absorption through the roots. This result is known to occur when lead arsenate is used continually. On the sandy soils of South Carolina, for example, dusting with calcium arsenate for control of the boll weevil has made the soil unfit for cowpeas (86). A similar situation is becoming increasingly evident in the Pacific Northwest and other sections of the country.

Scott and Karr (72) made a study of the effect of arsenicals and natural cryolite on growth and yield of lima beans and peppers in sandy and clay-loam soils. Lead arsenate and calcium arsenate in concentrations of 500 to 3000 pounds per acre resulted in considerable reduction in growth, root development, and yield, while the cryolite had little effect. Soluble arsenic was detected in the soils where the lead and calcium arsenates were added.

Soil poisoning from the use of cryolite is not likely to take place because of the presence of calcium compounds, which induce the formation of the insoluble calcium fluoride. Lysimeter tests at the Tennessee Station have shown that the ultimate fate of added fluorine results in the production of the non-toxic insoluble fluorides, and that no cumulative effect could result from the combined use of cryolite as an insecticide (38).

Acid lead arsenate is known to cause defoliation of peach trees, especially where three or more sprays are used. Tests indicate that cryolite, while it is harmless to foliage, does sometimes affect the fruit, producing "tip-end" injury. Some seasons show more injury than others. Tests in Tennessee indicate that one spray of cryolite, such as the "shuck-fall", may be used with safety and thus lessen the damage caused by the subsequent lead arsenate sprays.

Compatibility with other insecticides and fungicides.—As indicated above, lime reacts with cryolite and therefore is not compatible. Other lime-containing chemicals, such as lime-sulfur, bordeaux mixture, and nicotine-lime dust, likewise are incompatible. Such fungicides as sulfur and the insoluble copper compounds may be used with cryolite. Cryolite may be mixed with any of the arsenicals. Calcium and magnesium arsenate, however, are decomposed by sodium fluosilicate, liberating large amounts of soluble arsenic.

Inert diluents, such as sulfur, clays, talc, and flour, make the best carriers for cryolite.

TOXICITY TO INSECTS

As with the arsenicals, each of the fluorine compounds has its own individuality and specific uses. Sodium fluosilicate is the most toxic and resembles paris green in toxicity and uses. Cryolite is less toxic and simulates lead arsenate in its broad aspects.

Generally, lead arsenate and cryolite are about equally effective against insects. There are specific instances, however, in which cryolite is more effective.

M.L.D., or median lethal dose, is the milligrams of insecticide required per gram of insect weight to give 50-percent kill. The studies are carried out under controlled conditions, and insects of known weights are given known amounts of insecticide. Over a period of several years, data have been obtained on the toxicity of many insecticides to different insects. In table 2 is a summary, including original work not yet published.

Table 2—*The M.L.D. of various flourine compounds.*

Compound	Insect	M.L.D. range	Investigators
Sodium fluoride	*Bombyx mori*	.11-.15	Shepard and Carter (76)
Sodium fluosilicate	" "	.10-.13	" " (76)
Barium fluosilicate	" "	.09-.12	" " (76)
Sodium fluoaluminate	" "	.06	" " (76)
Lead arsenate	" "	.09	Campbell and Filmer (11)
Sodium fluoaluminate	*Hyphantria cunea*	.45	Ellisor and Floyd (21)
" "	*Anticarsia gemmatilis*	.17	" " (21)
" "	*Autographa brassicae*	.42	" " (21)
Calcium arsenate	" "	.50	" " (21)
Sodium fluoaluminate	*Pieris rapae*	.68	" " (21)
Calcium arsenate	" "	.74	" " (21)
Sodium fluosilicate	*Melanoplus femur-rubrum*	.16	Richardson and Haas (68)
Paris green	" "	.19	" " (68)
Sodium fluoride	" *differentialis*	.11	Richardson and Thurber (69)
Lead arsenate	" "	2.0-4.0	" " (69)
Sodium fluoaluminate	*Heliothis armigera*	.07	Marcovitch and Stanley (50)
Magnesium arsenate	" "	.72-2.67	" " (50)
Rotenone	" "	.49	Hansberry and Richardson (28)
Sodium fluoaluminate	*Leptinotarsa decemlineata*	.11-.20	Original data
Sodium fluoride	" "	.13-.22	" "
Sodium fluosilicate	" "	.11	" "
Lead arsenate	" "	.14-.24	" "
Calcium arsenate	" "	.07-.14	" "
Paris green	" "	.034-.045	" "
Sodium fluoaluminate	*Protoparce sexta*	.11-.36	" "
Paris green	" "	.015	" "
Lead arsenate	" "	.085	" "
Sodium fluosilicate	" "	.23-.42	" "

Examination of table 2 reveals the fact that some insecticides are more specific against certain insects than others. Rotenone and magnesium arsenate, for example, are not toxic to *Heliothis armigera*, while cryolite is very toxic. Field tests afforded confirmation of this disparity. Against the tobacco hornworm, lead arsenate is twice as toxic as cryolite, while paris green is much more toxic than either.

EFFECT ON HIGHER ANIMALS

It is always of interest to know what effect insecticides will have on man and the higher animals, since in one way or another we are continually coming into contact with them. Often the impression prevails among the laity that anything that will kill a bug is poisonous. This raises the question as to what constitutes a poison. As our knowledge of insecticides has increased we have found that certain insecticides have a selective action against insects and are harmless to man. These obviously possess an important advantage. Even among those elements that are recognized as poisonous there are compounds that may be classified as relatively safe and non-poisonous. A glance at table 3 will make this point clear.

Table 3—*Acute toxicity of various substances, by mouth, to higher animals.*

Compound	Animal	Minimum lethal dose	Source
Potassium arsenite	Rabbit	5 mg. per kg.	Sollman (84, 85)
Calcium arsenate	Dog	38 " " "	Kuhn (33)
Lead arsenate	Dog	500 " " "	Kuhn (33)
Sodium fluoride	Rabbit	200 " " "	Muehlberger (60)
Sodium fluosilicate	"	125 " " "	" (60)
Barium fluosilicate	"	175 " " "	" (60)
Cryolite	Rat	13,500 " " " (not fatal)	Marcovitch (47)

An examination of this table shows that the soluble salts of arsenic and fluorine are much more toxic than the insoluble salts, and that in general the arsenical salts exceed the fluorides in toxicity.

Cryolite is unique in that it is not fatal to rats in any dose up to 13,500 milligrams per kilogram of body weight. For a person weighing 60 kilograms, this dose is equivalent to 1½ pounds of cryolite. It may be asserted confidently, therefore, that cryolite can not cause the death of a human being. This compound, even though it has been in use for a long time, is not even mentioned in books on pharmacology or toxicology. No fatalities from cryolite have ever been recorded in the literature.

In this connection a definition of the word "poison" is pertinent (51). The New York State law on pharmacy terms any chemical a poison that is liable to be destructive to adult human life in quantities of 60 grains (4 grams). Sollman (84), in his Manual of Pharmacology, gives this definition: "A poison is any substance which by its ordinary action is capable of seriously endangering health when it is applied to the body externally or in moderate doses (to 50 grams) internally." Under this definition, cryolite should not be classed as a poison. On the other hand, the soluble sodium fluoride, in quantities of approximately 4 grams, has been known to cause death, and consequently is a poison. So far as acute toxicity is concerned, cryolite satisfies the requirements for an ideal insecticide by having a high factor of safety for farm animals.

Chronic toxicity.—When farm animals sometimes come into contact with baits or spray residues of cryolite, some apprehension exists as to the effect of the chemicals on livestock; for example, when cows feed on grass under trees that have been sprayed with cryolite. Such evidence as is available indicates that the amount of fluorine consumed in this manner will have no effect on growth or cause any harm. When sodium fluoride was fed to rats in diets containing 200 p. p. m. there was no retardation of growth (80). The highest cryolite spray residues reported have been 50 p. p. m. Such high residues seldom remain long on plants in the East, since rains generally remove them. But even 50 p. p. m. in the diet is incapable of interfering with the growth or health of an animal. In addition it is now known that in small doses cryolite is only one-half as toxic as sodium fluoride. The Wisconsin Experiment Station (96) has shown that fresh green grass in itself has a protective action against fluorine. This could easily be due to the high iron and aluminum phosphate content found in forage crops, many of which analyze 2000 p. p. m. (95). As discussed under "Sodium Fluoride," aluminum compounds may act as antidotes to fluorine intoxication. Comparative feeding tests with fluorides and arsenic indicate that cryolite is several thousand times safer (47). So far as farm animals are concerned, the hazard presented by cryolite spray residues appears to be of no consequence.

SPRAY RESIDUES

Wormy, malformed apples or vegetables do not carry a sales or palate appeal. That our fruit may be protected from insect attack, it must be sprayed with certain chemicals, a tiny amount of which necessarily remains on the fruit at harvest time. Spray residues were given little attention until the English discovered them on apples in the form of arsenic in 1926. A tolerance of .01 grain of arsenic per pound of fruit was placed on American apples. Growers turned to cryolite as a substitute, since there was no known evidence that small quantities of fluorine were harmful.

During the last decade a considerable amount of information on the physiological effects of fluorine has accumulated. In 1931, two different workers, Churchill (15) in Pennsylvania and Smith (82) in Arizona, discovered independently that soluble fluorides in the water supplies of certain localities were the cause of "mottled enamel," or "brown stain," of teeth. One or two parts of fluorine per million in the water supply was capable of producing some mottling, and this was considered by the Food and Drug Administration as justification for placing a drastic tolerance of 1.4 parts per million (.01 grain per pound of fruit) on apples. This tolerance went into effect on June 20, 1933, to protect the public from discolored teeth.

Such a tolerance of fluorine placed it in a class with arsenic, which had the same tolerance. Fluorosis, or mottled enamel, is a water-borne disease that appears to have little or no relation to the tolerance arbitrarily placed on apples. To obtain more exact information, the Tennessee Experiment Station began tests on the toxicity of fluorine in drinking water as compared with that of cryolite spray residues (49).

The work was carried out with young rats, which appear to be well adapted for such studies. In the drinking water, 4 parts of fluorine per million produced striations on the incisor teeth, whereas cryolite fed as a powder in the food required from 7 to 11 parts per million to produce the same effect.

An additional factor that must be considered when fluorine occurs in the water supply is that the water is used for cooking as well as for drinking. To determine the effect of fluorine in a water supply that was used also for the preparation of food, a cooked diet of beans and rice and powdered milk was employed. The water used in cooking contained 1 part of fluorine per million. Dried navy beans require several hours to cook, so that 1700 cc. of water was used for 500 grams of beans. The cooked beans were found to have a fluorine content of 2.38 p. p. m. When they were fed to the animals, and with the drinking water containing 2 p. p. m. of fluorine, definite striations were produced.

The above experiments clearly indicate that fluorine in a water supply containing 1 p. p. m. is much more potent than the same proportion in foods, since water is used for cooking and the preparation of beverages, which in turn concentrate the fluorine. This concentration of fluorine is exemplified further by the analysis of a sample of teakettle scale from Arizona, which showed 8,072 p. p. m. of fluorine.

Another factor that does not apply to fluorine in foods is that of temperature. The food intake is fairly constant, whereas the fluid intake greatly increases during the warmer parts of the season, and especially in hot, dry desert-like climates, which necessitate the intake of large quantities of water for effective dissipation of heat. Water is ingested in greater amounts than all other substances combined. As much as 300 percent more water may be lost from the skin when the relative humidity is decreased from 69 to 31 percent. In a dry atmosphere a man at work requires a large quantity of water, often as much as 15 to 30 pints a day. Thus it is apparent that under hot, dry climatic conditions much more fluorine would be swallowed in a water supply containing 1 p. p. m. than in cool, humid localities.

Spray Residue Removal.—The tolerance on fluorine was raised by the Department of Agriculture, on November 14, 1938, to .02 grain per pound of fruit (2.8 p. p. m.). The spraying practices for the control of the codling moth on apple in the arid Northwest have fluorine residues ranging from .04 to .32 grain of fluorine per pound of fruit, depending on the number of cover sprays employed. In the East, fluorine residues are comparatively low because the rainfall is usually sufficient to remove most of the spray material at harvest. In the arid Northwest, however, extensive washing operations are necessary to remove the excess spray materials to meet the tolerance. Chemical solvents, such as 1.5 percent hydrochloric acid, are employed in special fruit washers of the double-process, overhead-flood type. With this type of washer, sodium silicate (9.6 percent) or sodium carbonate is used in the first treatment for 20 to 40 seconds, followed by 1 to 1.5 percent hydrochloric acid solution for the same length of time. The temperature of the solutions generally is maintained at 90° to 110° F. Wetting agents are used with the acid to aid in removal of the lead and arsenic residues, but they seem to be of less value in the case of fluorine residues. When three cover sprays of cryolite are employed, the above treatment will bring the apples below the tolerance of .02 grain.

FLUORINE IN FOODS AND BEVERAGES

Foods and beverages contain varying amounts of fluorine (47), ranging from 1 to 2 p. p. m., although some may have up to 12 p. p. m., as shown in table 4.

Table 4—*Flourine content of various foods and products.*

Food or product	p.p.m.
Apple (pulp)	.20
Cabbage	.59
Spinach	.95
Yeast tablets	2.00
Corn	1.00
Wheat (Arizona)	2.00
Corn germ	15.00
Prepared baby food (with bone meal)	12.00
Bone meal	450.00
Sardines	7.30
Salmon (canned)	4.50
Baking powder (phosphate)	228.00
Tea	66.00

As noted above, the foods containing the greatest amount of fluorine are sea foods, phosphate baking powders, bones, tea, and prepared baby foods with bone meal. The bones of marine animals contain ten times as much fluorine as those of land animals, while fossil bones may contain up to 3.48 percent. Since bone meal is used as a source of calcium in prepared baby foods, the question arises as to the injurious effect of the flourine which is present in amounts several times that of the tolerance. When a popular baby food containing 12 p. p. m. of fluorine was fed to rats, definite striations were produced; but when 1/3 milk powder was used, no visible striations resulted. It seems strange to allow 12 p. p. m. of fluorine in baby foods and yet legislate drastic regulations on much smaller quantities on other foods. The widespread use of canned fish, salmon, sardines, teas, phosphate baking powders, and bone products, and the fact that mottled enamel is localized according to the water supply, serve to emphasize the much greater effective toxicity of fluorine in solution when employed for both drinking and cooking.

FLUORINE AND SOUND TEETH

Does fluorine have a physiological role in the animal kingdom? Until recently the occurrence of fluorine in the body was considered accidental and, when the teeth were discolored, detrimental. During the past few years, however, different groups of investigators in different fields of science have accumulated independently a large body of impressive evidence that fluorine in some way exerts an inhibitory influence on the inception of dental caries; that a relationship exists between the incidence of tooth decay and the presence of fluorine in the domestic water supply up to 1 or 2 p. p. m.

To bring this evidence together, a symposium on "The Public Health Aspects of Dentistry with Special Reference to Fluorine," was held at Dallas, Texas, under the auspices of the American Association for the Advancement of Science (59). The general idea that mottled enamel has a deleterious effect on teeth was not easy to abandon.

The proponents of the toxic effect of fluorine were in the ascedency until 1937, when Armstrong and Brekhus (59) analyzed the enamel of sound and carious teeth and found the fluorine content of the former to be 111 p. p. m., as compared with 69 for the latter. In calcium, phosphorus, magnesium, and carbonate content, no difference could be detected between the two.

The inhabitants of the island of Tristan da Cunha are known to have a low incidence of dental caries. About 15.8 percent of the teeth are slightly mottled. This condition is believed to be due to a large consumption of marine fish—a striking example of mottling and caries-resistant relationship where the fluorine is not derived from drinking water.

A number of independent investigators report a conspicuous reduction in rat molar caries when fluorine is added to the caries-producing diet, or in the drinking water, or applied to the surface of the teeth. Enamel and dentine from rats fed fluorides were less soluble than were those from the teeth of normal rats.

An epidemiological survey of a mottled-enamel area, Galesburg, Illinois, was conducted by Dean (59), and contrasted with that of Quincy, Illinois, a nearby locality without mottling. The caries rate of Quincy, with .2 p. p. m. of fluorine in the water, was three times greater than that of Galesburg, with a water supply analyzing 1.9 p. p. m. of fluorine. Studies in other localities confirmed the general conclusion of an inverse correlation between the incidence of dental caries and the fluorine content of the water. The difference between 0.0 and 1.0 p. p. m. of fluorine in the domestic water supply has been shown to be highly significant from the standpoint of the amount of dental decay in a community.

The mechanism by which fluorine acts to produce sound teeth is believed to be related to the fluorine present in the dental structure. There is also the possibility of the inhibiting effect of the fluorine ion on micro-organisms or their metabolic processes. *Lactobacillus acidophilus*, the organism associated with dental caries, is not as numerous in the mouths of individuals in mottled-enamel areas.

Cox (59) has recommended that studies be made leading to the addition of fluorides to community water supplies where that element is too low to provide protection against dental caries. There is need for further data on the occurrence of fluorine in foods, drugs, and water supplies, both public and private. Armstrong (59) believes that "the whole evidence obtained from chemical analyses, animal experimentation, and epidemiological surveys of dental caries would seem to make a strong case for the beneficial role of fluorine in the prevention of dental decay."

A recent report (87) from the draft records of the 1918 army, the 1863-1864 Federal army, and the 1940-1942 draft records reveal that the worst teeth are found in New England. The teeth of the men from Vermont were 35 times poorer than of those from Arkansas. The lowest draft rejections for dental defects occurred in the West. New England water supplies are flourine-free. These findings coincide with those of the U. S. Public Health Service on the relation of flourine in the drinking water to tooth decay.

INSECTS CONTROLLED BY CRYOLITE

CODLING MOTH

Few insects cause as much damage as the codling moth (*Carpocapsa pomonella*) or have received as much attention. The situation is complicated by the problem of spray residues and their removal, especially in the Northwest, where the lack of rainfall and the large number of applications of lead arsenate required for control make it difficult to clean apples down to the tolerance limit. Much work has been done and a large number of insecticides have been tested in the hope of finding a substitute for lead arsenate.

Newcomer (62, 63) began testing fluorine compounds in the state of Washington as early as 1925, and by 1930 his work showed that cryolite was a genuine substitute for lead arsenate. From then on cryolite was given a place in the spray program of the Pacific

Northwest, and now is established as a reliable insecticide. That area has become the greatest consuming center for cryolite, its use there having reached several million pounds annually.

Cryolite is especially valuable for reducing the number of stings, and for that purpose is used in the last two or three cover sprays of July and August following early applications of lead arsenate. Such a split-spray program builds up a lighter deposit of lead and makes its removal less difficult. Cryolite has not caused any visible injurious effects on foliage or fruit of the apple tree in Washington. It is especially effective in the inverted, or "Dynamite," spray mixtures.

For best results, cryolite is used at the rate of 3 pounds to 100 gallons of water, with herring oil as a sticker. Three-fourths of a gallon of emulsified mineral oil or 1 pint of oil per 100 gallons of spray material plus ¼ pound of soap is sufficient for satisfactory results.

The residue removal is generally satisfactory when the fruit is washed in an effective flood-type machine, using 1.5 percent hydrochloric acid.

In the East, lead arsenate still is the standard spray material because it weathers better than cryolite and is less readily removed by rain. In time, however, cryolite may find a place in a mixed-spray program.

APPLE FLEA WEEVIL

Under the sod-mulch culture system of growing apples, the apple flea weevil (*Orchestes pallicornis*) may become a very serious pest (31). All the commonly available insecticides prior to 1933 gave negative results in Ohio. With the appearance of fluorine compounds, barium fluosilicate and cryolite were given a trial and found to be peculiarly toxic to this insect. In 1934 the following mixture gave the best control:

Dutox (barium fluosilicate)	5 pounds
Flotation sulfur	8 pounds
Goulac	3 ounces
Water	100 gallons

The sprays were applied during the pink and prepink periods, covering both the under- and upper-sides of the leaves. Equally good results were obtained in 1935 with cryolite. The flotation-sulfur plots were free from the leaf burn observed on the lime-sulfur plots. For best results the control program should be repeated the following year to decrease the weevil population in the orchard to the lowest possible stage.

ORANGE TORTRIX

Several orange worms, including the orange Tortrix (*Argyrotaenia citrana*), *Platynota stultana,* and *Holcocera iceryaeella,* are pests of considerable importance to the fruit and foliage of the orange in California. In experimental work at the Citrus Experiment Station, Basinger and Boyce (4) obtained satisfactory control, both in the laboratory and in the field, through the use of cryolite and barium fluosilicate applied as dusts and sprays. Organic compounds were ineffective.

Later work showed that .5 pound per mature tree can be used safely without exceeding the tolerance. A dust composed of 50-percent cryolite and 50-percent talc was satisfactory.

Platynota stultana is an important pest of carnations in California. Bohart (6) tested a large number of insecticides and obtained the best control with natural cryolite at the rate of 6 pounds to 100 gallons of water.

WALNUT HUSK FLY

Boyce (7) tried a number of materials against the walnut husk fly (*Rhagoletis completa*) in the laboratory and showed that cryolite exhibited a satisfactory degree of efficiency in killing the flies without foliage injury. The more soluble fluorines, such as sodium fluoride and sodium fluosilicate, were more rapid in their action, but produced foliage injury. The speed of toxic action was correlated with the solubility of the compounds in water. The less soluble calcium fluoride was not effective.

The following dust combination produced 97.3 percent control:

Synthetic cryolite	30 percent
Talc	65 percent
Mineral oil	5 percent

The dust method is more economical, but the following spray (8) may be used at the rate of 30 to 40 gallons per acre:

Cryolite	3 pounds
Mineral oil	1 quart
Water	100 gallons

Fig. 11.—Plum curculio at work on young peach.

PLUM CURCULIO

A suitable substitute for lead arsenate on peach as a control for the plum curculio (*Conotrachelus nenuphar*) would be a boon to growers. Three sprays are sufficient to cause some defoliation and, in some instances, severe injury.

Tests with cryolite indicate that it is safe on foliage, even when 5 or 6 applications are made. It controls the curculio equally as well as lead arsenate (89). The fruit of the peach, however, is sensitive to fluorine. Cryolite therefore cannot be used throughout the whole of the spraying season. Experimental work in Tennessee has shown that one spray, such as the shuck-fall, will not produce any injury. With a split-spray schedule of cryolite and lead arsenate, the injury to fruit should be reduced to a minimum. Dusts of cryolite seem to be safer than sprays.

MEXICAN BEAN BEETLE

The first practical tests with fluorine compounds were made against the Mexican bean beetle (*Epilachna varivestis*) in 1924. From 3 to 4 dust applications of sodium fluosilicate diluted with 9 parts of lime gave good control (41). Because of the tendency to produce foliage injury, the fluosilicate gave place to cryolite, which was found to be foolproof. Arsenicals are especially injurious to the bean plant; even the least soluble, such as lead arsenate and magnesium arsenate, will produce injury. Though burning of the foliage is not always visible, careful records show a reduction in yield where magnesium arsenate is used.

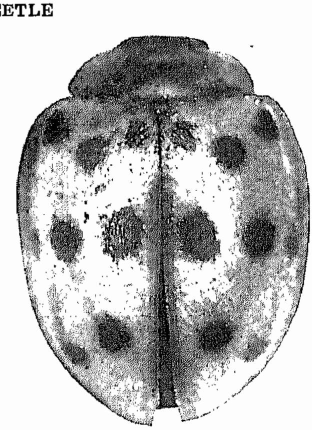

Fig. 12—Adult of the Mexican bean beetle.

For this reason arsenicals have been abandoned in favor of cryolite (44) or rotenone. Several years' tests have shown that spraying gives better control than dusting. For best results the cryolite is used at the rate of 3 pounds to 50 gallons of water and applied to the underside of the leaves. The first application should be made when the egg masses become numerous and a large proportion are hatching. Generally about 100 gallons of spray per acre will be required. Large bean vines may require 150 to 200 gallons per acre to wet them thoroughly. Two or three applications, a week or 10 days apart, should give satisfactory control.

Cryolite may be used also as a dust, diluted with a suitable carrier, such as wheat flour, sulfur, or clay. Hydrated lime should not be used. One part of cryolite with 1 part of clay or flour makes a very satisfactory dust, which may be applied at the rate of

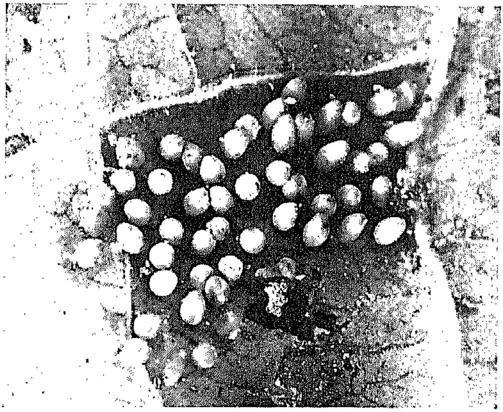

Fig. 13—Egg mass of the Mexican bean beetle.

20 pounds per acre. Three or four applications directed to the underside of the leaves, where the larvae feed, should give good control.

Fig. 14—Bean leaf, showing larvae of the Mexican bean beetle and their work.

BEAN LEAF BEETLE

The work of the bean leaf beetle (*Ceratoma trifurcata*) is most apparent when the plants are small. Unlike the bean beetle, it bites through the leaves and produces large round holes. This insect also attacks cowpeas and soybeans. Since the adult beetle injures the beans most severely in the seedling stage, a dust should be applied as soon as its work is observed. The same dust recommended for the Mexican bean beetle should be used.

TOMATO FRUIT WORM

The tomato fruit worm (*Heliothis armigera*) attacks many plants, including tomato, corn, cotton, and lima beans. A series of tests using sprays and baits were started in Tennessee in 1936. Rotenone was found to be ineffective, while calcium arsenate, lead arsenate, and sodium fluosilicate produced injury to the fruit (50).

Fig. 15—Tomatoes with larvae of tomato fruit worm feeding in them. (About ½ natural size).

The best results were obtained with the following cryolite bait:

Corn meal or cottonseed meal..............50 pounds
Cryolite.. 5 pounds

Table 5—*Tomato fruit worm experiments at Knoxville, Tennessee,* 1940. Tomatoes harvested from July 1 to July 24.

Treatment	Rate of application per acre	Tomatoes wormy[1]
Baits	Pounds	Percent
Corn meal plus 4% cryolite	40	9.1
Cottonseed meal plus 10% lead arsenate	50	11.2
Cottonseed meal plus 10% cryolite	50	15.7
Sprays	Gallons	
Cryolite, 6 lbs. to 100 gals.	180	22.0
Lead arsenate, 6 lbs. to 100 gals.	180	21.0
Cryolite, 6 lbs. and copper "34", 6 lbs., to 100 gals.	180	31.0
Copper "34", 6 lbs. to 100 gals	180	41.9
Check—not treated	----	47.3

[1]Analysis of data shows an error of 5.5 percent.

The untreated plots showed 47 percent wormy fruit. Baits were more effective than sprays, reducing the injury to 9 percent. While lead arsenate bait gave excellent control, it produced considerable injury to the fruit. Injury was observed also from the lead arsenate sprays. There was no injury to the tomatoes by cryolite bait or spray. Copper "34" when used with cryolite materially reduced the control of worms.

Fifty pounds of bait will treat an acre. This method has the advantage of being inexpensive and requiring no machinery. The bait may be carried in a bucket and a pinch or two applied to each fruit and flower cluster. From 3 to 5 applications will be required at 10-day intervals. For best results, the bait must be on the vines when the first fruits are the size of peas.

Fig. 16—Applying cryolite bait for control of the tomato fruit worm. This method is inexpensive and requires no machinery.

For spraying, the cryolite is used at the rate of 3 pounds to 50 gallons of water.

In California (93) good results have been reported with a dust, as follows:

Cryolite..70 parts
Talc..30 parts

From 10 to 30 pounds of the mixture was used per acre, depending on the size of the plants.

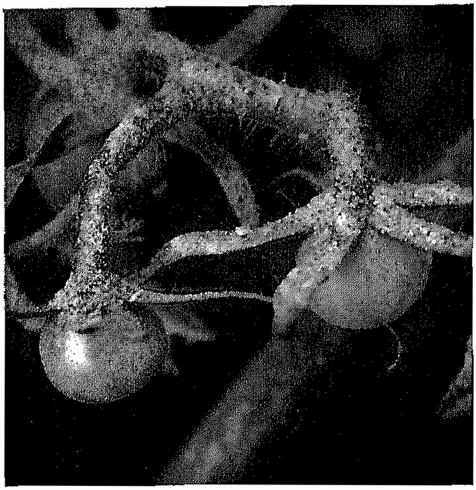

Fig. 17—Small tomatoes ready for first treatment with bait.

CONTROL ON LIMA BEANS

Next to corn and tomatoes, lima beans probably suffer most from the fruit worm in Tennessee. In 1936, several hundred acres of limas grown for canneries in East Tennessee were almost completely destroyed. That year, because few bean beetles were present, the vines were not sprayed.

As in the case of tomatoes, eggs are deposited on the blossom clusters and leaves. Upon hatching, the small worms feed for a time on the flowers and leaves, and then migrate to the developing pods. When the worms are about half grown they begin boring into the pods and eat the small beans within. The depredations are easily recognized by the round entrance holes.

Experiments conducted in Monroe County in 1939 are summarized in table 6. Four applications were made. The data in

Table 6—*Results of treatments on lima beans for control of H. armigera in Monroe County, Tennessee,* 1939.

Treatment	Rate of application per acre	Larvae per plant Sept. 15	Average weight of pods Sept. 25
	Gallons	Number	Ounces
Magnesium arsenate, 5-100; lime, 7½-100	150	1.37	197.3
Rotenone 5%, 2½-100	150	1.29	195.0
Check	None	1.35	191.0
Natural cryolite, 8-100	150	.52	222.5
Alorco cryolite, 7-100	100	.70	214.5
" " 8-100	150	.56	238.3
" " 8½-100	200	.28	232.3
" " 10%, in cottonseed meal	[1]	.85	198.5

[1]50 pounds per acre.

table 6 show that all the fluorine treatments effectively reduced the number of larvae. Counts were made also on the number of loopers (probably *Autographa brassicae*) present. Plants treated with fluorine sprays had about one-tenth the number of loopers found after the other treatments.

On September 25 the lima beans were at the right stage for canning. Data on yield were obtained by removal of pods and recording of weight in ounces per 100 feet of row. As indicated in table 4, both magnesium arsenate and rotenone were shown to have no effect. The best control was obtained with a cryolite spray—8 pounds to 100 gallons of water—applied at the rate of 150 gallons per acre.

The plot sprayed with cryolite gave a yield of 3,148 pounds of beans per acre, while the unsprayed plot gave a yield of 2,195 pounds. The sprayed beans showed a net profit of $24.75 per acre.

In Virginia, Brannon (9) obtained good results with cryolite at the rate of 3 pounds to 50 gallons of water. The following dust also was used with good effect:

Cryolite	60 parts
Talc	40 parts

Three or four treatments are necessary, beginning about the time the beans come into bloom.

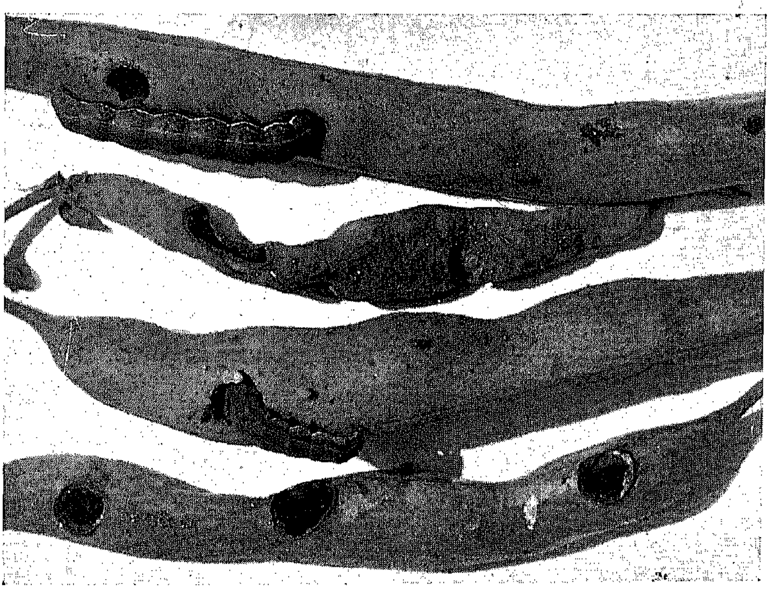

Fig. 18—Corn ear worm larvae boring into pods of bunch beans. This same type of injury is produced on lima beans.

PEPPER WEEVIL

Campbell and Elmore (12) present data on the commercial control of the pepper weevil (*Anthonomus eugenii*) in California by means of cryolite. The use of calcium arsenate resulted in heavy infestations of aphids (*Myzus persicae*), causing considerable defoliation of plants, whereas cryolite was without practical effect on the lice. The use of pyrethrum dust in 3 fields showed little control. The dust mixture used consisted of—

Cryolite	50.0 percent
Cube	10.0 percent
Talc	40.0 percent

It was applied at the rate of 15 to 25 pounds per acre per application. Six dustings were given the plants. The residues were successfully removed by washing machines. "The treated fields produced more than 3 times as many pods as the untreated fields . . . . The average net profit on treated fields was $141 to $241 more per acre than on untreated fields."

CRANBERRY INSECTS

The cranberry fruit worm (*Mineola vaccinii*) is the most important cranberry insect in Massachusetts, often destroying a third or more of the crop. A dust of cryolite during the blossoming period and again 10 days later has given satisfactory control. In 1940 about 17,000 pounds of cryolite was used on Massachusetts bogs (23). It takes 4 or 5 days to effect a kill, but it stops worm feeding on foliage very soon. Good results may be obtained by using 5 pounds of cryolite in 100 gallons of water as a spray, at the rate of 400 gallons per acre.

For the false army form (*Xylena nupera*) and blossom worm, (*Epiglea apiata*), a spray of 6 pounds of cryolite in 100 gallons of water, 400 gallons to the acre, is recommended by the Cranberry Experiment Station (23). Cryolite will also control the weevil (*Anthonomus musculus*) that feeds on the buds and young fruits.

The gypsy moth caterpillar (*Porthetria dispar*) often attacks the terminal buds, leaves, and flowers. The above spray of cryolite as used for the weevil will keep this insect in check.

STRAWBERRY LEAF ROLLER

Insecticide tests against the strawberry leaf roller (*Ancylis comptana*) in Ohio (61) showed that lead arsenate was not effective. Both natural and synthetic cryolite yielded good results, especially when a spreader was used. The foliage should be well covered when the first eggs are hatching, and coverage should be maintained during the period that the larvae are actively feeding. Three applications, 5 pounds to 100 gallons, at weekly intervals will give commercial control.

In Kansas, cryolite sprays with neutral soluble fish oil showed good control (35).

SUGAR-CANE BORER

As early as 1925, Holloway, Haley, and Ingram (30) showed that a dust of sodium fluosilicate would control the sugar-cane borer (*Diatraea saccharalis*).

Later work in Louisiana indicates that cryolite will give a high degree of control without plant injury (67). Weekly applications were made during the time the young larvae were on the exposed foliage and before they tunneled into the plants. Bordeaux-calcium arsenate and dual-fixed nicotine were without effect.

STRAWBERRY WEEVIL

Laboratory tests with various insecticides were conducted by Pollard and Thomas (65) against the strawberry weevil (*Anthonomous signatus*). The most effective mixture was 21 percent cryolite with clay or sulfur, producing a mortality of 75 percent. Nicotine bentonite was of no value.

VELVETBEAN CATERPILLAR

The velvetbean is another plant that is sensitive to foliage injury by arsenicals. Tests in Louisiana by Ellisor, Gayden, and Floyd (22) show that cryolite offers a practical means of control against the velvetbean caterpillar (*Anticarsia gemmatilis*). The cryolite was mixed with half talc, and the mixture applied at the rate of 12 to 16 pounds per acre.

In South Carolina last year 500 acres were dusted with cryolite, 50 percent talc being used as a diluent.[2]

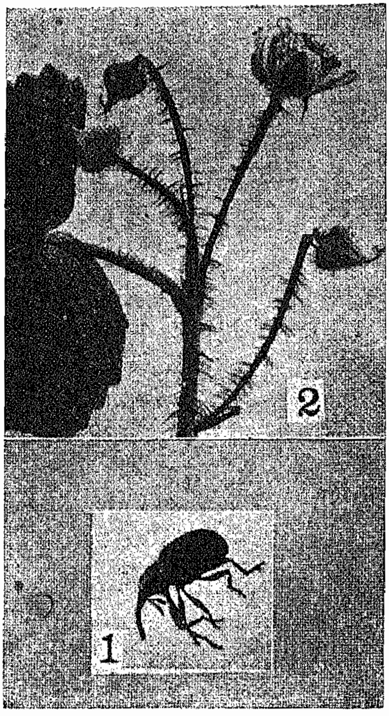

Fig. 19—Strawberry weevil.
1. Side view, enlarged about 7 times.
2. Raspberry buds cut by the weevil. It works the same on strawberry.

[2]Letter from W. C. Nettles, Extension Entomologist.

TOBACCO FLEA BEETLE

The common flea beetles, such as the potato flea beetle (*Epitrix cumcumeris*) and the tobacco flea beetle (*Epitrix parvula*), readily yield to dusts of fluorine compounds (48). In Connecticut, Lacroix (34) found that barium fluosilicate kept its efficiency for 10 days or so and gave longer protection than rotenone or pyrethrum. Allen and Shands (1) also state that a cryolite-dust mixture containing 80 percent sodium fluoaluminate gave better plant protection over a period of 6 days than 1 percent rotenone-dust mixture.

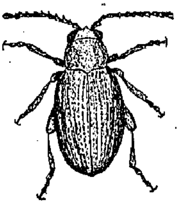

Fig. 20—Tobacco flea beetle.
Enlarged 9 times.

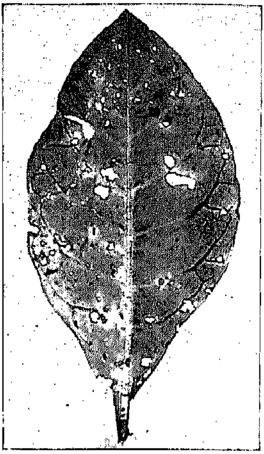

Fig. 21—Work of tobacco flea beetle.
If not controlled, flea beetles may cause great losses by direct injury to leaves and by transmission of diseases.

TOBACCO HORNWORM

From the standpoint of control, lead arsenate is the most economical material for the protection of tobacco against the tobacco hornworm (*Protoparce sexta*). The extensive use of lead arsenate is not recommended by the Tobacco Insect Council because

Fig. 22—Dusting tobacco with cryolite to control hornworms and flea beetles.
Note how the dust has formed a fog and is settling down, giving an excellent coverage. Late evening is a good time to apply dust, as the air is still, leaves are wilted and will not break, and there is no dew to wet the operator.

the use of this material may leave two objectionable residues, lead and arsenic.

To correct the residue situation, the Tobacco Insect Conference (91) advises a dust containing 70 to 80 percent sodium fluoaluminate, to be used at the rate of 10 to 15 pounds per acre. The hornworm is not as susceptible to cryolite as it is to arsenic, and consequently about twice as much per acre is necessary for comparable control. This is shown by the M. L. D. figures, which are presented in the forepart of the bulletin.

Fig. 23—Small hornworm. Best results from poisoning are obtained with small worms; large ones are difficult to kill.

Cryolite spray is more economical than the dust. It should be applied at the rate of 6 pounds to 50 gallons of water, using 60 to 70 gallons per acre (91). A sufficient number of nozzles, properly spaced and arranged, are essential for obtaining satisfactory plant coverage.

CUCUMBER BEETLE

Successful results with fluorine compounds against the striped cucumber beetle (*Diabrotica vittata*) have been reported in a number of states, including California, Tennessee, Arkansas, and Indiana. Gould (26) obtained good results with a mixture of 20 percent cryolite and 80 percent talc. Other insecticides tested were calcium arsenate and basic copper arsenate, but the highest yield was obtained with the cryolite mixture. The first insecticide application was made as soon as the plants came through the ground. Dusts were applied at 3- to 5-day intervals until 2 days before the first picking.

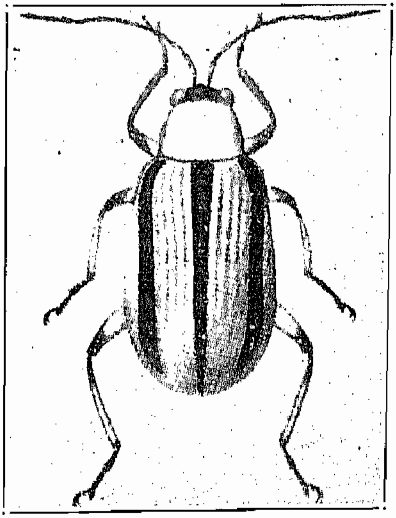

Fig. 24—Cucumber beetle.

CABBAGE WORMS

Cabbage worms, such as *Pieris rapae,* and the looper (*Autographa brassicae*) may be controlled by a dust of cryolite diluted with 1 or 2 parts of talc or flour. The dusts should be applied as soon as the first indications of worms are observed, and the applications should be repeated at weekly intervals until control is obtained.

BLISTER BEETLES

Blister beetles, such as *Epicauta marginata* and *E. Pennsylvanica,* are especially susceptible to fluorine compounds. Dietz (19), in tests with more than 5000 insects, showed that barium fluosilicate with flour or clay (1-4) killed a uniformly high percentage of beetles. Lime reduced its efficiency. In the field, from 25 to 35 pounds per acre of the mixture gave good control on potatoes and other vegetable crops, without injury to the plants.

COTTON BOLL WEEVIL

In general, calcium arsenate is the preferred insecticide for the cotton-boll weevil (*Anthonomus grandis*). Its continued use sometimes results in soil poisoning, especially on sandy soils. Experimental work in South Carolina (86) on the effect of insecticides on plant growth showed that 200 pounds of calcium arsenate added to the soil injured cowpeas. No adverse effect was observed when as much as 1600 pounds of cryolite was added.

In control studies against the boll weevil, cryolite with a synthetic liquid sticker and spreader (Lethane) was comparable to calcium arsenate (66).

PINK BOLLWORM

The pink bollworm (*Pectinophora gossypiella*) is difficult to control with insecticides because of its manner of feeding within the bolls or squares of cotton. Experimentation in the Presidio Valley of Texas, had shown that arsenicals were not effective. The best results were secured from a 33-percent cryolite dust (14). An 85-percent cryolite dust gave a reduction of 53.4 percent, and a cryolite oil spray, 59.6 percent. Six applications of each insecticide were made at 4-day intervals.

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Bagworm (*Thyridoptoryx ephemeraeformis*)
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Grape-leaf folder (*Desmia funeralis*)
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Tomato pinworm (*Gnorimoschema lycopersicella*)
New Mexico Agr. Exp. Sta. Bul. 288. 1942.

SUMMARY

In 1924 the Tennessee Experiment Station called attention to the value of sodium fluosilicate as an insecticide. Since then the fluorine compounds, particularly sodium fluosilicate and cryolite, have had a marked development, opening up a new field in the control of crop insects.

Sodium fluoride is used in the control of household insects such as cockroaches, chicken lice, silverfish, and ants. With pyrethrum, sodium fluoride acts synergistically against roaches.

Sodium fluosilicate, a by-product of the fertilizer industry in the manufacture of acid phosphate, has found a place for itself as a mothproofing agent, and as the toxic ingredient of baits for the control of grasshoppers, crickets, cutworms, and various weevils.

The virtues of cryolite as an insecticide lie in the fact that it will not injure foliage nor result in soil poisoning. In its effect on insects, cryolite is in the same zone of toxicity as lead arsenate. For certain insects, cryolite is specific and more toxic than magnesium arsenate or calcium arsenate.

Another marked advantage of cryolite is the fact that it is incapable of causing any fatality if swallowed. Animals fed the equivalent of 1½ pounds of cryolite for a person weighing 125 pounds, did not succumb. No fatalities from cryolite have ever been recorded in the literature. Cryolite has, therefore, a high factor of safety for farm animals.

Cryolite spray residues as they occur in practice are non-poisonous and incapable of having an adverse effect on teeth.

Fluorosis is a water-borne disease, endemic in hot, arid climates where well water, containing more than 2 parts of fluorine per million, is used for both drinking and cooking.

The present tolerance of fluorine allowed on fruits and vegetables by the U. S. Department of Agriculture is 2.8 p. p. m. or .02 of a grain per pound. Many common foods, however, show a fluorine content of 1 or 2 p. p. m. and some have up to 12 p. p. m. Sea foods are especially rich in fluorine, sardines, for example having 7.3 p. p. m. Baby foods containing 2 percent bonemeal show a fluorine content of 12 p. p. m., which is several times that allowed on apples.

Since 1937, an impressive body of evidence by different investigators has appeared showing that good, sound teeth have a higher fluorine content than poor, carious teeth. The fluorine content of the former was found to be 111 p. p. m., while the latter had 69. The caries rate of Quincy, Illinois, with .2 p. p. m. of fluorine in the water, was 3 times greater than that of Galesburg, with a water showing 1.9 p. p. m. The recent records reveal that New England draftees have the worst teeth, while those from the West have the best. New England water supplies are fluorine-free, corroborating the findings of the U. S. Public Health Service on the relation of fluorine in the drinking water and tooth decay as noted at Galesburg, Illinois, and other localities of the West. All the evidence points to the general conclusion of an inverse correlation between the incidence of dental caries and the fluorine content of the water.

The Pacific Northwest has become the greatest consuming center for cryolite. Several million pounds are now used there annually to fight the codling moth.

In California cryolite has been shown to give satisfactory control against the orange worms and the walnut husk fly.

In the East large amounts of cryolite, both as dust and spray, are used for the suppression of the Mexican bean beetle and other pests.

Cryolite appears to be especially toxic toward the tomato fruit worm (*Heliothis armigera*). This same insect also attacks cotton and lima beans. On tomatoes the cryolite is best applied in the form of a bait, using 10 percent cryolite with corn meal or cottonseed meal, at the rate of 50 pounds of bait to the acre. On lima beans the best results were obtained with a cryolite spray—8 pounds to 100 gallons of water—applied at the rate of 150 gallons to the acre.

Flea beetles, cabbage worms, cranberry insects, blister beetles, and cucumber beetles are other insects that are readily controlled by cryolite.

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