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THE INCORPORATION OF TRACE
ELEMENTS, INSECTICIDES AND
HERBICIDES IN COMPOUND FER-
TILISERS

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Fisons Limited.

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THE INCORPORATION OF TRACE ELEMENTS, INSECTICIDES AND HERBICIDES IN COMPOUND FERTILIZERS

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(1) INTRODUCTION

The increasing use of trace elements, insecticides and herbicides by farmers is one of the recent advances in agriculture. In general, trace elements are still applied to the soil or to the growing crops as straight dressings of an inorganic chemical or as a solution of a soluble inorganic salt or complex. Insecticides and herbicides are more usually applied as a solution (often in an expensive organic solvent or as an emulsion) or as a dust which consists of an unreactive carrier on which the 'additive' is sprayed or with which it, if a solid, has been finely ground and admixed.

In all cases, the high first cost of such material is increased for the farmer by the need for a further treatment of the soil or crop in specialized equipment requiring capital and maintenance.

Compound Fertilizers can often be used as carriers for such materials and hence if the application is agronomically acceptable, it is possible to reduce the total cost to the farmer by applying the 'additive' at the same time as the main plant foods. It is proposed to set down in this paper the theoretical considerations which will influence any decision to manufacture what we in the United Kingdom know, generally, as "Fertilizers plus Additives". In addition, the paper gives some indication of the effect of such additives on manufacturing conditions. It indicates the effect on concentration of the fertilizer by incorporating such additives, the uniformity of composition, the effect on quality and storage properties of the new fertilizers.

It is assumed in this paper, that there is no agronomic objection to the application of the 'additive' to the soil in conjunction with the fertilizer.

(2) GENERAL CONSIDERATIONS

(a) Trace Elements

Trace elements will usually be refined inorganic ores or salts of inorganic or organic acids and bases in which the trace element required is present in the base. The latter will always be soluble in water and usually heat stable. The amount required per acre may be small or large but the concentration of the element in its parent material will probably be low, necessitating fairly large additions to the mixture which will affect the NPK concentration and rate of

application of the combined fertilizer plus 'additive' to the soil. Agronomically, the quantities of trace elements required in relation to NPK content may vary considerably. Nearly all of them can be expected to undergo chemical reaction with the other ingredients of fertilizers, possibly leading to reduction in water solubility of one or more constituents, loss of the 'additive' or of a plant food, especially in a granulation process, where heat is present. They will have no toxicity values to humans. Trace elements which may be added to fertilizers will include boron, cobalt, magnesium, copper, zinc, manganese, molybdenum, sodium and iron.

(b) Insecticides

These are nearly all straight organic materials which may be liquids or solids. Solutions in organic solvents, water emulsions or mixtures on inert bases can be readily obtained. Many of them are both toxic to humans and heat sensitive. They are required in very small concentrations on the fertilizer. The range of products is a large one, but Aldrin, Dieldrin and Heptachlor are now in common use as additives to fertilizer, while there are several marketed insecticides for straight application based on malathion.

(c) Herbicides

The number of herbicides available to the farmer is large but those in commercial use are mainly salts of organic acids with inorganic bases; the acid radicle being the effective part. As such, they are water soluble to a minor or major degree or can be produced as a water solution or readily emulsified by choice of a suitable inorganic base and/or emulsifying agent. In general, such solutions are either non-toxic or of low toxicity to humans. Fertilizers containing such additives are unlikely to be toxic to humans in view of the low concentrations in which such materials are used. They are, however, not so highly selective in their action that damage to other plant life will not occur. Therefore it is essential to avoid contamination of other fertilizers. In this group, we can list M.C.P.A. derivatives and allied compounds, maleic hydrazide and tecane.

(d) Methods of Manufacture

Additives or a mixture of an additive with an inert base may be incorporated as a solid material in either a granulation process or in dry powder mixtures either during or after manufacture of the fertilizer. Alternatively, if available in the solution form, consideration can be given to incorporation of additives by spray treatment of the fertilizer either during or after its manufacture.

Before deciding on a form of manufacture for any proposed fertilizer the following main points should be considered:

(i) The type of fertilizer required

Granular or Powder.
Nature of ingredients.
Production process.

(ii) Amount of Additive

If the percentage is less than 2% of the total mixture, then unless the fertilizer manufacturing process is one in which a slurry stage is produced, it is possible that distribution within the mixture will be poor if the additive is included as a raw material in the initial mixture.

(iii) Heat Sensitivity of the Additive

This may be such as to forbid the passage of the additive through the dryer and is, therefore, a limitation in the production of granular compounds.

(iv) Concentration of the Fertilizer

The amount of additive may be such as to necessitate a reduction in the concentration of NPK plant foods. This point may decide the form of the additive if the latter is itself available at different concentrations.

(v) Rate of Application of Fertilizer

Generally, additives are required at certain definite concentrations per acre for control purposes. If however, the fertilizer is one which is applied at various and widely different rates of application, then it would not be possible under present day conditions where fertilizer production is a continuous process, to manufacture a number of NPK fertilizers in which the concentration of additive is varied. Such conditions would favour the use of a method of incorporation of additives after manufacture.

(vi) Chemical nature of the Additive

Can it be obtained economically as a solution in a water or organic solvent and/or can it be emulsified? Alternatively, can it only be obtained economically as a solid? If so, can it be in the form of a fine dusting agent or must it be admixed with an inert base? The latter form would necessitate consideration of the concentration of the additive and hence the quantity to be applied to the fertilizer.

(vii) Chemical Reaction to the Additive with the Basic Ingredient of the Fertilizer.

The presence of the additive may cause undesirable chemical reactions; for example, the possible loss of water soluble P_2O_5 . Alternatively, the base fertilizer may destroy the additive, and hence possibly limit the time between manufacture and application to the soil.

(viii) Toxicity

Toxicity of an additive to the operators in the factory, to the workers in the field or phytotoxicity to certain crops may dictate the method of manufacture.

(ix) Release of Additive

To do its work, must the release of the additive be rapid and hence its presence more desirable on the surface of the fertilizer particles to which it is added?

(x) Effect on Bagging Materials

(xi) Effect on Storage Life

(3) PRACTICAL EXPERIENCE.

(a) Trace Elements.

(1) Boron.

We have added Boron in the form of borax to mixtures containing Muriate of Potash, Triple and Single Superphosphates, before granulation. On an NPK analysis of 10:10:18 it is standard procedure to add 0.67% B_2O_3 in the form of borax. This is equivalent to approximately 40 lb. of borax per ton of fertilizer for use on sugar beet. Addition of borax is made to the mixture prior to granulation. The quantity of boron is such that addition as a solution after manufacture of the granules would add a quantity of water which would lead us to doubt the keeping qualities of the stored fertilizer.

The presence of ammonium salts liberates water of crystallization from borax and hence its addition as a powder to the outside of granules after manufacture is undesirable. An alternative method of manufacture of boronated fertilizers, is the dusting of granules which have been given a light spray of water with sodium pentaborate, higher in B_2O_3 content and with a lower moisture of crystallization than borax.

There is little information of an agronomic nature available as to whether it is necessary for the crop for the boron to be in a water soluble form. If a water insoluble form of boron was acceptable, then alternative methods would include the addition of calcium borates, boron silicates or boron frits.

The processing of mixtures containing boron through a standard rotary granulation process aimed at producing a water soluble P_2O_5 fertilizer and in which steam and water are used in the conditioner, presents in itself little difficulty, a good physical quality granule of excellent storage properties resulting. A study of the chemical changes which occur is however, more interesting. Tests carried out on two grades of fertilizer, one grade at each of two Works showed that -

- (a) loss of boron at the conditioner can be expected.
- (b) during the process a change occurred in the form of the boron from an entirely water soluble material to one in which only part was soluble.

Relevant details are given in Appendix I.

Samples of boronated NPK fertilizers prepared from Sulphate of Ammonia, Muriate of Potash, Triple and Single Superphosphates and Borax at various Works were analysed. The significant details of the analyses are given in Appendix II. The picture is neither clear nor consistent but it would appear that -

- (a) where a loss of boron has occurred, then the ratio of water-soluble P_2O_5 /total P_2O_5 is that which would be expected in a similar NPK Compound without boron.
- (b) where there was no loss of boron, the water-soluble P_2O_5 /total P_2O_5 ratio is markedly reduced.

The subject requires further investigation but it may be necessary to give careful consideration to processing technique if it is necessary to give guarantees for both the water soluble P_2O_5 and boron contents of a fertilizer.

(11) Magnesium.

Hypomagnesaemia in cattle is known to be associated with low magnesium levels in herbage. Attempts have been made to raise the magnesium levels by application of magnesium in the fertilizer dressings. The quantity of magnesium required is fairly large and a typical analysis would be 4% MgO on an NPK fertilizer containing 8% Nitrogen, 6% P₂O₅ and 10% K₂O.

The following raw materials are available as sources of magnesium.

Epsom salts.	16.4% MgO
Kieserite.	28.0% "
Magnesite/Sylvanite/Kainit.	5.5% "
Dolomite.	21.8% "
Carnalite.	8.8% "
Patent Kalimagnesia.	10.0% "
Magnesium oxide.	98.0% "
Magnesite.	46.5% "
Dolomite Marl.	18.2% "
Magnesium phosphates.	various.

For incorporation in NPK fertilizers we can dismiss magnesium oxide as it will liberate ammonia. Dolomite, Magnesite and Dolomite Marl are carbonates and will cause reversion of water soluble P₂O₅. Magnesite/Sylvanite/Kainite, Carnalite and Patent Kalimagnesia are all so low in MgO content even after allowing for the K₂O associated with them, that there would not be sufficient space to formulate a reasonable compound. Magnesium Pyrophosphates are insoluble in water and the acid phosphates of magnesium only sparingly so. The ratio of MgO/P₂O₅ in acid phosphates is such that their incorporation as sole sources of both MgO and P₂O₅ would not give a satisfactory product. The acid salts and the full salt would react with ammonia and would not supply water soluble P₂O₅. We can, therefore, consider only Epsom salts or Kieserite as a source of MgO and Kieserite is by far the cheaper of the two materials. Patent Kalimagnesia would be of use if it was desired to eliminate the chlorine ion from the compound fertilizer.

The quantity of MgO carrier required is such that we cannot consider its addition at any other point in the granulation process than with the incoming raw materials. Its incorporation inevitably limits the NPK plant food concentration which can be reached in the final product.

Our production experience with the incorporation of Kieserite into NPK fertilizers has been confined to limited relatively small scale studies with inconclusive results. The product was a hard granule with good keeping qualities. The plant food analyses tended to show greater variation than with similar NPK compounds, but no reversion of water soluble P₂O₅ was detected.

(3)

(iii) Cobalt

The addition of cobalt in the form of its sulphate as a preventative for sheep pine is occasionally requested. The levels are of the order of 8 lbs. hydrated cobalt sulphate per ton 20% P_2O_5 granular superphosphate, or 10 lbs. per ton of a 10% N., 10% P_2O_5 , 18% K_2O granular compound.

Such quantities are too small to be incorporated in the raw materials and to guarantee even distribution. On the other hand, the solubility of cobalt sulphate with seven mols. of water of crystallization is approximately 60 gms/100 gms water at 30°. Hence there is no difficulty in making up a 3.3% solution in the cold. The quantity to be sprayed per ton is thus of the order of 2½ gall/ton. Our experience is that such a quantity (see under Aldrin below) can be sprayed to give uniform distribution and such a solution is readily absorbed by the granules. The moisture content of the granules will be raised by about 0.89%, but it is our experience that this addition of moisture even to the surface of the granules providing that it is in the form of a concentrated solution, does not cause setting of the fertilizer providing that ammonium nitrate is not one of the ingredients. (see under Tecane below).

(iv) Manganese.

We have not prepared compounds containing manganese on the full plant scale. Manganese sulphate is available as the monohydrate at a purity of about 99% and hence an MnO content of 32%. Alternatively, there is a commercial product consisting of a mixture of manganese sulphate together with various manganese oxides and having a Mn content of between 28% and 30%, of which 17% is soluble in water.

Pilot plant quantities of a non-nitrogen fertilizer containing 16% P_2O_5 and 16% K_2O with quantities of Mn varying between approximately 1% or 8% have been made from manganese sulphate, muriate of potash, triple and single superphosphates. The products produced appreciable loss of W/S P_2O_5 directly proportional to the quantity of Mn added.

<u>% Mn.</u>	<u>W/S P_2O_5 / Total P_2O_5 as %</u>
3.36	96.1
4.71	95.8
6.52	94.9
8.18	93.7

Hence addition of 1% Mn can be expected to decrease % conversion of P_2O_5 by 0.6%.

(v) Sodium

In the United Kingdom, kainite is added to sugar beet as a top dressing to supply sodium to the crop. Since sodium can replace potassium to some extent for this crop, there would appear to be little objection to formulating a mixture to include the sodium ion.

/Continued

(3) (v) Sodium /Contd.

As a source of sodium and to a lesser extent, potassium, the use of kainite causes a reduction in the concentration of the fertilizer. It is preferable to use common salt as the sodium donor, and to take the required balance of potassium from muriate, since the presence of chloride ion does not appear to be harmful. Even so, some reduction in concentration of the fertilizer must occur.

Such compounds have not been manufactured by us.

(b) Insecticides

(i) Aldrin

Aldrin (1, 2, 3, 4, 10, 10 - hexachloro - 1, 4, 4a, 5, 8, 8a - hexahydro - 1, 4 - endo, exo 58 - dimethanonaphthalene) is available commercially as a powder with a very high degree of purity of its active ingredients. It can be admixed with many types of inert fillers and hence extended to give the required quantity of aldrin in any convenient bulk. The main use of aldrin is for control of wireworm in potatoes, or wheat bulb fly in cereals. Quantities of the order of $1\frac{1}{2}$ lbs./2 lbs. per acre are required, and typical application rates would be 4 lbs./ton of a 10:10:18 compound or about 6 lbs./ton of a 6:15:15 compound. The material is heat sensitive, and it is undesirable to pass aldrin through the drier of a granulation plant. Moreover, the quantity per ton of fertilizer is too small to guarantee uniform distribution if so incorporated. It should contact the soil as soon as possible and should therefore be confined to the surface layers of the granules.

Fortunately, aldrin can be readily dissolved in comparatively cheap solvents of a mixed paraffinic nature, and concentrations of 32% W/V can be readily achieved.

Our standard procedure is to spray granular fertilizers which are either falling in a stream from a belt or rolling in a small drum immediately prior to bagging. A convenient place to carry out this operation is in a blanked-off section of a rotary screen above a bagging plant hopper. This procedure eliminates the need to store in the Works bulk heaps of material treated with a slightly toxic material. No explosive concentrations caused by the evaporation of the solvent have been detected in plant units. No loss of activity has been detected in treated material stored for 15 months. Freshly treated granules cause slight attack of rubber belts due to the presence of the solvent.

Appendix III gives estimates of the degree of uniformity of treatment which can be obtained.

Treatment with aldrin solution does not increase the tendency of a fertilizer to set in either a bag or a bulk heap. However, it is necessary to give some attention to the construction of the paper bag. The presence of the paraffinic hydrocarbons cause leaching of any bitumen present in the paper plies, and a bad staining results. Polythene coated papers are not resistant to the solvent and a stain again results. We have found that the most satisfactory arrangement is to have a wax coated paper in contact with the treated fertilizer and then to have the normal bitumen outer plies to prevent entry of moisture to the fertilizer. Where leaching has occurred, we have been unable to detect loss of aldrin in the fertilizer.

/Continued

(3) (1) Aldrin /Contd.

Powder mixtures can be treated in the same manner.

(11) Heptachlor and Dieldrin

Heptachlor (3, 4, 5, 6, 7, 8, 8 - heptachloro - 1, 2, 3, 3a, 7a - tetrahydro - 4, 7 - endo - methylene indene) and Dieldrin (1, 2, 3, 4, 10, 10 - hexachloro - 6, 7 - epoxy - 1, 4, 4a, 5, 6, 7, 8, 8a - octahydro - 1, 4 - endo, exo - 5, 8 - dimethanonaphthalene) are available and can be applied in the same way as aldrin. The normal solvent for dieldrin is xylene.

Except for pilot plant stage work, we have no experience of the use of these two insecticides, but as the quantities required are similar to those of aldrin, the effect of their use can be forecast. For example, a consideration of the nature of the solvent will indicate production hazards and effects on bags etc.

(c) Weedkillers

(1) Tecane

Tecane is the sodium salt of trichloroacetic acid, and is used to give control over wild oat in sugar beet. The general application rate is about $12\frac{1}{2}$ lbs. of 100% salt per ton of a 9, 9, 15 fertilizer. In considering the incorporation of such a material in a fertilizer, the most important point is the necessity to avoid contamination of manufacturing plant and other fertilizers. We are therefore forced to consider only methods of application after manufacture and immediately prior to bagging. A solution method, as with aldrin, is an obvious choice. Alternately we could consider dusting the fertilizer with the finely ground salt. The quantity is such, however, that distribution might not be uniform and, while the material is not toxic to humans, it can be considered as likely to give rise to skin complaints.

In the cold, a 30% W/W solution in water can be obtained (sp. gr. 1.19). A solution such that $12\frac{1}{2}$ lbs. tecane is contained in $3\frac{1}{2}$ gallons solution is convenient as we have seen that uniform distribution can be obtained at lower spray rates than this. Unfortunately this necessitates the addition of water, which will increase the moisture content of the fertilizer by 1.3%.

In adopting this procedure, our main concern was the effect of the increased moisture content on storage life. Tests were conducted on base granular fertilizers (prepared from sulphate ammonia, muriate of potash, single and triple superphosphates at about 1% moisture content) and showed that after 6 months in bags, stored 20 bags high, and with applications of tecane solution at rates equivalent to both 10 lbs. and 15 lbs. tecane/ton fertilizer (i.e. equivalent moisture additions of 1.04 and 1.56%) no serious effect on condition was apparent. The details of the test and the results are given in Appendix IV. Previous correlations carried out by us on this type of test, assure us that with results such as these, the farmer is unlikely to experience difficulty in handling the material as long as it is applied up to 2 months after treatment.

(3) (11) Substituted Phenoxycetic Acids.

This group of compounds is a wide one, and I do not propose to discuss each one, but to confine my remarks to general terms, and to illustrate them where possible.

As a group, the more common sodium salts which are used as the basis of solutions for application to grassland as weedkillers, are not readily soluble in water. The solubility can be increased by preparing the potassium compounds. The basic acids are not heat stable and every care must be taken to ensure that they do not contaminate plant or other fertilizers. The acids are solids at room temperatures and are seldom prepared and marketed as such. Hence the obvious method of incorporation in fertilizers is again by spray treatment of a solution after manufacture of the fertilizer. The quantity of weedkiller required per acre is not large, but this coupled with the low solubility of the salts and the low fertilizer application rates on grassland militates against a sound production procedure. For example, limiting conditions for the preparation in the cold by spray treatment of a granular inorganic fertilizer containing a weedkiller occur if the weedkiller is available as a 22% W/W solution, the fertilizer application rate is 2 cwt./acre and the quantity of weedkiller required is 1.5 lbs./acre.

Attention must also be paid to the ingredients in the fertilizer mixture. We have incorporated 2, 4 - Dichlorophenoxyacetic acid into a powder lawn fertilizer containing organic materials. Our procedure was to use one of the organic materials in the mixture as an extender for a hot solution of the sodium salt of 2, 4 - D. The application rate was 6 lbs. of the sodium salt/ton fertilizer. The solubility is low (27.9 gms/100 mls. water at 64° C)

and the consequent increase in moisture content of the carrier led to bacterial activity. The use of an inorganic ingredient as a carrier was not successful, nor was the spraying of the whole mixture with the solution, as the resultant increase in moisture content of the mixture led to bacterial activity, mould growth on the exterior of packages, package failure of both cardboard cartons and paper lined hessian bags.

However, by suitable choice of the selectivity of the various members of the group, a solution of salts of mixed acids can usually be found which can be applied by solvents, especially if the fertilizer application level is above 5 cwt./acre.

(4) ACKNOWLEDGEMENT

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A P P E N D I X I

BORAX CONTENT OF SAMPLES OF FERTILIZER TAKEN
AT DIFFERENT STAGES DURING GRANULATION

Works	1	2
Compound	5:11:6	9:9:15
$\%$ total borax content on dry basis: raw materials mixture ex conditioner ex drier ex product belt	1.82 1.35 1.11 1.09	1.82 1.73 1.94 1.66
$\%$ water soluble borax content on dry basis: raw materials mixture ex conditioner ex drier ex product belt	1.82 1.28 1.04 1.03	1.82 1.33 1.22 1.39
$\%$ insoluble borax content on dry basis: raw materials mixture ex conditioner ex drier ex product belt	nil 0.06 0.07 0.07	nil 0.41 0.73 0.27
Losses of total boron (as $\%$ of input) at conditioner at conditioner + drier at end of production	25.8 39.0 40.1	4.9 6.5 6.5 8.8
$\%$ "conversion" of borax (water soluble : total) in raw materials mixture ex conditioner ex drier ex product belt	100 94.7 93.5 94.7	100 76.5 62.2 87.0

1. Results from Works 1 are the mean of 9 results and for Works 2 the mean of 8 results.
2. At Works 1, total, water soluble and insoluble borax were determined separately, while at Works 2, the insoluble borax is the difference between total and water soluble borax.
3. Moisture content of the product only was determined at Works 2. Moisture contents of ex conditioner and ex drier samples at Works 2, have been taken at 6.5% and 1.7% (moisture content of product was 1.3%).

APPENDIX II

TOTAL BORON CONTENT AND CONVERSION OF P₂O₅ IN FACTORY

MADE SAMPLES OF BORONATED FERTILIZER

Compound	Works	% B ₂ O ₃		W/S P ₂ O ₅ as % total P ₂ O ₅	Free Acid (% P ₂ O ₅)
		added to mix.	Found in Product		
10:10:18 {	3	0.65	0.46	94.5	0.28
	1	0.65	0.69	84.7	0.14
	4	0.67	0.68	89.0	0.02
	4	0.67	0.53	91.9	0.24
5:12½:12½ {	4	1.39	0.75	92.3	1.26
	4	1.39	1.15	93.2	1.3
	1	0.81	0.94	93.7	2.06
	1	0.81	0.84	93.97	2.3
6:15:15 {	4	1.39	1.04	90.1	1.10
	4	1.39	1.28	90.9	1.14
5:11:6	1	0.67	0.87	83.0	0.92

A P P E N D I X III.

UNIFORMITY OF ADDITION OF ALDRIN BY SPRAY TREATMENT

A number of 1 cwt. bags of treated fertilizer were selected at random, each bag was sampled by riffing, and the aldrin content of each sample determined.

Works	No. of bags sampled	% Theoretical content	% Found		
			Mean	Max.	Min.
3	8	0.178	0.19	0.20	0.18
5	6	0.178	0.235	0.312	0.135
5	6	0.178	0.189	0.204	0.171
6	4	0.178	0.167	0.215	0.148

APPENDIX IV.

SETTING CHARACTERISTICS OF TECANE TREATED FERTILIZER

In measuring the tendency of bagged fertilizers to set, our standard procedure is to stack the bags in columns twenty bags high, and to treat them at given time intervals. Testing procedures consist of dropping each bag once on its flat face from a height of 3 feet. The bag is then lifted on to a 2" square mesh flat screen and the bag out open, and the paper withdrawn. Any material remaining on the screen is weighed and this weight is expressed as a percentage of the total weight of fertilizer in the bag. This percentage is called the "caking index". Normally the bottom bag of each pile is ignored and only bags 10 - 19 counting from the top of each column are tested. The mean caking index is calculated.

Normal granular fertilizer was treated with 30% W/W water solution of tecane, at rates to give 10 and 15 lbs. tecane/ton. Sufficient material was stacked to allow tests at 1, 2 and 6 months after manufacture. Three replicates were carried out at each test period.

The following table gives the results obtained:

Treatment	Replicate No.	1 month			2 months			6 months		
		Mean	Max.	Min.	Mean	Max.	Min.	Mean	Max.	Min.
Control Untreated: 9:9:15	1	NO	SET	0	NO	SET	0	NO	SET	0
	2									
	3									
9:9:15 + 10 lbs. tecane/ton fertilizer	1	3.18	8.5	0	5.85	11.92	0	5.2	13.8	0
	2	0	0	0	2.05	17.59	0	5.4	16.5	0
	3	2.22	7.1	0	4.14	18.18	0	0.9	2.7	0
9:9:15 + 15 lbs. tecane/ton fertilizer	1	3.83	11.5	0	15.16	18.52	5.61	2.9	6.6	0.94
	2	1.65	3.7	0	2.89	10.81	0	6.1	12.6	0
	3	4.55	9.5	0	5.84	10.9	3.70	2.4	13.5	0