crops are susceptible to salt injury, and cost of labor for repeated fertilizer application is high would also be good candidates.

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MICRONUTRIENT GLASSES

Properties and Use of Micronutrient Glasses in Crop Production

E. R. HOLDEN
Soil and Water Conservation Research Division, Agricultural Research Service, U. S. Department of Agriculture, Beltsville, Md.

N. R. PAGE
Department of Agronomy and Soils, Clemson Agricultural College, Clemson, S. C.

J. I. WEAR
Department of Agronomy and Soils, Auburn University, Auburn, Ala.

The chemical nature and particle-size characteristics of commercially available micronutrient glasses influence the nutrient content of crops. Suitable glasses release micronutrients more steadily than slowly soluble mineral substances, thus far investigated, and have more ability to minimize seasonal variation in elemental content of a crop. This control of release increases freedom to vary application, currently a principal factor limiting use in coarse-textured soils and in commercial fertilizers. Correct levels of reactivity, as measured by nutrient release in ammonium acetate, and pattern of release in soil are considered in terms of nutrient uptake.

Interest in slowly soluble micronutrient materials developed after 1927 when Brenchley and Warington (*4*) demonstrated that boron is essential to plant growth. The high sensitivity of plants to this element was generally known because boron, as an impurity in certain potassium fertilizers, had caused heavy crop damage (*23, 24*). Glass products (*15, 17*) as well as mineral substances (*6*) were investigated during the 1930's. The results indicated that slowly soluble carriers would be superior to soluble forms under some conditions. However, significant use did not come until about 25 years later. Undefined causes of variation in nutrient content of crops presented much difficulty in evaluating vegetative data. Experimental work was also hindered by a lack of suitable test materials. These obstacles have since been overcome and the information now available i sufficient tc permit commercial applications.

Basic Problem

Coarse-textured acid soils, usually low in soluble nutrients, constitute the most compelling reason for development and use of slowly soluble carriers. Soluble nutrients when applied to sandy soils increase the nutrient content of a crop in early growth more than they do on loams or clay soils. Consequently, toxic concentrations are reached in the crop, especially during germination, at relatively low levels of application. Later in the season, after the applied nutrients have been leached from the root zone (*14, 19*), deficiencies may develop. With seasonal variation in nutrient level being so great, there is little freedom to vary application, making it difficult to adjust the level of a single application to meet the needs of crops throughout the growing season.

The basic problem is inadequate control of dissolved supply by coarse-textured soils. The soil, being low in colloidal matter, has insufficient capacity to buffer solution concentration by sorption processes. In addition, capillary size is large, and water solutions move down-

Table I. Micronutrient Content of Commercially Available Glass Carriers

Glass	Nutrient Content, % Fe	Mn	Cu	Zn	Mo	B
501[a]	12.3	4.9	2.0	4.0	0.13	2.0
502[a]	3.9	9.7	2.0	4.0	0.13	2.8
505[a]	...	18.6	...	...	...	2.6
513[a]	...	5.2	...	...	...	2.8
176-E	...	...	...	...	...	6.3
176-F	...	...	...	...	...	6.0

[a] "Guaranteed analysis" supplied by manufacturer.

ward in the soil carrying dissolved nutrients away from the root system. The purpose of a slowly soluble carrier is to maintain dissolved supply at adequate levels under these conditions. By weathering gradually in the soil, the carrier furnishes micronutrients in small amounts to replace losses as they occur. Soil supply need not be constant, but it should be held within proper range continuously (*2*, *27*, *29*).

Use in Commercial Fertilizers. Separate application of micronutrients increases operating expense and otherwise complicates crop management. Difficulties of this kind can be avoided by using a fertilizer mixture containing all necessary nutrients. A complete fertilizer also offers more uniform distribution of the micronutrients by virtue of the greater bulk of the material applied to the soil.

Soluble micronutrient compounds can be used only at relatively low levels in formulating a general purpose fertilizer. The amounts in the fertilizer must be low enough to avoid toxicity at high application. The safe level is further limited by increased toxicity when the fertilizer is localized by drilling or banding in the growth of row crops (*5*). These factors make it necessary to use micronutrient quantities so low that they are of doubtful value and, indeed, under some conditions it is impossible to have micronutrient content high enough to correct deficiency.

The over-all problem is a matter of contending with difficulties involved in adjusting application of the micronutrients to crop requirement. While use in fertilizer mixtures tends to result in low application, soil conditions often narrow permissible range of application. In either case, judicious use of a truly slowly soluble carrier serves to compensate for these limitations. When application to the soil is low, crop requirement is met through more efficient utilization of the applied micronutrients. When application is high, low initial availability and dissipation during gradual release tend to prevent the development of toxic concentrations. Accordingly, permissible range of application is broadened. This property of a slowly soluble carrier might also be used to make a fertilizer formulation suitable for use with more than one specific crop (*20*).

Commercially Available Glasses

Two general types of slowly soluble glass carriers have been developed—boron glasses and multinutrient glasses containing from two to six elements essential in trace amounts for plant growth. Boron glasses were developed largely because maintenance of boron at suitable levels in soils is one of the most serious micronutrient problems. The multinutrient glasses of current manufacture are a possible practical answer to the occurrence of more than one micronutrient deficiency (*13*, *22*).

The nominal micronutrient contents of commercially available agricultural glasses are given in Table I. They also contain as much as 50% silica and lesser amounts of calcium, potassium, sodium, and aluminum in such ratio as needed for a required level of chemical reactivity. Multinutrient glasses are presently ground more finely than boron glasses; the amount of material passing 200 mesh is usually about 90 and 50%, respectively.

Nature of Dissolution. While glasses are often classed with mineral materials as slowly soluble carriers, they differ in the nature of their dissolution. According to Morey (*16*), regardless of the dissolved salts present, reaction between a glass and water at ordinary temperatures will proceed until decomposition of the glass is complete. The end result is a solution of the soluble products and quartz. Morey further concluded that the term "solubility" has no meaning in such a case, and all measurements which have been made represent "not solubilities, but rates of reaction." Possible confusion may be avoided by using the term "reactivity" to designate the tendency of a glass to release its water-soluble constituents. This term is also applied in the same sense to crystalline substances with low solubility (*7*). The pattern of release from such substances, however, is not likely to be the same in a soil as that of a silicate glass. Rate of release from a mineral material is controlled, fundamentally, by concentration of the dissolved phase in the immediate vicinity of the discrete particles.

Much of the silica formed when a glass reacts with water accumulates as a fragile coating on the unreacted portion of the particles. Electrolytes, such as calcium or barium chloride, depress rate of reaction, presumably through the flocculation of colloidal silica at the reactive surface. Reaction rates also vary directly with temperature and indirectly with pH. However, when glasses are agitated with mild aqueous solvents, the amounts of an element released are a satisfactory index to response in crops (*18*, *26*).

Table II. Relative Reactivity of Agricultural Glasses

Glass	Grinding Time, Hours	Material Passing 200-mesh, %	Boron Dissolved, % of Total: In ammonium acetate[a]	In moist soil[b]
176-E	2.0	46	60	93
176-F	2.0	50	53	81
176-E	0.5	17	40	73
176-F	0.5	17	28	65
502	..	90	86[c]	..
501	..	90	46[c]	..

[a] Boron released from glass during 16 hours of agitation with 0.44 molar ammonium acetate, pH 4.0.
[b] Boron released from glass during 9 months in soil, as computed from uptake data.
[c] This value, calculated from data supplied by the Ferro Corp., is for the 200- to 325-mesh material, the standard size used in the testing of multinutrient glasses (*26*). The glass also releases Fe, Mn, Cu, Zn, and Mo, in amounts which are percentage-wise about the same as the amount shown for boron release.

Vegetative Evaluation

Plant response studies have usually pertained to the effect of glass carriers on boron content of crops. The principal objectives have been to determine the optimal level of reactivity for a glass and to evaluate the benefit derived from use in coarse-textured soils. The test glasses and reference materials have been applied to soils either by broadcasting in field experiments or by mixing throughout the soil in greenhouse studies.

In practice, there is greater need for a slowly soluble carrier with more sensitive crops, such as cotton or soybeans. Where toxicity in early growth has been of prime concern, investigators have usually grown this type of crop. The long term influence of a carrier on soluble supply has been followed largely by determining the effect on boron content of consecutive harvests of alfalfa.

Moderate Reactivity. Useful glass carriers of boron increase boron content of the crop in the late season period more than an equal application of the nutrient in soluble form. Glasses of this kind are considered moderately reactive (*11*). The reactivity of glasses within this range, as measured by nutrient release in ammonium acetate and dissolution in soil, is given in Table II.

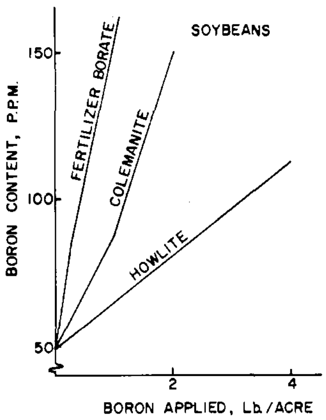

Figure 1. Influence of slowly soluble boron minerals on boron content of a crop as compared to that of fertilizer borate (from Table II in reference *28*)

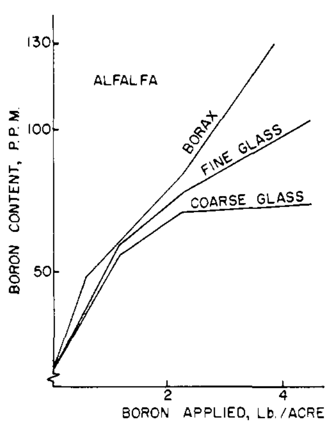

Figure 2. The influence of a moderately reactive boron glass on boron content of a crop as compared to that of borax (from Tables V and VII in reference 8)

Uptake in Early Growth. Experimental studies have demonstrated that the danger of boron toxicity can be reduced by use of slowly soluble forms, either mineral (*27, 28*) or glass (*9, 12*). The effect of mineral substances on nutrient content of a crop in early growth is compared to that of readily soluble fertilizer borate in Figure 1. The amount of boron that can be safely applied to a soil may be approximately doubled with colemanite, or increased perhaps sixfold with howlite, a naturally occurring borosilicate. Moderately reactive glasses differ in that they have nearly the same effect on boron content of a crop as soluble boron (borax) at low levels of application (Figure 2). However, the influence of the glasses on crop boron at high application, when protection against toxicity is needed, is considerably less than that of borax. These results indicate that boron application may be approximately doubled with the fine grind of glass 176-F or increased threefold with the coarse grind. The fine glass, having been more widely tested, will be used in other illustrations.

Minimization of Seasonal Variation. The long term effect of a glass on boron content of a crop is compared to that of an equal amount of soluble boron in Figure 3. Response to the glass, though initially lower, becomes greater than that to borax in late growth. Only about one half as much boron was needed to keep boron content of the crop from falling below the lowest level occurring with borax. Broadly, the effect of the glass is to minimize seasonal variation in crop boron (*25*), as indicated in Figure 3.

The magnitude of seasonal variation becomes progressively greater as application of soluble boron is increased, whereas little change of this kind occurs with moderately reactive glasses.

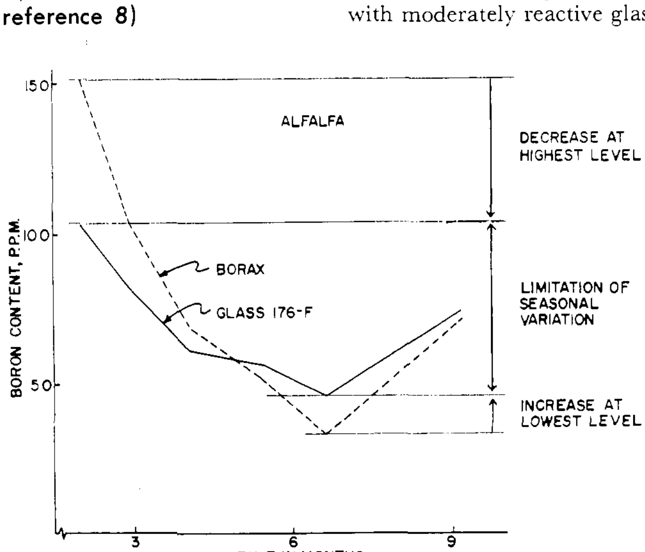

Figure 3. Minimization of seasonal variation in crop boron by slow release from a glass

Application level equivalent to 40 pounds of borax per acre (from Tables V and VII of reference 8)

Boron response to glass carriers is compared directly with that to colemanite under field conditions in Figure 4. The mineral material provides some protection against toxicity in early growth, but boron content of the crop drops more rapidly during the season. At the end of 6 months, the level in the crop was lower than that with Glasses 176-F and 502, even though the colemanite (10.1% boron) contained 1.7 and 3.6 times as much boron, respectively. From the standpoint of seasonal variation, the respective differences between high and low values were only one half and one fourth as great as with colemanite. The results definitely confirm Winsor's earlier conclusion (*29*) that glasses have a slower but more lasting effect on dissolved supply of the soil.

Results of plant response studies (*3, 30*) indicate that certain manganese, zinc, or iron glasses could be used satisfactorily as slowly soluble carriers of these micronutrients. The necessary study of their influence on uptake with respect to time has not yet been made. However, the advantage of slow release of such elements is suggested by the results obtained in parallel applications of manganous oxide and manganous sulfate, given in Table III. The principal effect of the less soluble oxide was to lower manganese content of the crop at the high levels reached in early growth of soybeans, thereby providing some reduction in seasonal variation.

Relationship of Nutrient Uptake to Release from Glass

Underlying the strong moderating influence of a glass carrier on uptake is the pattern of slow, but continuous, nutrient release. The steadiness of release can be discerned from relative response in a greenhouse study (*8*). In Figure 5, the increase in boron content of alfalfa (boron response) obtained with a glass (ΔR_g) relative to that with borax (ΔR_s) is plotted against time of harvest. With seasonal variation thus excluded, the general increase in the ratio during 9 months attests to the continuation of dissolution from the glass.

Boron uptake is favorably affected by suitable glasses when under stress of low levels of soluble supply. The prominent

Table III. Influence of Manganese Compounds on Manganese Content of Soybeans

Mean values of four replications

Compound	Amount Added to Soil, Lb./Acre	Manganese Content (in P. P. M.) of Crop When Sampled		
		June	July	August
None	. . .	435	283	329
$MnSO_4$	20	623	324	370
MnO	20	513	324	417

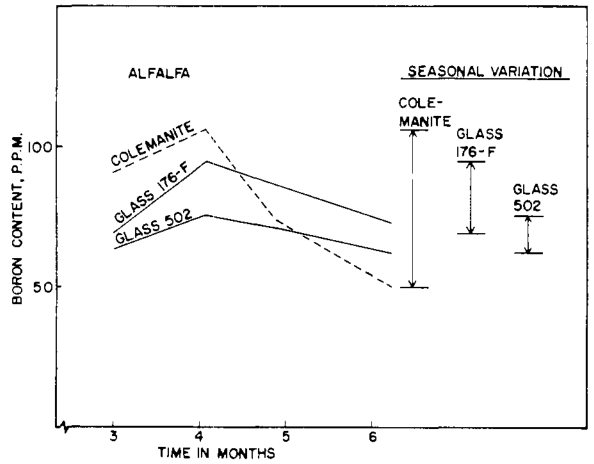

Figure 4. Seasonal variation in crop boron with 20 pounds of the carrier applied in the form of a boron glass (176-F), a multinutrient glass (502), and colemanite (72% > 80-mesh)

Original data supplied by N. R. Page

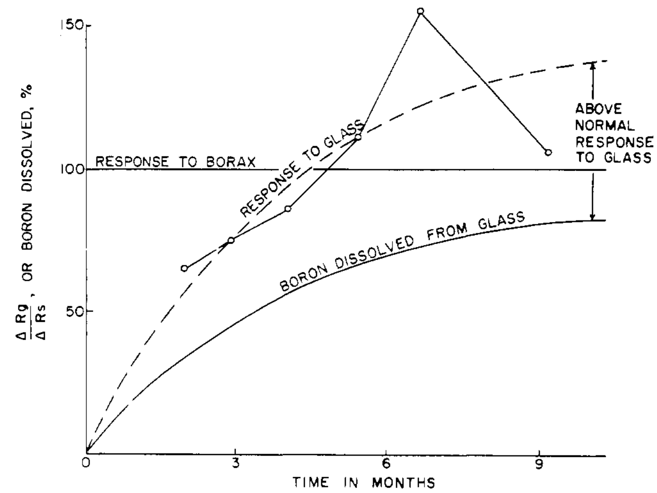

Figure 5. Boron response to glass carrier (176-F) relative to that of soluble boron (borax), and its relationship to the estimated amount of boron dissolved from the glass

peak at 6.6 months (fifth harvest) corresponds to the point at which boron content of the crop was the least (Figure 3). The large increase in relative response at this time was consistent with moderately reactive glasses. It did not occur with more reactive or less reactive glasses, both having very low release rates in the latter part of the season. Thus, this influence relates to the amount of newly released boron present. Neglecting variation of this kind, the general influence of the glass is represented by the broken line curve.

The amount of boron released from glass carriers during 9 months of crop growth was estimated by determining the circumstance under which two recognized relationships are obeyed. First, for a given general type of particle-size distribution (as in a group of either sieve fractions or grinds, but not interchangeably), boron response would be approximately proportional to the total amount of boron released. Secondly, nutrient release would be a function of the theoretical geometric decrease in particle volume, as demonstrated by Bear and Allen in limestone studies (*1*). Through the latter relationship, the amounts released may be related to particle diameter by the equation:

$$G_1 = G_0\left(1 - \frac{d_1^3}{d_0^3}\right)$$

where G_0 is the original quantity of glass and G_1 is the quantity of reacted glass, having respective diameters of d_0 and d_1. By applying this equation to sieve fractions and grinds of glass 176-E, the calculated amounts of boron dissolved at 9 months were proportional to relative response when the assumed reduction in particle diameter was 95 microns. The values for the grinds of 176-E, and those for glass 176-F, estimated by proportion from relative response, are given in the last column in Table II. The dissolution curve in Figure 5 is drawn by projection from the response curve, maintaining the ratio between relative response and dissolution established for the particular glass at 9 months, thus tracing the pattern of dissolution.

The dissolution curve denotes "expected" response, were response not dependent partly on the conservation of supply through slow release from a glass. Only the dissolved portion is subject to loss from the root zone through leaching or sorption processes. The gain in nutrient uptake, as the result of lower loss within the soil, is designated in the figure as the "above normal response to glass." The magnitude of this quantity depends on the balance maintained between rate of loss and rate of release from the glass. In this case, the actual response curve was 70% greater than indicated by the dissolution curve. Under some field conditions, it may be several times as large.

To provide significant amounts of newly released nutrients in late growth, some of the glass must necessarily remain unaltered in the soil. Optimal results were obtained in the late season period, when the estimated amount of undissolved glass boron remaining in the soil was about 5 to 20% of that added. The influence of more reactive glasses is much the same as that of a readily soluble carrier. Hence, optimal performance lies near the upper limit of the range of moderate reactivity. When release rate is reduced moderately, the ability of the glass to minimize seasonal variation is increased, which is more important than high efficiency under some circumstances. At the lower limit of moderate reactivity, a glass releases about 50% of its boron.

Adjustment of Reactivity to Type of Crop

The appropriate level of reactivity for a glass carrier depends to a large extent on the length of time required by the crop to reach maturity. Glasses releasing about 85% of their nutrients in ammonium acetate, pH 4.0, are often suitable for use with crops maturing within a period of 4 months. Where fertilization is to cover a period of 6 to 9 months, ammonium acetate-extractable nutrients should be lowered to at least 50 or 60%. For reducing toxicity damage, if need be, or for use in a multiseasonal pattern (*10*), ammonium acetate-extractable nutrients may be lowered to about 30% without undue loss in ability to correct deficiency.

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CHELATES FOR MICRONUTRIENTS

Properties of Chelates and Their Use in Crop Production

J. C. BROWN and L. O. TIFFIN
U.S. Department of Agriculture, Beltsville, Md.

Precipitated iron may serve as a reserve iron supply for plants but a mechanism is required for making it available to plants. Synthetic chelating agents have been used effectively to extract iron from soils or as iron chelate to keep iron in a soluble form in growth media. The apparent stability of iron chelates differs. The capacity of plants to absorb iron from iron chelate depends upon the kind and concentration of chelating agent, concentration of iron, plant species, and for some plants, whether the plant is green or chlorotic. Chlorotic Hawkeye soybeans differentially absorbed iron and chelating agent. Iron supplied to chlorotic Hawkeye soybeans at $2 \times 10^{-6}M$ FeEDDHA appeared in the stem exudate as Fe malate. Roots and chelating agents compete for the iron in a nutrient solution. Roots which compete most effectively appear to have a reductive process, which affects the stability or availability of iron at the root. The factors which affect the availability of iron may or may not be a part of the actual absorption mechanism.

Among the important functions of metal ions in biological systems is their action as cofactors in enzyme systems. The microelements are particularly important and an adequate available supply is necessary for plant growth and development. Most microelements will hydrolyze and precipitate at pH 6, if they are not carried as chelate compounds. This is particularly true of iron. Agriculturally, there has long been a need for a soluble or available source of iron for plant growth.

Synthetic iron chelates have been used effectively to keep iron soluble and available for plant growth (*8, 13, 18, 21, 22, 30*). Four synthetic chelates are discussed — ethylenediaminetetraacetic acid (EDTA), diethylenetriaminepentaacetic acid (DTPA), cyclohexanediaminetetraacetic acid (CDTA), and ethylenediaminedi(*o*-hydroxyphenylacetic acid) (EDDHA).

Properties of Chelates

The apparent stability constants for FeEDTA, FeCDTA, FeDTPA, and FeEDDHA are 24.8, 29.3 (*1*), 27.9, and 30 (*11*), respectively. FeEDDHA is the most stable of the iron chelates. EDDHA, accordingly, would be expected to be the most competitive for iron in a growth medium. The capacity of EDDHA to chelate iron was determined in a nutrient solution containing variable concentrations of EDTA, DTPA, and CDTA as competitive chelating agents (*6*). The competitive chelating agents were equilibrated at pH 6.5 for 1 hour with a complete nutrient solution containing either $2 \times 10^{-5}M$ or $4 \times 10^{-5}M$ of Fe added as $FeCl_3$. EDTA, DTPA, and CDTA were supplied at 0.16, 0.5, 1, 2, 4, 6, 12, 18, or $36 \times 10^{-5}M$ concentrations. After equilibration, $2 \times 10^{-5}M$ EDDHA was added to each of the nutrient solutions, and the FeEDDHA (Figure 1) concentration was determined colorimetrically at varied intervals with final measurements made after 30 days (*6*).

The chelating capacity of EDDHA decreased sharply when the concentration of each competing chelating agent reached $2 \times 10^{-5}M$: EDTA < DTPA < CDTA. By increasing the Fe concentration to $4 \times 10^{-5}M$, $2 \times 10^{-5}M$ EDDHA competed for Fe successfully with $2 \times 10^{-5}M$ EDTA, DTPA, and CDTA. In this case, there was sufficient iron for both EDDHA and the competing agent. An increase in concentration of EDTA, DTPA, or CDTA to $4 \times 10^{-5}M$ sharply decreased the amount of Fe chelated as FeEDDHA. The effectiveness of the competitors was related to the stability of their iron chelates: FeEDTA < FeDTPA < FeCDTA. Thus, chelating capacity of EDDHA is dependent upon both the concentration of the Fe and the competitive chelating agent in solution. Absorption of iron by roots may likewise be dependent upon both the concentration of the Fe and the competing ligands in solution.

Use of Chelates

The above chelating agents can be used to extract iron from soils. The