

Ag and Food Interprets . . .

- ▶ **Silica dusts as insecticides look promising**
 - ▶ **Preneutralization cuts costs for fertilizer formulators**
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Silica Gel As Insecticide

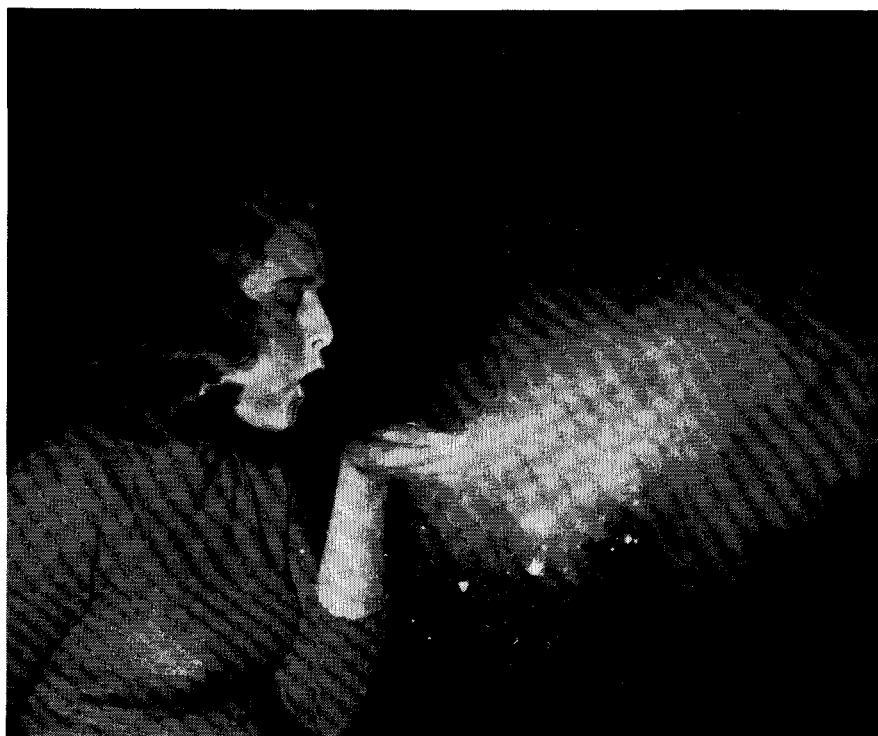
A new role for an old chemical is exciting because of indications it is not toxic to humans, and insects may not develop resistance

RELEASE of experimental quantities of Dri-Die insecticide by Davison Chemical has unleashed a whole barrage of talk and excitement about a "new method" of killing insects. The new angle is that Dri-Die is an inorganic chemical (contrasting with the organics that have been used almost exclusively since World War II) that kills by a mode of action thought to be physical rather than chemical.

Also causing excitement are preliminary indications that because its mode of action is physical rather than chemical, there is a probability that insects will not develop resistance to it, as they have to so many organics.

Dri-Die is a finely divided silica aerogel, of a type that has been made by Davison and many others since World War II. It kills insects that walk through it by absorbing the outermost waxy layer of the insect's body, causing loss of vital body moisture—that, at least, is the prevalent theory now.

Using a dust to kill insects is not really new; it has been used for over 2000 years. In some areas of the Middle East, plain road dust is still



A cloud of silica gel—this one a product of Monsanto's that was made for use in lacquers and varnishes. It is the fineness of silica aerogels that makes them useful for a number of products—including insecticides perhaps

frequently used as an insecticide. And it has been known for some time that the talcs, pyrophyllites, diatomites, attapulgites, and other natural silicates used as insecticide carriers and diluents aid insect control. In fact it was a study of dust diluents and carriers by Walter Ebeling of the University of California that led to the revival of interest in dusts, and eventually to present knowledge of

silica gel's insecticidal properties. Using a number of different dust formulations of DDT, he found with the silica gel formulations that as the percentage of DDT diminished (and consequently the percentage of silica gel increased), insect kill increased.

Pursuing the matter further Ebeling and his associates came to the conclusion that silica gel could be used as an insecticide. In the lab, it kills

ticks, flies, four species of cockroaches, fleas, termites, and many other insects. In the field, it has demonstrated control over fleas, roaches, chicken mites on poultry, two-spotted mites, silverfish, bedbugs, and others. They concluded that of the various silica gels tested, Davison's SG-67 (now called Dri-Die for the insecticide market) was the most efficient killer.

At a panel discussion on silicon dioxide insecticides at last month's meeting of the National Pest Control Association in Washington, D. C., Barry I. Tarshis, an associate of Ebeling's, reported that in the laboratory they had achieved knockdown of roaches within 15 minutes after exposure to silicon dioxides, and kill in 45 minutes. He said silica gel kills faster than parathion, Diazinon, DDT, dieldrin, and many other toxicants.

To support the desiccation theory of silica gel's mode of action, he said lab tests showed that American cockroaches killed by silica gel suffered a 35% loss in body weight. Some 60% of cockroach body weight is water.

University of California researchers are testing silica gel in some 35 establishments—homes, food plants, restaurants, institutions. In homes, they are applying the powder with a gun, using about 0.5 to 0.75 lb. per house. It is applied under refrigerators, sinks, around drainboards, under stoves, and other places frequented by roaches.

At the same panel discussion, Earl K. Seybert of Davison described some of the properties of his company's SG-67, which is being supplied to pest control operators for testing. Davison began making silica gel during World War II as an adsorbent for use in gas masks. Since then, it has found many uses—in toothpaste, in human medicine (it was once used as a hangover remedy), and for many other products. It is manufactured by mixing sulfuric acid and natural sodium silicate; the reaction product sets into a hard gel. The gel is then broken up, washed to remove sodium sulfate, dried, and sized. Natural silicas, he said, are nonporous, but silica gel is so porous that it can absorb 100% of its own weight in water.

One of the most favorable properties of silica gel from the insecticide standpoint is that it is nontoxic. Years of experience with it in industry have shown it to be safe—there are no known cases of silicosis developed as a result of its use. However, because the product that UCLA researchers found best (SG-67 or Dri-Die) is chemically treated (it has a fluoride coating), Davison had it put through the toxicology hurdle at Hazleton

Laboratories. Hazleton reported back:

"Since no toxicity was observed at 10,000 parts per million level (1.0%), we consider this material to be an extremely safe chemical. From our previous studies it was apparent that 67 is a relatively nontoxic material when administered as a single dosage by the oral, dermal, and inhalation routes of exposure."

However, because its product is a desiccant, Davison recommends use of an industrial mask by the applicator to prevent unpleasant drying of the nose and throat.

On the basis of toxicity tests, USDA has approved an experimental label to place silica gel on the market for professional evaluation.

Others Interested

Some of the other manufacturers of synthetic silicas who are following these developments are Godfrey L. Cabot, Philadelphia Quartz, and Columbia-Southern. J. M. Huber is also interested, as undoubtedly are many others. Another manufacturer of silica gel interested in these findings is Monsanto, which has a promising material in its Santocel C.

Monsanto's product was not tested at the University of California. However, Monsanto has put a research team to work on it. At this early date, Monsanto has been able to report that Santocel C is effective against a number of household insects in the laboratory. It also has found Santocel C to be effective in stored grain at the level of 0.025% of the weight of the grain. Monsanto's interest was revealed at the Washington panel discussion by J. Marshall Magner of Monsanto, who pointed out that it will take time to find the best aerogel, and that any possible use of silica for crop protection is far from full evaluation. He said more information will have to be gathered on techniques and equipment for application, on residual toxicity, and on the mixing of silicas with other toxicants.

Another question still needing a definitive answer is the effect of humidity. Dr. Tarshis says that in an atmosphere of 40% relative humidity, 100% kill is achieved, and even at 100% relative humidity, efficiency is not diminished. But R. J. Norton of Crop Protection Institute does not go along with this. Dr. Norton cautioned the audience not to run too fast with silica. He feels it is a significant insecticide, but many problems must still be worked out. He does not go along with desiccant theories of its

mode of action, but he did not offer any other explanation of its killing power. He said he believes that its lethal action is a direct function of dosage—that is, more particles give better kill.

During recent months, some members of the NPCA insecticide committee have been trying Dri-Die. They report they did not get universally good results. Also, they found it difficult to handle and control.

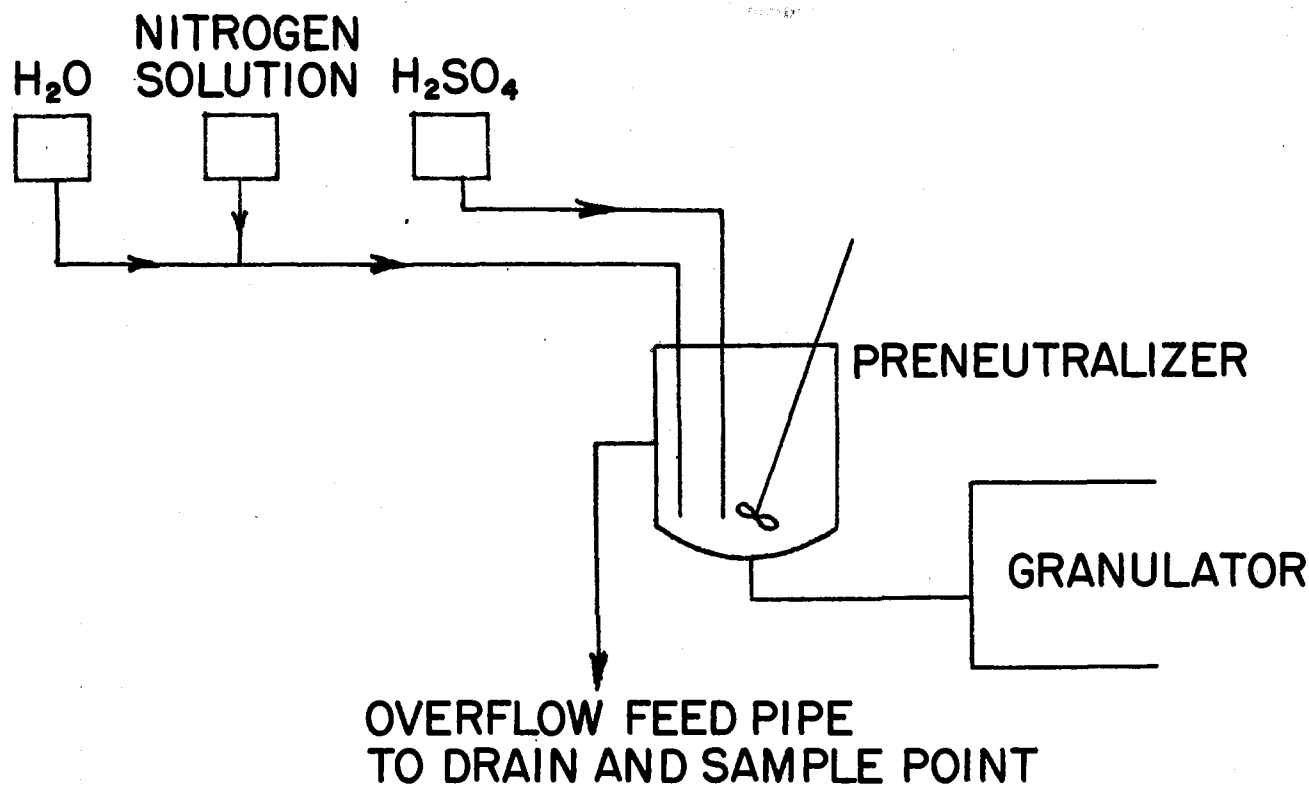
With the amount of information available now, the insecticidal development of silica gel could ride off in all directions. It may have application in agriculture. It may be the answer to the homeowner's scourge—cockroaches. It could become a widely used carrier or diluent for organic toxicants, adding its own killing power to that of the organics for superior efficiency. It could be all of these things, it could be one of them, or it could be none of them. In short, with silica gel it is the same old story—more research needed. An often heard caution, but nevertheless a necessary and wise one.

Preneutralization

New process permits making non-phosphate and inverted grades without dry nitrogen materials

A PROCESS which makes possible the manufacture of nonphosphate, high nitrogen—up to 20%—fertilizer grades without using dry nitrogen materials will soon be appearing in several places around the country. Heralded as a big step in the direction of still higher analysis, the process involves neutralizing the free ammonia in a nitrogen solution with sulfuric acid before adding to the mixer.

Original disclosure of this simple, yet significant addition to fertilizer technology was made by Virginia-Carolina in 1957. As announced and still practiced by V-C, the technique permits formulating an X-O-X fertilizer without dry nitrogen sources; 8-0-24 is the main grade produced this way at the company's Wilmington, N. C., plant. A 14-0-14 grade using relatively little dry nitrogen also is made there. In September and Octo-



Simplified flow diagram for preneutralization as set up by Spencer in a midwestern plant

ber of this year, both Allied and Spencer independently announced that the process can also be used to granulate high nitrogen grades like 2-1-1.

And the same principle can be applied to conventional grades such as 1-1-1, says Spencer, with significantly lower costs. For example, preneutralization (Spencer's name for the process) can cut 12-12-12 formulation costs by \$1.00 to \$2.00 a ton.

Thus far, Spencer has helped set up one commercial unit at a midwestern plant, while several other plants in the company's marketing area are now installing necessary equipment. And Allied's Nitrogen Division, which says it has been working on this approach since 1950, reports that still other fertilizer manufacturers are planning to install its process. Grades produced will include 14-7-7, 16-8-8, 14-0-14, and 15-10-10. Both companies are offering their customers assistance in design and operation.

Principle, Equipment Simple

The idea behind preneutralization is certainly not new, although it has not been used commercially in the fertilizer industry. In a granulation process, free ammonia has to be absorbed

or neutralized. Conventionally, this function is performed by superphosphate or phosphoric or sulfuric acid in a TVA ammoniator or pug mill. Neutralizing the free ammonia with acid before allowing the solution to converge with the solid material obviates the need for phosphate, resulting in X-O-X formulations. In the case of inverted grades, preneutralization permits the use of considerably higher nitrogen concentration than would be the case if free ammonia had to be neutralized.

Equipment, too, is simple. According to Spencer, only a small outlay for additional equipment is necessary. For instance, one estimate holds that \$3000 to \$5000 will cover the added equipment for a \$100,000 fertilizer plant. The major piece of added equipment is a stainless steel tank into which the nitrogen solution and sulfuric acid are piped for neutralization. The tank is located near to and above the ammoniator or pug mill so the hot solution can flow by gravity. In some instances it may be on the roof of a plant; in others, in a corner of the plant interior, or even outside.

Allied says it is working on several refinements and variations of the process, and may be revealing these in the near future.

The process outlined by Spencer begins with acidity control in the preneutralizer tank. When acidity gets too high, nitrogen can be lost as nitric acid. If the mix is too alkaline, loss of nitrogen results from flashing of ammonia. High acidity also increases formation of ammonium bisulfate, which gives a more hygroscopic product. And excessive fume formation accompanies a high-acid state. Corrosion is kept at a minimum when acidity is carefully controlled.

Sulfuric acid used is 66° Be' to get high enough temperatures in the preneutralizer to cut down recycle. A small amount of water may be added through a sparger to control temperature. The finished granulated material is dried at a low temperature, and a parting agent such as kaolin clay or diatomaceous earth is added to prevent caking.

Spencer notes that dolomite is probably the best filler for X-O-X grades. Dolomite adds magnesium to the fertilizer and acts as an anti-acid without liberating excess ammonia. Sand works as a filler, too, but the final product's appearance suffers. Agricultural limestone is not recommended since it causes noticeable ammonia liberation over long periods.

Potash Particle Size

Granulating high potash-low nitrogen mixes forces compromise between quality and production rate

PRODUCERS OF POTASH have over the past few years become increasingly conscious of the particle size distribution of their agricultural products. The reason: granulated complete mixes. This form of fertilizer has grown from a standing start in 1950 to better than 4 million tons a year in 1957.

However, being conscious of their particle size distributions has not led potash companies to clearly defined grades in all cases. Potash firms now produce in three major size grades, but only two of these are defined to everyone's satisfaction (although different producers have different distributions and particle shapes even within those two grades). The three ranges are:

- **Standard.** For general use in mixed fertilizers and for granulation of high nitrogen and intermediate grades. Most of these are finer than 16 mesh and larger than 50 or 100 mesh (with 5 or 10% fines). The exact distributions are not very important within these ranges.

- **Granular.** These are primarily for application to the soil as is, and are somewhere around the -6+14 (+20 in some cases) mesh range.

- **Coarse.** This is the size range used in making high potash granular mixed fertilizers, and is the size range now in question. It has shaken down to being somewhere smaller than 6 mesh and larger than 30 mesh, but where within this range is best is still a matter of much debate.

Reasons for this unstable state in the coarse size range are easy to find. Since granulation is still a relatively new field, some of the manufacturing problems associated with it are still unsolved. Although standard grade potash can be used in manufacture of 1-1-1 grades, for example, it leads to production troubles—recycle ratios soar—with the high potash grades such as 1-4-4. But using coarse potash for compositions like 1-4-4 causes varia-

tions in granule composition between large and small particles and can cause, too, a relatively unsightly product containing particles still almost all potash in some cases.

Also, no one granular product has become dominant, although two nutrient ratios—1-1-1 and 1-4-4—are the most popular. And, since choice of nutrient ratio depends on local soil requirements, and choice of grade depends on farmer acceptance, probably no single grade of granules will ever become so dominant that it would set the standard of potash acceptance. For example, if 5-20-20 were far and away the most popular granule, then potash producers would produce potash particles which would make the best 5-20-20 granules. Then only those additional high-potash products which could use the same size potash particles would be economical to make.

But such is not the case. Thus the problem is resolved into what size coarse potash should be used to get the best granules of the greatest number of low nitrogen-high potash mixes. No definitive answer has come forth because of the great number of variables which affect granulation:

- Ratio of nutrients.
- Concentration of nutrients.
- Formulation (raw materials and processing conditions) used to get the desired ratio and concentration.
- Particle size of final fertilizer product.
- Equipment in which the mix is granulated.
- Way in which the equipment is used. Some processors seem to have developed more know-how than others in this regard.
- Way in which the coarse potash is made. Briquetting followed by crushing and screening, agglomeration, flotation, and crystallization are some of the methods now in use.

Potash producers are concerned about the problem because they cannot be sure what size range to make their coarse product. Sometimes, granulators suddenly shift from what they have been using to some other size range they hope may be better. Hence, potash companies must watch closely their inventories of coarse material.

Granulators using different equipment or different ratios demand different size distributions, too. So the problem of making all these different products fast enough and cheaply

enough perplexes the potash people.

The granulators are not completely happy, either. With present technology, their products are not uniform, either in size or in composition. They could make them so, but not cheaply enough to compete. They therefore strike the best balance they can between cost of production and quality of product. It is with their attempts to improve this balance that the question of potash size distributions is allied.

Where all this will end no one can now tell. But granulated complete mixes are undoubtedly here to stay. Therefore, potash companies and granulators have to and do work together to get the best answers they can. It is to their mutual benefit to do so, and they have, in their opinion, almost—but not quite—solved the problem already. Meanwhile, research work by governmental agencies such as USDA and TVA, along with their own development work, is helping to pave the way to future progress.

Tolerance Fees

FDA raises fees; industry seeks legislation to abolish them

FDA'S LATEST INCREASE in pesticide tolerance fees sets the stage for a battle in Congress next year. On one side of the skirmish, FDA will point out that it is required by law to charge fees high enough to cover the cost of issuing tolerances. On the other, the National Agricultural Chemicals Association will contend the fees are inequitable and discriminatory against pesticide chemicals manufacturers. Rep. A. L. Miller (R.-Neb.) will voice the objections of NAC and the pesticides manufacturers, telling Congress the fees constitute "a tax to do business."

FDA sets tolerance fees and collects them from the pesticides industry under authority granted when the Miller Pesticide Amendment was passed. Since the amendment went into effect in February 1955, FDA has raised its fees twice. Its new scale, effective since September, boosts the cost of filing a petition for one tolerance level from \$1000 to \$2500. Fees for use of the same chemical at the same level on commodities not covered in the original petition are up from \$100 to \$250.

FDA Commissioner George P. Larrick observes that these increases are required by the language of the Miller Amendment, which calls for "the payment of such fees as will . . . be sufficient . . . to provide, equip, and maintain an adequate service . . ." The changes may have been hastened, however, by last winter's Budget Bureau order directing all government departments to charge fees covering the full cost of services they perform for the public. When this order came out, FDA thought its charges were sufficient. But after analyzing costs of administering the Miller Amendment for three years, the agency decided previous fees were too low. With the 150% increase, filing fees now pay the full cost of reviewing and studying tolerance petitions, searching the literature, and doing necessary laboratory work.

Legislation Needed

NAC thinks the cost of administering the pesticides law should be

The toxicological testing necessary before requirements of the Miller Amendment can be met are complicated. The oral (right), dermal (below, right), and inhalation (bottom, left) tests are only a small part of the range of toxicological tests necessary. These tests are expensive, add to the total expenditure necessary before a new pesticide is introduced. It is not surprising, therefore, that industry is vigorously protesting increases in the fees FDA collects before a tolerance is granted

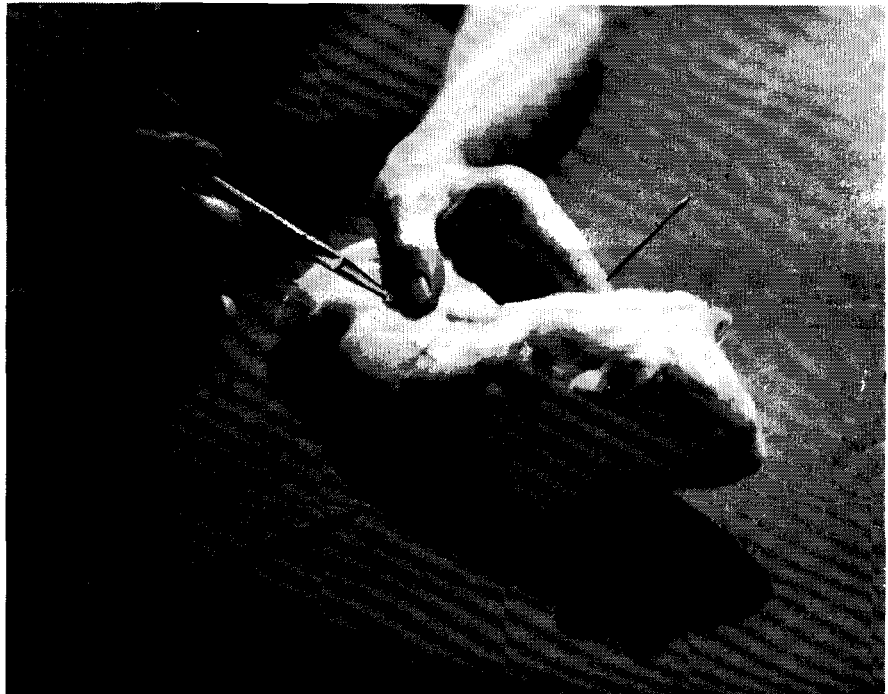
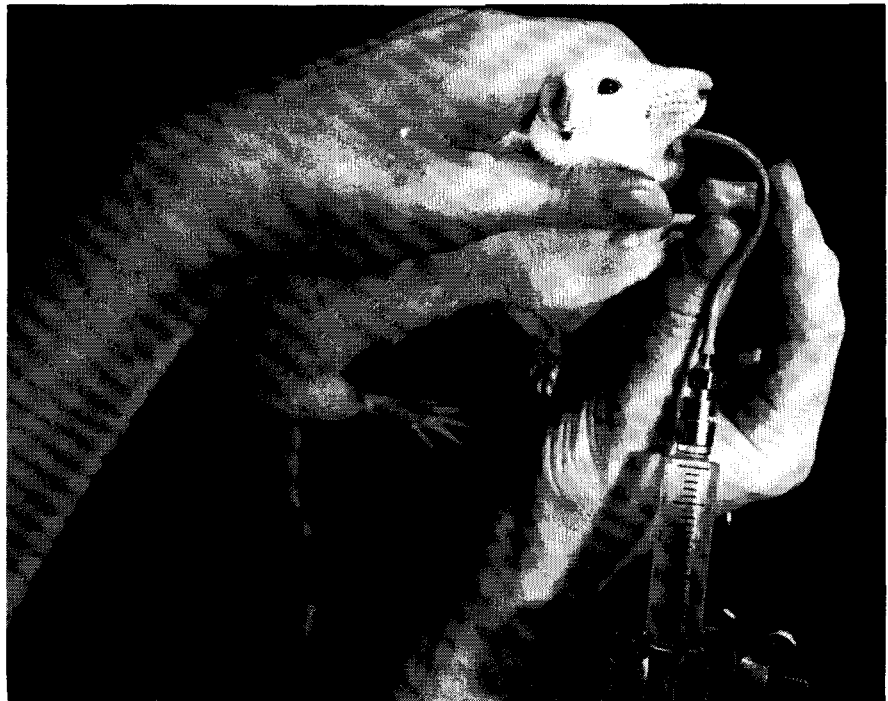
handled as an operating expense of the Government. Further, NAC says the Government has not put into effect in other areas its program aimed at making all agencies self-sustaining.

When Congress convenes next January, pesticides manufacturers will take up the fight against "intolerable tolerance fees" through NAC and their congressmen. Rep. Miller, author of the Miller Amendment, will carry the ball. He has already drafted a bill that would wipe out all administrative charges for pesticide toler-

ances. Gist of Rep Miller's thinking:

- FDA acts on pesticides to protect the public, not to confer a benefit on a chemical company.
- Making industry pay government costs is unjust when the law requires approval of pesticides before they can be sold or used.

Congress never intended to charge industry for the cost of regulating the use of chemicals in producing foods, says Rep. Miller. In support of this



FDA Filing Fees for Pesticides Tolerances

	Old	New
Original Petitions		
One tolerance level, up to nine crops	\$1000	\$2500
Two or more levels, up to 14 crops	1500	3750
Exemption	1000	2500
Temporary exemption or tolerance	1000	2500
Additions to Petition		
Each amendment	300	750
Each tolerance level lower than those covered in original petition	100	250
Each crop in addition to those covered in original petition	100	250
Rejected Petitions		
If technically incomplete (fine)	100	250
Supplement to technically incomplete petition	100	250
Refiling Petition		
Within six months after withdrawal	300	750
More than six months after withdrawal	Same as original petition	

attitude, he points to the food additives bill passed just before Congress adjourned in August (AG AND FOOD, October, page 725). That bill does not allow FDA to impose fees covering the cost of approving food additives.

Weigh Importance of a Tolerance

Because of the time factor and other uncertainties involved in getting action on tolerance fees, most pesticides producers say they have had to accept the higher costs in order to carry on their businesses. Some see the increases taking a larger bite of their research budgets. Others now weigh carefully the importance of having a tolerance. For example, if potential sales of a chemical for use on a given crop are limited, the profit to be derived from having the tolerance may not match the cost of getting FDA approval.

FDA fees on pesticides petitions have now reached amounts, Rep. Miller adds, that impose a burden on small companies. Even the larger ones must add the charges to the cost of marketing pesticides, raising still further the extra costs of developing new agricultural chemicals under the Miller Amendment. Thus, farmers and growers eventually are forced to pay more for materials they must use to protect their crops, concludes Rep. Miller. His proposed bill would eliminate all fees charged by FDA on tolerance petitions. However, a company would still pay actual committee costs if it requested FDA to appoint an advisory group to study a petition.

Plant Food From Sewage

Use of sewage sludge as fertilizer tops 200,000 tons a year. Cities content to lose money on it

WHEN FERTILIZER MANUFACTURERS discuss business they usually come to the gloomy agreement that profits are far too low. But there is one segment of the plant food industry that openly admits it loses money on every pound of material it sells, and is quite content to do so. Despite the fact that its profits are nonexistent, this group knows what it is doing.

This year the use of activated sewage sludge as a fertilizer will exceed 200,000 tons. The three cities which account for most of it will lose upwards of five dollars on each ton produced. But they figure the money is well spent. At the moment, converting sewage solids to fertilizer is the cheapest route to total disposal of municipal wastes.

"Total" is the key word. Stockpiling the solids from a disposal plant, or dumping them into a handy river or lake is only half an answer to the disposal problem. Though it costs more to process sludge into a salable product than the product brings in return, the operation is easily justified in terms of cleaner streams and fewer piles of refuse.

Selling sludge as fertilizer may not be the ultimate answer. For instance, Chicago's sanitary district is experi-

menting with a wet-combustion process that might be an even better way to get rid of wastes. But in the foreseeable future, production and use of sludge fertilizer will probably continue its slow but steady growth.

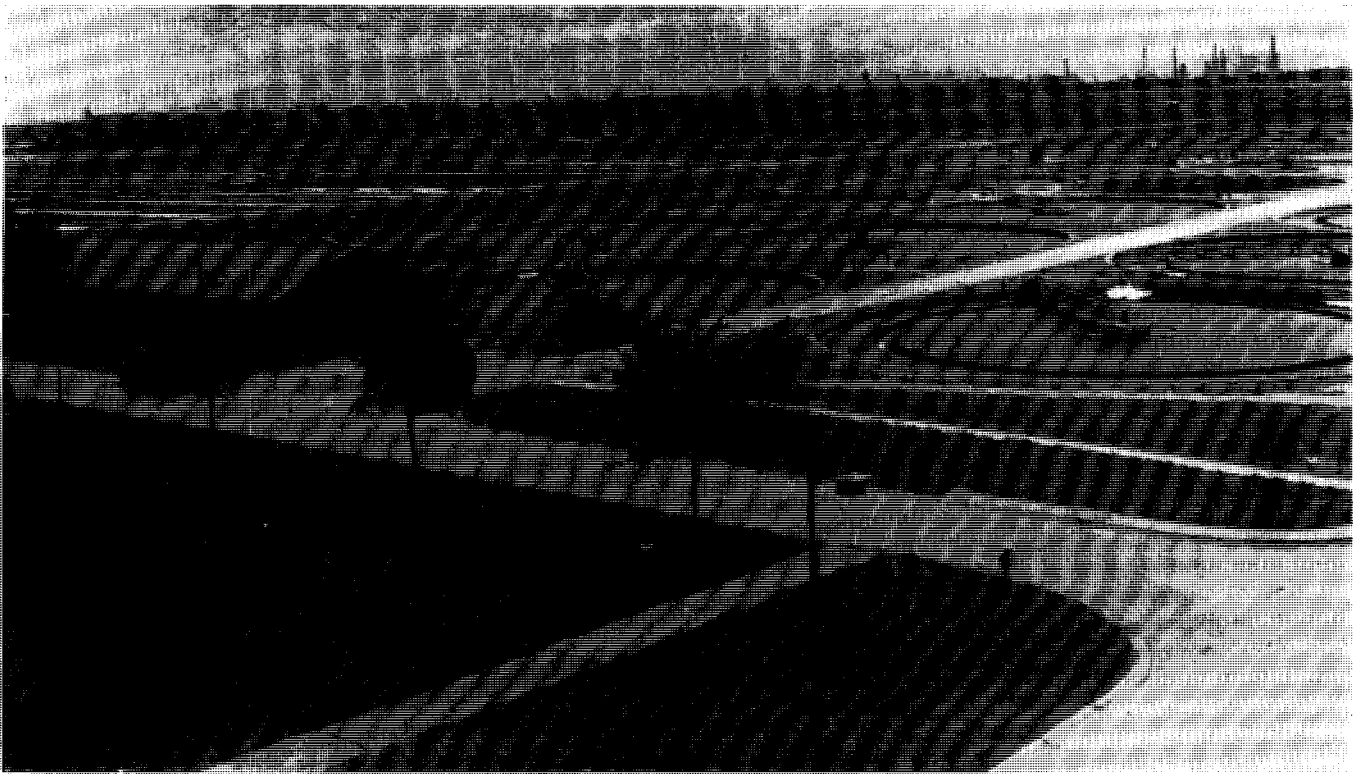
Activated sludge, the heat-dried residue from aerobic digestion of municipal waste, entered the agricultural scene as a fertilizer during the 1920's. Annual consumption rose; USDA figures show that about 100,000 tons of activated sludge was used last year for direct application. Based on the output of cities that make activated sludge, another 100,000 tons a year is used in mixed fertilizers. Other types of sludge with lower nutrient values account for another 35,000 tons.

The Sanitary District of Greater Chicago is the largest producer, with a daily output of 450 to 500 tons. Last year this added up to 131,000 tons, which brought the district some \$2 million in revenue. The Milwaukee Sewerage Commission can make 180 to 240 tons per day, and about 70,000 tons is sold annually under the trade name Milorganite. Third big producer is the City of Houston which turns out about 16,000 tons per year under the name Hou-Actinite. Besides these cities which distribute beyond their own areas, many others sell or give away low-grade sludge locally.

Chicago feels that selling fertilizer is not a proper activity for a sanitary district. At present, H. J. Baker is exclusive distributor of Chicago sludge. Houston is one step closer to marketing. It sells in bulk to formulators who package the sludge and sell it as is, or use it as an ingredient in mixed goods. Milwaukee has gone all the way. It produces, packages, and distributes at the retail level. Milwaukee was the first city to market sludge nationally, and bags of Milorganite are now a familiar sight on the shelves of garden supply stores everywhere.

Demand for activated sludge is quite good, and prices are firm. From June through September Houston quotes a price of \$2.75 per unit of nitrogen per ton, plus 50 cents a unit of available phosphoric acid. For October to May the nitrogen price goes up to \$3.00 a unit. H. J. Baker, selling Chicago material, lists prices in the same range. For average analysis sludge, these figures mean that cities receive from \$15 to \$18 per ton. The exception is Milwaukee, which prices its product higher to pay for packaging and distribution. In 1957 Milwaukee got an average of \$35.81 for each ton of Milorganite sold.

It's no secret that cities make no money on sludge. But making money



A view of the activated sludge plant at the southwest sewage treatment works in Chicago

is not the object. It cost Milwaukee \$42.57 per ton to make sludge last year, giving a loss of \$6.70 per ton. Other cities are less explicit with cost figures, but all admit that their activated sludge operations lose money, on paper. One man says that today a municipality cannot make and sell activated sludge without losing at least \$5.00 a ton; the usual figure is quite a bit higher.

But that is just one side of the story. The alternative to making activated sludge is to handle sewage solids with a less expensive digestion process that would reduce operating costs, but would not turn out a salable product. Many cities have low-cost means available for disposing of digested sludge. But others do not, and making fertilizer can be justified when the cost of getting rid of digested sludge exceeds the amount that would be lost in an activated sludge plant.

Digested sludge is the end product of anaerobic digestion of sewage solids. Activated sludge is made by passing air rapidly through sewage in the presence of aerobic bacteria. The final product contains about 40% inorganic matter; the rest is dead bacteria.

Because there are many minor differences in the way various cities treat sewage, there are wide differences in the fertilizer value of various sludges. The nature of sewage fed to the disposal plant also affects the plant food content of the sludge. For instance,

an industrial area might turn out a sludge with less P_2O_5 than a residential zone where sewage contains large amounts of household detergents. A typical activated sludge analyzes 5.6-5.6-0.4 on a dry basis. Digested sludges run much lower; data from the Federation of Sewage Works Associations show the average analysis for digested sludge to be 2.0-1.1-0.2. For comparison, farm manure runs about 1.2-0.6-1.2.

As a fertilizer, activated sludge has a considerable edge over digested materials. Besides its higher plant food content, its nitrification rate in soil is much faster. Tests show that after 16 weeks only 18% to 25% of the nitrogen in digested sludge is available to plants, compared to 50% to 60% for activated. Also, activated sludge is usually heat-treated, and therefore presents no sanitary problems. Digested sludge sometimes contains living organisms, and must be used with some caution. Generally speaking, digested sludge is safe for use on any crop except vegetables which are eaten raw.

Sludge fertilizer winds up mainly on lawns, golf courses, and flower or vegetable gardens. On crops, it is used in addition to, rather than in place of, chemical fertilizers. Milwaukee's Milorganite is used mostly on grass. Business is split about 60-40 between home use and use by golf courses and institutions. Houston says about one

third of its production is used on rice, and other large amounts go to citrus and vegetable farms in Florida. Chicago thinks lawns are its biggest outlet, while sizable amounts are shipped south for use on vegetables.

Per unit of plant food, sludge is more expensive than most other fertilizers. But users pay the premium because of the material's value as a soil conditioner. Sludge also contains a long list of trace elements—a good selling point even if it is hard to pinpoint the value of these minerals. Since sludge is insoluble in water, its plant food value is released slowly to soils, and it is less likely than soluble materials to be washed off a lawn by a heavy rain coming right after application.

For the future, use of sludge fertilizer will probably see no spectacular booms or busts. The cities that make activated sludge are well established in the business. As disposal problems become more acute other municipalities will look to sludge fertilizer as a solution. The city of Dallas, for example, is studying potential markets, plans to build a fertilizer pilot plant. But since an activated plant is technically more difficult to operate than a plant that just digests sewage, and since the activated process has higher operating costs than other disposal methods, there is no reason to expect any major increase in activated sludge output.