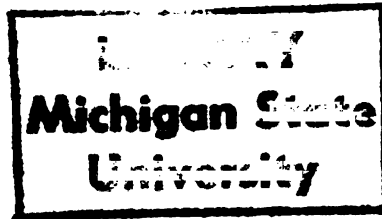




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Agronomic Considerations for the Application of a  
Molybdenum-rich Sewage Sludge to an Agricultural Soil

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AGRONOMIC CONSIDERATIONS FOR THE APPLICATION OF A  
MOLYBDENUM-RICH SEWAGE SLUDGE TO AN AGRICULTURAL SOIL

By

Gary Michael Pierzynski

A THESIS

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Michigan State University  
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## ABSTRACT

### AGRONOMIC CONSIDERATIONS FOR THE APPLICATION OF A MOLYBDENUM-RICH SEWAGE SLUDGE TO AN AGRICULTURAL SOIL

By

Gary Michael Pierzynski

Laboratory, greenhouse, and field experiments were conducted to evaluate methodology and to monitor crop and soil responses from the application of a Mo-rich sewage sludge to a Metea loamy sand soil.

Molybdenum concentrations were determined in plant tissue digests and soil extracts by direct-current plasma emission spectrometry (DCP) and compared to Mo analysis with nitrous oxide/acetylene flame atomic absorption spectrometry (AAS). Soil samples were extracted with 1.0 M  $\text{NH}_4\text{HCO}_3$  + 0.005 M DTPA (AB-DTPA), 1.2 M  $\text{NaC}_2\text{H}_3\text{O}_2$ , Tamm's acid ammonium oxalate, and 0.5 M  $(\text{NH}_4)_2\text{HPO}_4$ . Soil pH levels were also monitored.

Regression analysis of the DCP results on the AAS results indicated a highly significant relationship and ultimately that the DCP method was suitable for Mo determinations. Sludge applications resulted in significant increases in the Mo concentrations in corn, soybeans, and alfalfa grown in the field and greenhouse experiments. Increases in soil pH also contributed to significant increases in the uptake of Mo. The AB-DTPA extractant was recommended for further testing.

To Joy, Twin A, Twin B, and Missy

## ACKNOWLEDGMENTS

I would like to express my sincere gratitude to Dr. L. W. Jacobs for his active participation and guidance in my entire graduate program. The time that he has given me, the opportunities that he has presented me, and the patience that he has shown me have not gone unnoticed.

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## CHAPTER I

### LITERATURE REVIEW

#### Introduction

Considerable research has been done on Mo and its role in soils, plants and animals. In 1956, Soil Science devoted an entire volume to Mo, listing 392 literature citations pertaining to Mo research. One could easily hypothesize that a doubling or tripling of the amount of literature has occurred in the last 29 years.

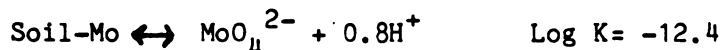
The intent of this literature review then is to 1) indicate the major factors involved in Mo chemistry in the soil, 2) indicate the major factors pertaining to the role of Mo in plants and animals, and 3) present a thorough review of the literature regarding availability indices for Mo and contamination of soils with Mo wastes. Citations are given that either were important in the elucidation of the role of Mo in the environment or that give results or conclusions relevant to this study. Several works can provide a more comprehensive examination of the literature regarding Mo in soils, plants or animals (Jarrell et al., 1980; Beeson and Matrone, 1976; Mortvedt et al., 1972; Underwood, 1977).

#### Soil Molybdenum

Goldschmidt (1954) estimated that the average Mo concentration of the lithosphere is  $2.3 \text{ mg kg}^{-1}$ . Levels of  $0.2$  to  $5.0 \text{ mg kg}^{-1}$  are indicated by Lindsay (1979) as a common range occurring in mineral soils

with levels reported up to 75 mg kg<sup>-1</sup> by Goldschmidt (1954). Kubota (1977) suggested a median Mo level of 1.2 to 1.3 mg kg<sup>-1</sup> in soils with a range of 0.1 to 40 mg kg<sup>-1</sup>. Elevated levels of Mo in the soil have been associated with black shale parent material (Thomson et al., 1972), granitic alluvial fans (Kubota et al., 1961) and the Lower Lias geologic formation in England (Lewis, 1943).

The predominant form of Mo in the soil solution is the molybdate anion (MoO<sub>4</sub><sup>2-</sup>) at pH greater than 4.24. The HMoO<sub>4</sub><sup>-</sup> anion and the unionized acid H<sub>2</sub>MoO<sub>4</sub> are the only other forms of Mo considered important in the soil solution. Various minerals have been examined as being responsible for controlling the solubility of Mo in soils. PbMoO<sub>4</sub> (wulfenite) is the most stable mineral that could form in soils. Vlek and Lindsay (1977b) found that equilibrating soils with wulfenite increased molybdate levels and concluded that the soils were undersaturated with respect to this mineral. Follett and Barber (1967) found that the solid phases of calcium molybdate and ferrous molybdate were too soluble to exist in a Maumee sandy loam soil. Lindsay (1979) proposed the use of a Soil-Mo compound expressed by the reaction



based on previous data from Colorado soils without Mo amendments.

In the absence of any identifiable solid phase Mo mineral controlling Mo solubility, adsorption processes have been studied in great detail. A basic approach was taken by Smith and Leeper (1969) who explained the need to add Mo fertilizer to acid Australian soils every few years by showing that Mo removable by leaching was less than 4% of that added to acid soils after a 2.5 to 10 month incubation period. Significant proportions of the added Mo became firmly adsorbed by the

soil after only a few weeks of incubation. Reisenauer et al. (1962) studied Mo adsorption by the hydrous oxides of iron, aluminum and titanium as well as by four California soils. Adsorption capabilities of the three materials followed the order--  $\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O} > \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O} > \text{TiO}_2 \cdot x\text{H}_2\text{O}$ . Adsorption of Mo decreased with increasing pH for all hydrous oxides and soils and was also dependant on the concentration of Mo in the equilibrium solution. The adsorption characteristics of the hydrous oxides and soils followed the Freundlich adsorption isotherm but not the Langmuir isotherm. Jarrell and Dawson (1978) reported that the amorphous fraction of Fe oxides extracted with ammonium oxalate (pH 3.3) were significantly correlated with the amount of Mo adsorbed by eight western Oregon soils at an equilibrium concentration of  $0.03 \text{ mg L}^{-1}$ .

Sulfates and phosphates also affect the amount of Mo a soil can adsorb. Barrow (1970) found that the Mo adsorbed increased with increasing levels of sulfate or phosphate at constant pH levels ranging from approximately 4.0 to 6.4. The ratio of Mo adsorbed to phosphorous adsorbed increased twenty fold as pH decreased from 6.0 to 4.0. These findings were supported by those of Jones and Ruckmann (1973) who reported a decrease in Mo uptake by subterranean clover with sulfur fertilization. Stout et al. (1951), using tomato plants in solution culture, also showed decreased Mo uptake with increasing sulfate levels, but they found large increases in Mo uptake by subterranean clover with increased phosphorous levels in the soil.

The role of pH in soil Mo chemistry has been studied extensively, primarily because of the drastic affect pH has on plant available Mo. In areas where Mo deficiencies in plants can occur, the increase in plant available Mo with increased pH can be an added benefit of liming acid

soils. If the Mo deficiencies are severe enough, a Mo application can induce the same crop response as a more costly lime application. Conversely, in areas where soils can produce toxic levels of Mo for ruminants in forages, lime additions can aggravate an existing problem.

Follett and Barber (1967) found that additions of CaO to a Maumee fine sandy loam soil increased the Mo activity in the water extract. Jones (1957) found that metahalloysitic, nontronitic, and kaolinitic clays showed maximum molybdate adsorption at a pH of approximately 4.0 with significant amounts of molybdate desorbed by the clays as pH increased. Theng (1971) obtained similar results with clay fractions of three New Zealand soils and hypothesized that desorption of Mo, as pH increased from 4.0, was a major factor in the increase of plant available Mo with increasing soil pH.

Numerous studies have shown increased Mo uptake by crops after liming. Gupta (1969) found that lime applications substantially increased the Mo content of cauliflower and alfalfa in three acid soils responsive to both Mo and lime applications. Molybdenum levels in alfalfa increased from trace amounts to  $1.48 \text{ mg kg}^{-1}$  when pH was increased from 5.0 to 6.5 on an Acadia silty clay loam soil. An increase from  $0.5$  to  $3.6 \text{ mg kg}^{-1}$  Mo in alfalfa was noted by James et al. (1968) after an application of 3 tons of lime/acre (initial pH 5.0). Gurley and Giddens (1969) increased the Mo content of soybean seed from  $1.1$  to  $2.2 \text{ mg kg}^{-1}$  on a Cecil sandy loam after a pH increase from 5.6 to 6.9, and from  $1.8$  to  $3.2 \text{ mg kg}^{-1}$  on a Norfolk loamy sand after a pH increase from 5.4 to 7.1.

Other factors that influence plant available Mo include organic matter content and soil moisture content. Kubota et al. (1961) reported that high water table and surface organic matter accumulation of poorly drained soils in Nevada increased Mo levels in plants. The magnitude of the effect was determined by the parent material. Granitic alluvial fans produced the highest Mo concentrations in forages. No such effects were found on well-drained soils with similar parent materials and organic matter contents.

The effect of soil moisture content on the uptake of Mo by clover was further investigated by Kubota et al. (1963) who reported that wet soil conditions increased the Mo in the soil solution and in clover grown on four soils. Under greenhouse conditions, Jensen and Lesperance (1971) reported significant increases in the Mo concentration of alfalfa, birdsfoot trefoil and sudangrass with decreasing depth to the water table.

Gupta (1971), investigating the influence of organic materials (compost, farmyard manure and sphagnum peat) on exchangeable Mo in a Mo deficient soil, found that when the materials were individually added to the soil the levels of exchangeable Mo increased. When combined with Mo additions, the organic materials decreased levels of exchangeable Mo. Kretschmer and Allen (1956) found levels of Mo in forages grown on organic soils in Florida that would be toxic to ruminants.

The nature of the soil moisture content influence on plant available Mo was investigated by Lavy and Barber (1964). When soil saturation extracts contained greater than 4 ppb of Mo, mass-flow caused Mo to accumulate at or in the roots of corn and soybeans. When saturation extracts had less than 4 ppb of Mo, diffusion patterns were

usually observed. Vlek and Lindsay (1977a) hypothesized that mass-flow may partially account for the greater accumulation of Mo by alfalfa grown under greenhouse conditions as compared to field conditions due to increased transpiration in the greenhouse.

#### Molybdenum Availability Indices

Considerable efforts have been undertaken to develop soil extractants useful for detecting Mo deficient soils and for detecting soils that can produce toxic levels of Mo in ruminant forages. Limited success has been attained by some researchers, but a standardized available Mo soil test has not been developed.

Several factors have impeded the development of such a test. Plants require minute amounts of Mo for normal growth and can obtain all of their Mo requirements from subsurface horizons. Thus, extractable Mo from surface horizons may have no relationship to plant Mo content. Also, soils usually contain small amounts of Mo and even less extractable Mo; thus, the amount of Mo extracted on deficient or low Mo soils is frequently below the detection limits of the assay procedure. Finally, other factors that influence plant available Mo are not readily reflected by soil extractants.

Grigg (1953) performed one of the first comprehensive evaluations of a series of extractants for available Mo. Tamm's acid ammonium oxalate solution (0.18 M ammonium oxalate + 0.1 M oxalic acid, pH 3.3) was reported to be more suitable than 0.1 N NaOH, 0.5 N NaOH or 1 N  $\text{NH}_4\text{OAc}$  (pH 7.0) in determining Mo responsive soils in New Zealand. All soils responsive to Mo fertilization had 0.14 ppm of oxalate extractable Mo or less and a pH of 6.3 or less.

Tamm's reagent has since been tested under a variety of conditions and soils. Later work by Grigg (1960) indicated that Tamm's reagent was not as good as previously thought. Soils were then characterized into two broad groups— 1) responsive to Mo with acid-ammonium oxalate levels of 0.2 ppm or less or 2) not responsive to Mo with acid ammonium oxalate levels of 0.15 ppm or more. Some consideration of soil type was required to differentiate between the two groups. Other workers have successfully used Tamm's reagent. Haley and Melsted (1957) obtained a correlation coefficient of 0.63 between Mo composition of oats and acid-ammonium oxalate extractable Mo. Lowe and Massey (1965) reported a multiple correlation coefficient of 0.7 between Mo uptake by alfalfa and Tamm extractable Mo and soil pH from cultivated Kentucky soils.

Gupta and MacKay (1966) proposed a slight modification of Tamm's reagent by using 0.3 M ammonium oxalate (pH 3.0) and referring to the Mo extracted as exchangeable Mo. Early work by Gupta and Mackay (1966) indicated that this exchangeable Mo was significantly correlated with soil pH but not with organic matter content and increased as the soil texture became finer. Later work by Gupta (1970) failed to find a significant pH effect on exchangeable Mo.

A variety of other extractants have been partially successful. Lowe and Massey (1965) worked on a hot water extraction technique for available Mo and obtained a multiple correlation coefficient of 0.85 between Mo uptake by alfalfa and hot water extractable Mo and soil pH on cultivated Kentucky soils. Haley and Melsted (1957) reported a significant correlation ( $r^2 = 0.73$ ) between plant Mo composition and Mo extracted with 0.1 N NaOH. Two other extractants used, 0.03 N  $\text{NH}_4\text{F}$  and 0.1 N HCl + 0.03 N  $\text{NH}_4\text{F}$ , were not suitable for Illinois soils. Williams



and Thorton (1973) found that EDTA extractable Mo was better related to Mo uptake by perennial ryegrass than  $1 \text{ N NH}_4\text{OAc}$  or total soil Mo. Water and  $1 \text{ M (NH}_4)_2\text{CO}_3$  were found to be equally as effective in predicting Mo concentrations in alfalfa grown on alkaline soils in Colorado (Vlek and Lindsay, 1977a). Correlation coefficients as high as 0.98 were obtained for both extractants under greenhouse conditions.

A promising technique for determining available soil Mo using an anion exchange resin was proposed by Bhella and Dawson (1972). Molybdenum removed by the resin was significantly correlated with soil pH ( $r=0.803$ ) and uptake by subterranean clover ( $r=0.861$ ) from 30 western Oregon hill soils. Jarrell and Dawson (1978) divided 8 western Oregon soils into two groups, suspension pH less than 4.9 or suspension pH greater than 4.9, and reported correlations of  $R^2 = 0.64$  and  $R^2 = 0.72$ , respectively, between uptake of Mo by subterranean clover and anion exchangeable Mo. Karimian and Cox (1978) also found anion exchangeable Mo to be significantly correlated with soil pH.

#### Plant-Molybdenum Relationships

Molybdenum was first shown to be an essential plant nutrient by Arnon and Stout (1939). Studies by Boswell and Anderson (1969), deMooy (1970), Gurley and Giddens (1969), Mortvedt (1981) and Reisenauer (1956) have all shown yield increases on a variety of crops from Mo fertilization.

Many plant species can apparently accumulate high concentrations of Mo without adverse affects. There are no reports of Mo toxicity under field conditions but toxic levels of Mo in plant tissue have been

attained after extremely excessive doses under solution culture conditions. Wallace et al. (1977) found that  $10^{-3}$  M  $\text{H}_2\text{MoO}_4$  in nutrient solutions produced stunting and slight yellowing of bush bean plants. Leaves, stems, and roots contained 710, 1054, and 5920 mg Mo  $\text{kg}^{-1}$  dry weight, respectively. Nutrient solutions containing as much as  $2 \times 10^{-4}$  M  $\text{H}_2\text{MoO}_4$  showed no adverse affects. Hewitt (1953) indicated that Mo toxicity in sugar beets resembled Fe deficiency. Hunter and Vergnano (1953) induced Mo toxicity in oats when Mo concentrations in nutrient solutions were at 100 ppm or greater. Chlorosis, red-yellow leaf tips and reduced root size were the reported symptoms. Johnson (1966) reported Mo toxicity symptoms in tomatoes whose leaves contained 1000-2000 ppm Mo.

Studies reported by Kubota et al. (1961), Jensen and Lesperance (1971), Barshad (1948), and Kretschmer and Allen (1956) indicate that legumes tend to accumulate more Mo in tissue than do nonlegumes, further aggravating Mo toxicity problems in cattle that use legumes as a primary feed source. This is presumably due to the Mo incorporated into the enzymes involved in  $\text{N}_2$  fixation by legumes. Kubota et al. (1961) further differentiated some legumes by noting that clovers accumulate more Mo than alfalfa when grown under similar conditions.

#### Animal-Molybdenum Relationships

Molybdenum has been shown to be an essential mineral in animal nutrition even though minimum dietary requirements for Mo have not been established and Mo deficiency has not been observed with any species. Underwood (1977) presents a review of the literature pertaining to the metabolic functions of Mo in animals.



Of particular interest in Mo contamination studies is a Mo toxicity condition in ruminants, especially cattle. Molybdenosis, also called "teartness", "peat scours", or "alkalied", is a Mo induced Cu deficiency identified as causing severe diarrhea, loss of appetite, discoloration of hair, joint abnormalities, osteoporosis, lack of sexual interest, and, in severe cases, even death (Dye and O'Hara, 1959). Ferguson et al. (1938) first noted that severe diarrhea developed in cattle grazing on the "teart" pastures of England and attributed this to excessive Mo in the forages.

Critical levels of Mo in animal feeds have been difficult to establish due to apparent interactions of Cu and  $\text{SO}_4^{2-}$  in the feed sources. Dye and O'Hara (1959) reported symptoms of molybdenosis in cattle consuming forages containing as little as 5 ppm Mo. A commonly used value is 10 ppm.

The dangers of excessive levels of Mo in cattle feeds can be offset by adequate Cu levels. Miltmore and Mason (1971) suggested that Cu:Mo ratios less than 2 could induce molybdenosis. Alloway (1973) found that a Cu:Mo ratio of less than 4 was indicative of potentially toxic forages.

Sulfates in the diets of ruminants have been shown to reduce Mo uptake and, consequently, help to alleviate Cu deficiencies. Underwood (1977) and Beeson and Matrone (1976) review literature dealing with the Mo, Cu and sulfate interactions in animal feeds.



### Contamination of Soils With Molybdenum

Significant amounts of Mo can be added to the soil by irrigation waters, dusts, fly ashes, and sewage sludges. Molybdenum additions to the soil may result in increased Mo uptake by plants and consequently, molybdenosis in ruminants consuming forages grown on the affected areas.

The U.S. Environmental Protection Agency (1973) has established an upper limit of  $0.01 \text{ mg L}^{-1}$  for Mo in irrigation waters. In areas near Mo mining operations (primary areas in the U.S. are the Climax and Urad mines in Colorado and a few smaller mines in New Mexico) Mo concentrations in surface waters can exceed this level considerably. Jorden and Meglen (1973) surveyed several streams in Colorado and New Mexico and found Mo concentrations as high as  $3800 \text{ ug L}^{-1}$  (Colorado river and tributaries). Jackson et al. (1975) reported Mo concentrations in alfalfa increasing from 4.3 to 7.2 ppm after one growing season using irrigation water with 213 ppb Mo. A computer model developed in this study suggested that irrigation waters containing 400 ppb Mo would produce toxic levels of Mo in alfalfa after 3 years.

Vlek and Lindsay (1977a), assuming a yearly irrigation of 25 cm and disregarding deep percolation, calculated that  $2.5 \text{ g Mo ha}^{-1}$  is added for each ppb of Mo in irrigation water. Assuming an alfalfa yield of 10 metric tons/ha,  $10 \text{ g Mo ha}^{-1}$  would then be removed for each ppm of Mo in the crop. The conclusion was that, with 5 ppm Mo being the upper limit tolerable in forages, the concentration of Mo in irrigation water should not exceed 20 ppb (twice the EPA upper limit) if the outputs were to equal the inputs.

Coal and its combustion products can contain significant amounts of

Mo. Swanson et al. (1976) found Mo concentrations in coal to be as high as 50 ppm with a mean value of 3.0 ppm.

Molybdenum in coal can be applied to the soil either as particulate matter from smokestack emissions or via land application of fly ashes. Schwitzgebel et al. (1975) found Mo discharge into the atmosphere from coal-fired power plants to have annual rates as high as 15 metric tons/1000 megawatts of generated power. Furr et al. (1977) reported a mean Mo concentration of 25.4 ppm in fly ashes from bituminous coals in the eastern U.S.

Molybdenum applied to the soil with fly ashes is available for plant uptake. Doran and Martens (1972) found that fly ash additions to the soil equivalent to 0.53 or 1.05 mg of added Mo per 2100 g dry soil significantly increased Mo uptake by alfalfa. The availability of the fly ash Mo was nearly equal to equivalent amounts of Mo from its sodium salt. A fly ash containing 39.3 ppm Mo produced alfalfa with Mo concentrations too high to safely feed to cattle. Gutenmann et al. (1979) reported significant increases in the Mo concentration of alfalfa, birdsfoot trefoil, bromegrass, orchard grass, and timothy grass after an application of 112.5 metric tons/ha of fly ash containing 2.9 ppm Mo. The ash continued to supply Mo to the crops after 5 cuttings over a two year period.

#### Molybdenum Contamination from Sewage Sludges

The primary objective of this study was to evaluate the availability of Mo applied with sewage sludges to plants. Little research has been done to address this issue with only a few studies

that deal with Mo uptake from sludges directly and a few more that deal with it in a secondary manner.

The range of concentrations for Mo in sewage sludge can be quite wide. In a survey of Michigan sludges Jacobs et al. (1981) reported a range of 6-3700 ppm Mo with a median of 32 ppm. Dowdy et al. (1976) reported a range of 5-39 ppm Mo from a survey of 200 sludges from 8 states. Molybdenum concentrations ranged from <1 to 73 mg kg<sup>-1</sup> in 44 sewage sludges from Iowa communities (Tabatabai and Frankenberger, 1979).

In a greenhouse study conducted by Williams and Gogna (1981), a high Mo liquid sludge (103 ppm Mo) was applied to supply 0.21 and 0.41 kg ha<sup>-1</sup> of Mo, a low Mo liquid sludge was applied to supply 0.02 kg ha<sup>-1</sup> of Mo, and sodium molybdate was applied to supply 0.41 kg ha<sup>-1</sup> Mo. The soil was adjusted to two pH levels, 6.1 and 7.2, and ryegrass and white clover were grown. In the first year the high rate of the high Mo sludge produced white clover with Mo concentrations as high as 94 ppm and ryegrass with Mo concentrations as high as 20.4 ppm. The low Mo sludge did not significantly increase Mo levels in either crop for any cutting. The high pH soil had significantly greater concentrations of Mo for both crops for all cuttings as compared to the low pH soil. Overall, the Mo from the high rate of high Mo sludge was as available for plant uptake as the equivalent rate of the Mo salt. In a follow-up report, Williams (1982) reported that for the second year of cropping on the same pots the pH differences had disappeared and that the Mo from the high rate of high Mo sludge was being taken up in greater amounts than the comparable rate of the Mo salt. Ammonium acetate extractable Mo predicted availability of the Mo better than Mo extracted with Tamm's reagent.



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Davis (1981) conducted greenhouse studies to evaluate Cu and Mo uptake from a soil (total analysis showed 6 ppm Mo and 515 ppm Cu) that had had a variety of sludges applied to it and to evaluate Mo uptake from a sludge containing 359 ppm Mo. For the contaminated soil the Cu/Mo ratio was determined for 13 plant species. The plants were separated into 3 groups. The first group contained the grasses (including cereals) and members of the beet family (sugar beet and mangold) with Cu/Mo ratios from 2.38 to 5.87. The second group included the brassicas (turnip and kale) with Cu/Mo ratios from 0.46 to 1.28. The last group contained only legumes with Cu/Mo ratios less than 0.46. The conclusions were that geno-type had a large influence on Mo uptake and that Cu applied to the soil along with Mo did not produce favorable Cu/Mo ratios in legumes.

The second part of the study by Davis (1981) involved applications of a high Mo sludge at varying rates up to an equivalent of  $17 \text{ kg ha}^{-1}$  of applied Mo. Three crops were grown (ryegrass, clover and kale) at two pH levels (6.2 and 8.0). Ryegrass did not accumulate significant amounts of Mo at either pH. Kale had significantly greater Mo concentrations on the sludge treatments as compared to the control at the high pH only. Clover accumulated the greatest Mo concentrations (up to 31 ppm) and showed significant increases with increased sludge loadings at both pH levels. The greatest affect was seen at the high pH level.

Bradford et al. (1975) studied three sewage sludges from California and three commercial sludge products marketed in California. The six materials had a range of 2.0 to 25.0 ppm total Mo and a range of 0.1 to 0.37 ppm Mo in saturation extracts from these materials. The sludge

with the highest total Mo content had the lowest level of Mo in the saturation extract. Bean, tomato and barley plants were grown in sand culture and irrigated with the saturation extracts from the materials. The highest concentration of Mo occurred in tomato and barley plants receiving the saturation extract from the sludge with the highest level of water soluble Mo.

Applications of  $0-4.8 \text{ kg ha}^{-1}$  Mo from a liquid sewage sludge by King and Morris (1972) failed to produce a significant increase in the Mo concentration in rye clippings grown on a Cecil sandy clay loam under acid conditions (pH range of 4.5-5.2). These results were in agreement with those of Davis (1981) in that rye is not an accumulator of Mo.

A further complication in managing land application of sewage sludges is the soil pH. The application of sewage sludge to soil may raise or lower the pH. Normal recommendations for soil pH when sludge is applied are to keep the pH near neutral to reduce the availability of the cationic heavy metals. Numerous studies have shown that increasing pH increases Mo availability, regardless of the source of Mo.

Lahann (1976) suggested that Mo-rich sludges could be used for reclamation of acid mine spoils from coal strip-mining rather than being applied to agricultural lands. The sludge studied raised the pH of the spoil material which resulted in increased amounts of Mo in the drainage waters. When the pH of the runoff water was neutral it contained 270 ppb of Mo as compared to acid conditions (pH 5.6) when the water contained only 1.6 ppb. If the situation was closely monitored, this was shown to be a plausible alternative for disposal of Mo-rich sludges.

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## CHAPTER II

### USE OF DIRECT-CURRENT PLASMA SPECTROMETRY FOR THE DETERMINATION OF MOLYBDENUM IN PLANT TISSUE DIGESTS AND SOIL EXTRACTS

#### Abstract

Molybdenum concentrations were determined in plant tissue digests and soil extracts by direct-current plasma optical emission spectrometry (DCP) and compared to molybdenum analysis with nitrous oxide/acetylene flame atomic absorption spectrometry (AAS). Regression analysis of the DCP results on the AAS results indicated a highly significant relationship ( $r^2=0.97$ ) with a slope of 1.01 and a y-axis intercept of -0.40. The slope of the regression equation was not significantly different than 1.0 and the y-axis intercept was not significantly different than zero. DCP results were enhanced at low Mo concentrations as compared to AAS results when high levels of the macro-nutrients were present in the sample matrix.

### Introduction

Plasma emission spectrophotometry is becoming an increasingly popular method for determining a variety of elements in plants and soils. Two types of argon plasmas are currently in use, direct current (DCP) and inductively coupled (ICP), and both are capable of simultaneous determination of most macro- and micro-nutrients.

Commercial DCP's available today utilize a three electrode system consisting of two anodes and a common cathode in an inverted Y-configuration. A direct current discharge struck between the electrodes produces the plasma which is maintained by a flow of argon around each electrode. The sample is aspirated with argon gas into the plasma between the two anodes. The use of DCP's is less than that of ICP's and consequently, little information is in the literature about DCP methods. In addition, the use of plasma emission for Mo analysis has been limited, primarily because of enhancement of Mo readings that becomes evident at the low levels of Mo normally found in plants and soils.

Woodis et al. (1980) investigated the use of the DCP for determination of Mo in fertilizers. Seven emission wavelengths were investigated with the 313.26 nm emission line producing the least enhancement of a  $2 \text{ mg L}^{-1}$  Mo sample solution. Potassium was found to cause the enhancement and the effect was removed by the addition of  $4000 \text{ mg L}^{-1}$  K to all samples and standards. DCP results were found to be in excellent agreement with the dithiol colorimetric procedure for Mo determination.

Mortvedt (1981) compared the use of argon plasma emission

spectrophotometry (type of plasma not indicated) and the dithiol colorimetric procedure for the determination of Mo in plant tissues. Molybdenum concentrations were generally higher with the plasma method. Attempts to correct for the enhancement by inclusion of various combinations and concentrations of Ca, Mg, and K in the Mo standards were not successful, so this method was deemed unsuitable.

Measurement of Mo emission at the 379.8 nm line by DCP instrumentation has indicated spectral enhancement by Ca and Mg (Johnson et al., 1979b) and the alkali salts present in salt and brackish water (Eastwood et al., 1980). Spectral enhancement of Mo at this wavelength by molecular emission associated with carbon species has also been identified (Johnson et al., 1979a).

The over-lying problem in establishing a method for Mo determination is the lack of acceptable standards with certified Mo concentrations. It becomes necessary to compare new methods with older, more accepted methods that may be susceptible to analytical difficulties themselves. This paper compares Mo determinations made by DCP emission spectrometry and nitrous oxide/acetylene flame AAS in a variety of plant tissue digests and in two soil extracts.

### Materials and Methods

Soil and plant tissue (corn (*Zea mays* L.) and soybean (*Glycine max* L.)) samples used in this study were obtained from a field experiment designed to study the uptake of Mo from a Mo-rich sewage sludge. Various application rates were used in the field experiment and, therefore, samples were chosen to reflect a wide range of Mo concentrations. Plant samplings utilized were: diagnostic soybean leaves (uppermost fully developed trifoliate) taken at the onset of flowering, diagnostic corn leaves (ear leaf) taken at tasseling and corn grain. In addition, National Bureau of Standards (NBS) wheat flour sample 1567, NBS citrus leaves sample 1572, and an alfalfa (*Medicago sativa* L.) sample containing low levels of Mo were used. The NBS wheat flour sample has a non-certified Mo concentration of  $0.4 \text{ mg kg}^{-1}$  and the NBS citrus leaves sample has a certified Mo concentration of  $0.17 \text{ mg kg}^{-1}$ .

Digestion of plant material was done by placing 1 or 2 g of tissue or grain in a ceramic crucible and dry-ashing at  $450^{\circ} \text{C}$  for 10 hours. Ashed samples were then dissolved in 5 mls of  $6 \text{ M HNO}_3$  and filtered through number 2 Whatman filter paper. Soil samples were extracted with the ammonium bicarbonate-DTPA (AB-DTPA) extract as described by Soltanpour et al. (1982) and the acid ammonium oxalate extract (Tamm's reagent) as described by Reisenauer (1965).

Homogeneous digests or extracts were made by combining a number of similar acid digests or extracts. The homogeneous solutions were divided and analyzed by the two methods. Solutions analyzed by DCP contained  $1000 \text{ mg L}^{-1} \text{ Li}$  as an ionization suppressant while solutions

analyzed by AAS contained  $1000 \text{ mg L}^{-1}$  Al acting to suppress the formation of Mo oxides.

The plasma unit used in this study was a Spectraspan model SS3A manufactured by Spectrametrics, Inc., now a subsidiary of Beckman Instruments. The atomic absorption unit used was an Instrumentation Laboratories model Video-12. Instrument parameters were adjusted to the manufacturers recommendations.

Analysis wavelengths for each of the methods were chosen for maximum sensitivity based on manufacturers information. For DCP, the 379.8 nm emission line was used with a reported linear dynamic range of 0.04-100  $\text{mg L}^{-1}$ . For AAS, the 313.3 nm line was used with a reported linear dynamic range of 0.2-60  $\text{mg L}^{-1}$ . The low end of the linear dynamic range is frequently referred to as the determination limit. Definitions of the determination limit vary but are usually expressed as 5 to 10 times the detection limit.

The analyte addition method of analysis was used for all samples analyzed by AAS. Additions of Mo were 0, 0.5, 1.0, 1.5, and 2.0  $\text{mg L}^{-1}$ . To check the accuracy of the AAS method, log-log plots of the change in absorption (relative to the solution without added Mo) versus the added Mo concentration were done for each sample. A slope of one would indicate the theoretical response in absorption with a change in Mo concentration and would reflect the absence of interferences. Solutions analyzed by DCP were compared to external standards, a procedure that would be used for routine multielement analysis of plant and soil samples. Results for both methods reflect the mean of six replicate analyses.

### Results and Discussion

Twelve of the 16 samples had log-log slopes from the change in absorption versus concentration plots near 1.0 (Table 1). This indicates the expected response in absorption with a change in Mo concentration and that the analyte addition procedure yielded acceptable results. The log-log slopes of the remaining 4 samples ranged from 0.79 to 0.84.

Molybdenum concentrations given in Table 1 are expressed on a dry weight basis because the dilution factor for some samples varied slightly between the two methods. The total range of Mo concentrations in the analyzed solutions for the AAS method was 0.03 to 7.2 mg L<sup>-1</sup>. Sample solutions without added Mo for both alfalfa samples, both grain samples, and both extractants from the low Mo soil had Mo concentrations below the determination limit suggested by the manufacturer.

Regressing the DCP results on the AAS results utilizing all points yields an equation with a slope of 0.92 and an intercept of 0.3. The equation has a coefficient of determination ( $r^2$ ) of 0.97 and is significant at the 99% probability level. At the 95% probability level, the slope of the line is not significantly different than 1.0 and the intercept is not significantly different than zero.

Removing the four data points with log-log slopes less than 0.95 from the regression calculation yields an equation with a slope of 1.01 and an intercept of -0.4. The coefficient of determination remains at 0.97 and is significant at the 99% probability level. The slope of the line is not significantly different than 1.0 and the intercept is not significantly different than zero at the 95% probability level. Both

Table 1. Mean and standard deviation of Mo concentrations in plant tissues and soils determined by AAS and DCP and the mean slope of the log-log plots from the AAS method.

Sample	log-log slope	AAS	DCP
<u>Corn</u>			
low <sup>1</sup>	1.0	2.4 $\pm$ 0.4	2.7 $\pm$ 0.2
medium	1.0	78 $\pm$ 4	83 $\pm$ 4
high	0.84	162 $\pm$ 11	155 $\pm$ 5
<u>soybeans</u>			
low	1.0	2.2 $\pm$ 0.3	2.9 $\pm$ 0.3
medium	0.83	92 $\pm$ 10	106 $\pm$ 7
high	0.81	180 $\pm$ 17	156 $\pm$ 10
<u>alfalfa</u>			
A	1.0	0.3 $\pm$ 0.1	1.3 $\pm$ 0.2
B <sup>2</sup>	1.0	0.3 $\pm$ 0.2	1.0 $\pm$ 0.2
<u>corn grain</u> <sup>2</sup>	1.0	0.4 $\pm$ 0.1	0.4 $\pm$ 0.1
<u>wheat flour</u>	1.0	0.4 $\pm$ 0.1	0.4 $\pm$ 0.04
<u>AB-DTPA</u>			
low	0.99	0.1 $\pm$ 0.05	0.1 $\pm$ 0.01
medium	1.03	8.0 $\pm$ 0.5	7.5 $\pm$ 0.7
high	0.99	14.6 $\pm$ 0.4	15.3 $\pm$ 0.4
<u>Tamm's reagent</u>			
low	0.98	0.5 $\pm$ 0.1	0.5 $\pm$ 0.1
medium	0.79	34.2 $\pm$ 3.3	22.2 $\pm$ 2.1
high	0.98	81 $\pm$ 3	53 $\pm$ 3

<sup>1</sup>Low, medium or high refers to the expected relative Mo concentration based on the respective sludge application rates from the field experiment.

<sup>2</sup>Two grams of sample was ashed.

regression calculations indicate a favorable comparison between the two methods. Removal of the four data points improved the slope of the regression line slightly and made the intercept slightly worse.

The major limitation of the DCP method of Mo determination is the enhancement of Mo readings that becomes apparent at low Mo concentrations. The problem can be associated with samples that have high background levels of the macro-nutrients. The alfalfa samples were determined to have Mo concentrations  $0.7$  to  $1.0 \text{ mg kg}^{-1}$  higher by the DCP method as compared to the AAS method (Table 1). In addition, the NBS citrus leaves sample, which has a certified Mo concentration of  $0.17 \text{ mg kg}^{-1}$ , was found to have  $1.7 \text{ mg kg}^{-1}$  by the DCP method. The two methods gave comparable results with grain samples and soil extracts, which would have low background levels of the macro-nutrients.

Mortvedt (1981) noted the enhancement problem with the NBS orchard leaves sample. This sample has a certified Mo concentration of  $0.3 \text{ mg kg}^{-1}$  and was found to have  $0.85 \text{ mg kg}^{-1}$  Mo by an argon plasma method. The type of plasma or the wavelength used were not specified.

Data presented by Johnson et al. (1979a, 1979b) has identified spectral interference of Mo at the  $379.8 \text{ nm}$  line by Ca, Mg, and molecular emission from unidentified carbon species. Molecular emission would appear to be ruled out with these samples since Mo readings were not enhanced with the grain samples or the soil extracts, all of which would contain considerable quantities of carbon.

Potential enhancement by Ca and Mg was investigated by preparing standards with levels of these elements matching what would be expected in the digest solution of the NBS citrus leaves sample (certified concentrations are 3.15% for Ca and 0.58% for Mg resulting in 3150 mg



$L^{-1}$  and  $58 \text{ mg } L^{-1}$  in the digest solution, respectively). Analysis of the NBS citrus leaves sample with these standards indicated a Mo concentration of  $0.29 \text{ mg kg}^{-1}$ , a reduction of  $1.41 \text{ mg kg}^{-1}$  Mo as compared to analysis without Ca and Mg in the standards.

These results would suggest that the use of background levels of the macro-nutrients to reduce enhancement of Mo readings at low Mo concentrations is necessary. Johnson et al. (1979a) suggested a linear correction procedure for Ca enhancement at the 379.8 nm line by identifying the stray light concentration equivalent at various Ca concentrations up to  $500 \text{ mg } L^{-1}$ . At the  $500 \text{ mg } L^{-1}$  Ca concentration, these researchers obtained only a  $0.07 \text{ mg Mo } L^{-1}$  enhancement. This amount of enhancement would not be considered significant enough to effect the precision of Mo levels measured in soil extracts and plant digest solutions by DCP.

Therefore, based on the results from this study and those of Johnson et al. (1979a), Ca concentrations between  $500 \text{ mg } L^{-1}$  and  $3150 \text{ mg } L^{-1}$  will likely cause an enhancement of Mo readings and poor analytical results unless a correction procedure is used. The data reported in this paper suggests that the enhancement correction procedures should be used when solutions contain  $< 1.0 \text{ mg Mo } L^{-1}$ , particularly for samples like plant tissue digests having higher levels of Mg and Ca. Use of the linear correction method for Mg and Ca concentrations normally occurring in plant tissue digests and soil extracts warrants further investigation.

### Conclusions

Direct-current plasma emission is an acceptable method for determining Mo concentrations in plant tissue digests and soil extracts. Difficulties with enhancement of Mo readings may occur when low concentrations of Mo ( $< 1.0 \text{ mg L}^{-1}$ ) are analyzed in the presence of Ca and Mg, particularly at concentrations normally found in plant tissue digest solutions. Preliminary data suggests this enhancement problem can be partially overcome by the inclusion of Ca and Mg in the Mo standards at concentrations expected in the digest solutions.

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### CHAPTER III

#### MOLYBDENUM ACCUMULATION BY CORN AND SOYBEANS GROWN ON A SOIL AMENDED BY A MOLYBDENUM-RICH SEWAGE SLUDGE

##### Abstract

Applications of Mo ranging from 0 to 300 kg ha<sup>-1</sup> from a sewage sludge containing 1500 mg kg<sup>-1</sup> Mo were studied in two separate experiments with corn (*Zea mays* L.) and soybeans (*Glycine max* L.) over a three year period. Total soil analysis accounted for 97% of the applied Mo in two sludge treatments. Concentrations of Mo in whole plant, diagnostic tissue and grain samples from both crops were significantly increased with any sludge treatment. Lime residues in the sludge increased soil pH's from 4.8 to 7.1 resulting in further Mo accumulation in the plant samplings.

Four soil extractants (1.0 M NH<sub>4</sub>HCO<sub>3</sub> + 0.005 M DTPA, 1.2 M NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>, Tamm's acid ammonium oxalate, and 0.5 M (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>) in combination with soil pH were evaluated for their ability to predict Mo concentrations in the plant samplings. Soil pH became significant in the prediction equations when considering more than one year's data. None of the extractants were proven to be clearly better or worse than the others. Further testing of the ammonium bicarbonate-DTPA extractant is recommended because of its potential for simultaneous use with other plant nutrients and heavy metals in the soil during multielement analysis, as well as the ease by which it can be analyzed using plasma emission.

## Introduction

Land application of sewage sludges is an attractive waste management alternative for many municipalities. The accumulation of heavy metals in the soil has long been recognized as one of the main concerns with this option. The metals typically studied are Zn, Cu, Cr, Cd, Ni, or Pb with little information in the literature about Mo. The primary hazard of Mo contamination of soils is the potential for these soils to produce levels of Mo in forages that would induce molybdenosis in ruminants (Cast, 1976).

The range of concentrations of Mo in sewage sludges can be quite wide. In a survey of Michigan sludges, Jacobs et al. (1981) reported a range of 6 to 3700 mg kg<sup>-1</sup> Mo with a median of 32 mg kg<sup>-1</sup>. Dowdy et al. (1976) reported a range of 5-39 mg kg<sup>-1</sup> Mo from a survey of 200 sludges from eight states. Molybdenum concentrations ranged from <1 to 73 mg kg<sup>-1</sup> in 44 sewage sludges from Iowa communities (Tabatabai and Frankenberger, 1979).

Applications of up to 0.41 kg ha<sup>-1</sup> of Mo in a greenhouse study from a liquid sludge at two pH levels, 6.1 and 7.2, produced Mo concentrations of 94 mg kg<sup>-1</sup> in white clover and 20.4 mg kg<sup>-1</sup> in ryegrass at the high pH level (Williams and Gogna, 1981). Clover grown in the greenhouse in a soil amended with up to 17 kg ha<sup>-1</sup> Mo from a Mo-rich sewage sludge contained up to 31.0 mg kg<sup>-1</sup> Mo at pH 8.0 (Davis, 1981). Molybdenum applications of 0 to 4.8 kg ha<sup>-1</sup> from a liquid sludge failed to produce a significant increase in the Mo concentration in rye clippings grown on an acid soil with a pH range of 4.5 to 5.2 (King and Morris, 1972).

The purpose of this study was to evaluate the potential for Mo uptake by corn and soybeans grown under field conditions on a soil amended with a Mo-rich sewage sludge.

### Materials and Methods

Two field experiments arranged in randomized complete block designs with four replications were established in Spring, 1982 on a Metea loamy sand soil (mixed, mesic, Arenic Hapludalf) and continued for three growing seasons. Experiment 1 had a plot size of 5.8x30.5 m and was divided in half to grow two crops, corn (*Zea mays* (L.) cv. #3901, Pioneer) and soybeans (*Glycine max* (L.) cv. Corsoy). The sludge treatments were 0, 22, and 44 Mg ha<sup>-1</sup> (dry solids basis) in April 1982 with another 20 and 50 Mg ha<sup>-1</sup> applied in November 1982 for total additions of 0, 42, and 94 Mg ha<sup>-1</sup>, respectively. Experiment 2 had a plot size of 5.8x12.2 m and corn was grown. The sludge treatments were 0 or 94 Mg ha<sup>-1</sup> (dry solids basis) applied in April 1982 with 200 Mg sludge ha<sup>-1</sup> being applied to the untreated plots and an additional 100 Mg sludge ha<sup>-1</sup> being applied to the remaining plots in November, 1982. These total additions of 200 and 194 Mg ha<sup>-1</sup> were made to compare "single" and "split" applications.

The sludge had been anaerobically digested and dried on open-air drying beds. Starter fertilizers were not used but an application of urea supplying 168 kg N ha<sup>-1</sup> was made each year to untreated plots growing corn. Surface soil samples were taken before planting and after harvest each year by compositing twenty, 2.5 cm cores per plot. Two lime applications totaling 4290 kg ha<sup>-1</sup> were made in April 1984 in an attempt to raise the pH of the untreated plots from 4.8-5.0 to 6.5.

Plant samples were taken from both crops at three growth stages— above ground plants at 25-31 cm high for corn and 18-23 cm high for soybeans, diagnostic leaves at tasseling for corn and at the onset of flowering for soybeans, and grains at harvest time. Plots were harvested by handpicking (corn) or machine harvesting (soybeans) the center 3.1 m of six rows in each plot and weighing. The grains were subsampled to determine gravimetric moisture content and to use for grain analysis.

To facilitate mass balance calculations, bulk density samples were taken in October 1984 from the corn plots in experiment 1 as described by Blake (1965). Ten cores were taken from each plot. Depth of tillage measurements were made at this time by visual observation of sludge particles in 20 soil cores taken randomly from plots in experiment 2.

All whole plant and diagnostic leaf samples were washed with 0.1% sodium dodecyl sulfate for 10 seconds, rinsed thoroughly with deionized distilled water, dried at 65°C for 24 hours and ground in a Wiley mill through a 40 mesh screen. Grain samples were dried at 65°C for 48 hours and ground in a cyclone mill through a 40 mesh screen. Digestion of plant material was done by placing 1 g of tissue or 2 g of grain in a ceramic crucible and dry ashing at 450°C for 10 hrs. The ash was dissolved in 5 ml of 6 M  $\text{HNO}_3$  and brought to 10 ml with 2000 mg  $\text{Li}^+$   $\text{L}^{-1}$  as  $\text{LiCl}$ .

All soil samples were air-dried and crushed to pass a 2 mm sieve using a mortar and pestle. The pH of the soil was measured on a stirring 2:1 water:soil suspension. Molybdenum extraction procedures are presented in Table 1. The AB-DTPA extracts were neutralized with

Table 1. Molybdenum extraction procedures.

Extractant	Solution:soil (ml:g)	Shaking time
1.0 M $\text{NH}_4\text{HCO}_3$ , 0.005 M DTPA, pH 7.6 (AB-DTPA)	2:1	15 min.
1.2 M $\text{NaC}_2\text{H}_3\text{O}_2$ , pH 4.8 (NaOAc)	2:1	20 min.
0.175 M $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , 0.01 M $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (Tamm's reagent)	5:1	overnight
0.5 M $(\text{NH}_4)_2\text{HPO}_4$ , pH 6.0 (DAHP)	2:1	overnight



HNO<sub>3</sub> before analysis by plasma emission as described by Soltanpour et al. (1982). Total soil analysis was performed, according to Soltanpour et al. (1982), on surface soil samples taken in Fall 1984 from the corn plots in experiment 1.

Four sludge samples were taken in Spring 1982 and in Fall 1982. The samples were ground in Coors CD-99 aluminum oxide grinding vials. Sludge digestion was done by placing 1 gm samples in teflon beakers covered with teflon watch covers and refluxing on a sand bath at 120°C with 20 ml of concentrated HNO<sub>3</sub> overnight. The covers were then removed and the volume was reduced to approximately 3 ml. Fifteen ml of concentrated HF and 2 ml of concentrated HClO<sub>4</sub> were then added, the watch covers were returned and the beakers were allowed to reflux on a sandbath at 120°C overnight. The covers were removed and the samples were allowed to come to dryness. The dried material was dissolved in 12.5 ml of 6 M HNO<sub>3</sub> and brought to 25 mls with 2000 mg Li<sup>+</sup> L<sup>-1</sup>.

Analysis for Mo was performed on duplicate digests or extracts using direct current argon plasma atomic emission spectroscopy (DCP-AES). Emission was measured on the 379.825 nm wavelength. With the exception of the NaOAc and DAHP extracts, all digests and extracts were analyzed with a final Li<sup>+</sup> concentration of 1000 mg L<sup>-1</sup> acting as an ionization suppressant.

## Results and Discussion

### Sludge Applications

The average Mo concentration was  $1500 \text{ mg kg}^{-1}$  for the sludge applied in April and November of 1982. Total Mo additions for these two sludge applications were 0, 63, and  $141 \text{ kg ha}^{-1}$  for experiment 1 and 291 and  $300 \text{ kg ha}^{-1}$  for experiment 2.

Mass balance calculations were performed on the corn plots in experiment 1 to determine the amount of the applied Mo that could be accounted for in the plow layer after three years. To minimize the assumptions normally made for such calculations, bulk density and depth of tillage measurements were made. Bulk density measurements indicated a mean bulk density of  $1.4 \text{ g cm}^{-3}$  for the corn plots in experiment 1 with no significant differences between sludge treatments. A mean tillage depth of 23 cm was found by visual observation of sludge particles in soil cores taken from experiment 2. Tillage operations were identical for both experiments. The mass of a hectare furrow slice would then be  $3.2 \times 10^6 \text{ kg}$ .

The soil contained  $5.2 \text{ mg Mo kg}^{-1}$  which was increased by  $19.0 \text{ mg kg}^{-1}$  and  $42.5 \text{ mg kg}^{-1}$  for Mo-sludge additions of 63 and  $141 \text{ kg ha}^{-1}$ , respectively. These increases are equivalent to 61 and  $136 \text{ kg ha}^{-1}$  which represent 97% of the application rates calculated from the sludge analysis for both sludge treatments. Therefore, little movement of Mo out of the plow layer was found, which is in agreement with calculations made by Page (1974) where 89% of the Mo applied with sludges over a 12 year period was recoverable in the surface 20 cm of soil.

The sludge contained lime residues which increased the soil pH's of the treated plots (Table 2). The lime application in April 1984 had a small effect on the soil pH of plots over the 1984 growing season as indicated by the small rise in pH on the untreated plots.

### Crop Responses

Corn and soybean yields were not significantly affected by sludge Mo loadings from 1982-1984 (Table 3). Molybdenum concentrations in plant tissues and grains are shown in Table 4. With the exception of the whole soybean plants in 1982, all sludge treatments significantly increased the Mo concentration in all samplings of both crops. The concentrations of Mo in corn whole plant and diagnostic leaf tissue samples are consistently higher than comparable samplings of soybeans. Previous work by Kubota et al. (1961), Jensen and Lesperance (1971) and Kretschmer and Allen (1956) has suggested that legumes accumulate more Mo than nonlegumes, although neither corn nor soybeans were involved in these studies. Varietal differences with regard to uptake of Mo have been identified with corn (Dios and Broyer, 1965; Lutz et al., 1972). One could expect differences in Mo uptake between different varieties of soybeans, therefore, using a different variety of corn and soybeans could reverse these results.

Greater concentrations of Mo are translocated into soybean than into corn grain. Molybdenum additions to soils caused levels in soybeans to increase from 10-20 mg kg<sup>-1</sup> for control plants to about 100 mg kg<sup>-1</sup> and 240 mg kg<sup>-1</sup> for plants grown in sludge treated plots. The same Mo additions only increased corn grain Mo from <1 mg kg<sup>-1</sup> for

Table 2. Mean soil pH values from fall soil samples collected in 1982-1984.

Sludge Treatment	1982	1983	1984
- Mg ha <sup>-1</sup> -		<u>experiment 1</u>	
<u>corn</u>			
0	4.6	4.7	5.0
42	5.0	5.7	6.3
94	5.2	6.2	6.7
<u>soybeans</u>			
0	4.6	4.8	5.3
42	5.0	5.7	6.2
94	5.3	6.4	6.9
		<u>experiment 2</u>	
<u>corn</u>			
194 <sup>1</sup>	5.4	6.5	7.1
200	4.8	5.8	6.6

<sup>1</sup>194 Mg ha<sup>-1</sup> is the split sludge application, 200 Mg ha<sup>-1</sup> is a single sludge application made in fall 1982.

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Table 3. Mean grain yields<sup>1</sup> for corn and soybeans grown in 1982-1984.

Sludge Treatment	1982	1983	1984
- Mg ha <sup>-1</sup> -	----- kg ha <sup>-1</sup> -----		
	<u>experiment 1</u>		
<u>corn</u>			
0	5890	6000	4120
42	5540	4660	4060
94	6040	5890	4650
<u>soybeans</u>			
0	1860	2650	1240
42	1650	2840	1520
94	1760	2790	1670
	<u>experiment 2</u>		
<u>corn</u>			
194 <sup>2</sup>	6100	6340	4990
200	6470	5780	5030

<sup>1</sup>Yields were not significantly affected by sludge treatments.

<sup>2</sup>194 Mg ha<sup>-1</sup> is the split sludge application, 200 Mg ha<sup>-1</sup> is a single sludge application made in fall 1982.

Table 4. Mean Mo concentrations found in plant tissue and grain samples grown in 1982-1984.

Sludge Treatment	<u>whole plant</u>			<u>diagnostic tissue</u>			<u>grains</u>		
	1982	1983	1984	1982	1983	1984	1982	1983	1984
- Mg ha <sup>-1</sup> -	----- mg kg <sup>-1</sup> -----								
	<u>experiment 1</u>								
<u>corn</u>									
0	1.9	7.5	6.0	3.0	2.2	8.8	0.2	0.4	0.6
42	29.8	265	300	40.5	137	265	1.5	3.7	3.6
94	47.0	365	724	63.0	247	697	2.0	5.7	6.9
LSD <sup>1</sup>	8.6	95.2	124	8.2	46.3	130	0.2	0.8	1.0
<u>soybeans</u>									
0	2.7	3.1	5.4	2.1	2.4	9.3	14.3	8.9	19.9
42	43.9	140	185	38.1	103	158	70.9	107	114
94	56.4	321	459	52.8	268	452	122	241	242
LSD	17.8	61.2	77.8	13.5	37.2	72.4	19.8	39.0	33.3
	<u>experiment 2</u>								
<u>corn</u>									
194 <sup>2</sup>	92.7	730	1300	122	462	804	3.6	12.0	16.5
200	6.6	330	630	6.4	197	408	0.5	6.3	9.2
LSD	48.0	137	326	25.1	163	129	0.3	2.2	2.9

<sup>1</sup>Least significant difference at the 95% probability level.

<sup>2</sup>194 Mg ha<sup>-1</sup> is the split sludge application; 200 Mg ha<sup>-1</sup> is a single sludge application made in fall 1982.

control plants to 3-4 and 6-7 mg kg<sup>-1</sup> due to sludge treatments. Therefore, corn grain appears to be an efficient excluder of Mo relative to the other corn and soybean plant samplings.

In experiment 2, two applications of sludge resulted in significantly greater Mo concentrations in the corn samplings from 1983 and 1984 than the single sludge application (Table 4), even though Mo loadings were approximately equal. This is likely due to the lower pH of the plots receiving one sludge application (Table 2) since soils receiving split applications had an additional period of time for pH's to increase. An increase in soil pH has been shown to increase the availability of Mo from sewage sludges (Williams and Gogna, 1981; Davis, 1981). Similarly, increases in Mo concentrations from 1983 to 1984 in all treatments of both experiments are likely due to increases in soil pH brought about by the lime residues in the sludge or the lime applications in April 1984.

Animal consumption of soybean or corn whole plants or diagnostic leaves is relatively insignificant. However, Mo concentrations of 10-20 mg kg<sup>-1</sup> suggested by Kubota et al. (1967) as being hazardous to livestock are exceeded with any sludge treatment. Corn grain fed as high moisture corn is a common feed source for cattle. Data from experiment 2 indicates that if this grain were to be a large portion of a ration for cattle, the potential for molybdenosis would be increased.

#### Extractable Molybdenum

Extractable Mo data from the spring 1983 and 1984 and the fall 1984 soil samplings are presented (Table 5). The spring soil samples are



Table 2. Levels of extractable Mo from soils collected in spring 1983 and spring and fall 1984.		NADAC		Tamm's		DAHP			
Treatment	sp83	sp84	f84	sp83	sp84	f84	sp83	sp84	f84

Table 5. Levels of extractable Mo from soils collected in spring 1983 and spring and fall 1984.

TABLE 3. Levels of exchangeable Na from soil in spring 1983 and spring 1984												
Sludge Treatment	AB-DTPA		NaOAc		Tamm's			DAHP				
	sp83	sp84	f84	sp83	sp84	f84	sp83	sp84	f84	sp83	sp84	f84
----- mg kg <sup>-1</sup> -----												
<u>experiment 1</u>												
<u>corn</u>												
0	0.2	0.3	0.3	0.1	0.2	0.1	1.0	0.9	1.2	0.1	0.2	0.2
42	8.6	8.2	5.3	0.8	0.9	0.8	16.1	14.8	15.8	4.2	5.6	4.2
94	16.1	15.1	10.6	1.6	1.6	1.4	31.4	29.6	31.4	7.6	9.7	7.2
LSD <sup>1</sup>	1.8	1.4	1.8	0.2	0.2	0.1	4.0	2.9	5.9	1.0	0.7	0.8
<u>soybeans</u>												
0	0.2	0.2	0.4	0.1	0.3	0.1	1.2	1.1	1.5	0.1	0.1	0.3
42	9.0	8.7	5.6	0.9	1.0	0.8	18.2	15.3	14.6	4.4	6.1	4.4
94	20.7	17.4	10.8	1.8	1.9	1.4	38.9	31.7	29.4	9.8	12.1	7.5
LSD	2.5	1.6	1.3	0.2	0.2	0.2	5.8	3.8	2.5	1.0	1.2	0.8
<u>experiment 2</u>												
<u>corn</u>												
194 <sup>2</sup>	40.3	30.4	20.3	3.1	3.1	2.1	71.7	85.2	75.7	15.0	17.4	11.0
200	32.0	29.4	24.4	3.5	3.1	2.5	71.0	73.4	71.3	16.9	18.8	13.4
LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.<sup>2</sup>194 Mg ha<sup>-1</sup> is the split sludge application, 200 Mg ha<sup>-1</sup> is a single sludge application made in fall 1982.

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presented and used in regression calculations because it would be desirable to predict Mo concentrations that would occur in a crop from a soil sampling prior to the planting of that crop. The fall soil sampling data is presented for comparative purposes.

The sludge treatments significantly increased extractable Mo with all the extractants at each soil sampling in experiment 1 (Table 5). After the second application of sludge on experiment 2, no significant differences in extractable Mo were found indicating that comparable Mo loadings were made to both treatments. This further supports the reasoning that pH differences caused the lower accumulation of Mo in corn grown on plots receiving one large application of sludge as compared to "split" sludge applications.

The ability of the extractants to remove Mo from the soil follows the order: Tamm's reagent > AB-DTPA > DAHP > NaOAc. The ability of the anions in these solutions to remove Mo from the soil is in agreement with the order of anions presented by Kurtz et al. (1946) based on their ability to remove phosphate from the soil. Molybdate is similar to phosphate in that significant amounts can be adsorbed by iron and aluminum compounds. Tamm's reagent extracts the greatest amount of Mo because it will dissolve many of these iron and aluminum compounds, releasing adsorbed Mo, and making it available for chelation with the oxalate anion. The AB-DTPA extractant would be expected to extract more Mo than NaOAc or DAHP because, in addition to the ion replacement capabilities of the bicarbonate anion, the DTPA will compete with the Mo for Fe resulting in higher concentrations of Mo in the solution phase.

Levels of Mo extracted with AB-DTPA, NaOAc, and DAHP decreased from Spring 1983 to Fall 1984 while levels of Mo extracted with Tamm's

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reagent remained relatively constant (Table 5). Acid soils and oxides of iron and aluminum have been shown to adsorb large quantities of Mo (Smith and Leeper, 1969; Reisenauer et al., 1962). Substantial quantities of Fe were added to the soil with the sludge, which contained an average of  $21000 \text{ mg kg}^{-1}$  of Fe. This adsorbed Mo would not be easily extracted with weak extractants such as AB-DTPA, NaOAc, or DAHP but would be extracted with Tamm's reagent. The weaker extractants are indicating a decline in available Mo while the plant tissue data is indicating the opposite.

Multiple regression results between Mo concentrations in soybean diagnostic tissues and extractable Mo and soil pH are shown for the spring soil samplings from 1983 and 1984 (Table 6). Regression results for individual years indicate that a large portion of the variability in Mo concentration is accounted for by extractable Mo, and soil pH is not significant in the equations. None of the extractants are clearly better or worse than the others.

Combining the data over both years introduces more variability in Mo concentrations which is generally not accounted for by extractable Mo. Thus, pH enters as a significant variable in three out of the four equations since soil pH and the Mo concentrations in the tissues are increasing each year. However, soil pH and extractable Mo are not truly independent of each other in these experiments since both are acting as indicators of the sludge loadings. These two variables were studied as independent variables in another experiment described in the subsequent chapter.

Extractable Mo did not enter into the prediction equations as a significant variable for multiple regressions utilizing corn diagnostic

Table 6. Regression coefficients and significant variables from multiple regressions between Mo concentrations in 1983 and 1984 soybean diagnostic tissue and extractable Mo and soil pH from soils sampled in spring 1983 and 1984.

year(s)	<u>AB-DTPA</u>		<u>NaOAc</u>		<u>Tamm's</u>		<u>DAHP</u>	
	R <sup>2</sup>	S.V. <sup>@</sup>	R <sup>2</sup>	S.V.	R <sup>2</sup>	S.V.	R <sup>2</sup>	S.V.
1983	0.95 <sup>#</sup>	EMo	0.95	EMo	0.92	EMo	0.97	EMo
1984	0.89	EMo	0.94	EMo	0.88	EMo	0.92	EMo
both	0.84	pH	0.87	pH,EMo	0.84	pH	0.91	EMo

<sup>@</sup> significant variables (S.V.) in the prediction equation, EMo is the extractable Mo with the indicated extractant

<sup>#</sup> all regression coefficients are significant at the 95% probability level

tissue data and the spring soil samplings from 1983 and 1984. Soil pH was significant in the prediction equations in all cases. Regression coefficients, significant at the 95% probability level, between Mo concentrations in the tissue and soil pH were 0.86, 0.82, and 0.81 for 1983, 1984 and the two years combined, respectively. The difference in the ability of the extractants to predict Mo concentrations in the corn as opposed to the soybeans would be partially due to the deeper root systems of the corn crop. Generally, predicting nutrient concentrations in deep rooted crops with a surface soil sample is more difficult than for a shallow rooted crop.

Multiple regressions between Mo concentrations in the grains and soil pH and extractable Mo from the combination of the data from the spring 1983 and 1984 soil samplings indicated that extractable Mo was very effective in predicting Mo concentrations in these samples. Regression coefficients ranged from 0.79 to 0.85 for the corn grain and from 0.93 to 0.95 for the soybean grain with extractable Mo always significant in the prediction equations. Soil pH was a significant variable in the prediction equations in all cases except with the NaOAc and DAHP extractants and soybean grain. The influence of soil pH on Mo concentrations in corn and soybean whole plant and diagnostic tissue samples identified previously is not as prevalent in the grain samples. Thus, extractable Mo predicts Mo concentrations in the grain more efficiently than in other plant tissue.

Soil pH and Mo extracted with the four extractants used in this study could not acceptably predict Mo concentrations in the corn diagnostic tissue or in soybean diagnostic tissue when considering more than one season's growth. Analytical difficulties were encountered with



the NaOAc and DAHP extractants due to their high salt concentrations which would make them undesirable for routine use utilizing d. c. plasma emission. The remaining two extractants require more testing under a variety of soils, crops and Mo levels before either could be recommended for use with Michigan soils.

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## CHAPTER IV

### INFLUENCE OF SOIL pH ON EXTRACTABILITY AND PLANT AVAILABILITY OF MOLYBDENUM FROM INORGANIC AND SEWAGE SLUDGE SOURCES

#### Abstract

Molybdenum additions of 30, 60, and 120 mg kg<sup>-1</sup> from Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O and 20, 44, and 94 mg kg<sup>-1</sup> from an anaerobically digested sewage sludge significantly increased Mo concentrations in corn (*Zea mays* L.), soybeans (*Glycine max* L.), and alfalfa (*Medicago sativa* L.) grown under greenhouse conditions. Increases in soil pH significantly increased Mo accumulation in the three crops from both Mo sources. Molybdenum extracted with Tamm's acid ammonium oxalate was not significantly influenced by soil pH, while Mo extracted from sludge treated soils with the ammonium bicarbonate-DTPA extractant was significantly increased with increasing soil pH.

Multiple regressions between Mo concentrations in the crops and extractable Mo and soil pH indicated that soil pH was a significant variable in 17 out of 24 prediction equations. The ammonium bicarbonate-DTPA extractant was more effective in predicting Mo concentrations in plant tissue than Tamm's acid ammonium oxalate with sludge Mo additions and is more effective with sludge source Mo as compared to inorganic source Mo. However, whether AB-DTPA can be used to predict potential problems with Mo additions to soil-crop systems needs to be tested by field evaluation.

## Introduction

Land application of sewage sludges is an attractive waste management alternative for many municipalities. The accumulation of heavy metals has long been recognized as one of the main concerns with this option but little information is in the literature regarding Mo accumulation in the soil from sludge additions. The primary hazard of Mo contamination of soils is the potential for the soils to produce levels of Mo in forages that would induce molybdenosis in ruminants (CAST, 1976).

Increases in soil pH have been shown to increase the availability of native soil Mo, inorganic Mo additions, and sludge Mo additions (Davis, 1981; James et al., 1968; Gupta, 1969; Gurley and Giddens, 1969; Williams and Gogna, 1981). Normal recommendations for soil pH when sludge is applied are to keep the pH near neutral or above to reduce the availability of the cationic heavy metals. This management procedure will effectively increase the availability of the Mo in the sludge and the soil.

A field study, described in the previous chapter, conducted with the Mo-rich sewage sludge used in this work has indicated that lime residues in the sludge raised soil pH levels from 4.9 to 7.1 over a three year period after  $300 \text{ Mg ha}^{-1}$  of sludge was applied. The simultaneous addition of Mo and lime residues made it difficult to establish the influence of pH on the availability and extractability of the Mo in the sludge. The purpose of this study was to establish soil pH as a variable independent of sludge loading rates and then to evaluate the influence of soil pH on the uptake and extractability of Mo from inorganic and sludge sources.

### Materials and Methods

A Metea sandy loam (mixed, mesic, Arenic Hapludalf) taken from a field experiment that had been amended with 0, 42, 94, and 200 Mg ha<sup>-1</sup> of an anaerobically digested sewage sludge containing 1500 mg kg<sup>-1</sup> Mo was utilized in a greenhouse study. Details of the field experiment were described in the previous chapter. Molybdenum additions from the sludge were equivalent to 0, 20, 44, and 94 mg kg<sup>-1</sup> of dry soil. Each of the four sludge treatments were adjusted to three pH levels with CaO. The resulting pH levels are given in Table 1. Little or no change occurred in the soil pH levels over the course of the experiments. Molybdenum additions of 0, 30, 60, and 120 mg kg<sup>-1</sup> as Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O were made to the soil that had not received sludge amendments giving a 3 x 4 factorial arrangement of treatments. The remaining three sludge treatments were considered as individual experiments with soil pH as the treatment.

Two sets of pots were established, both arranged in randomized complete blocks, with two cuttings of alfalfa (*Medicago sativa* (L.) cv. Oneida) grown in one set of pots and two cuttings each of corn (*Zea mays* (L.) cv. #3901, Pioneer) and soybeans (*Glycine max* (L.) cv. Corsoy) grown in the other set of pots. Corn and soybean cuttings were alternated with corn being the first crop grown.

The alfalfa pots contained 7 kg of dry soil treated with 450 mg K kg<sup>-1</sup> as KCl at planting. Eight plants were grown in each pot which were cut back to 5 cm after 10 weeks of growth. The two alfalfa samplings reported represent 35 days of new growth each after the initial cutting.

The corn and soybean pots contained 4 kg of dry soil treated with



Table

Sludge

--- M

Table 1. Soil pH levels at the initiation of the greenhouse experiments.

Sludge loading	pH level		
	1	2	3
--- Mg ha <sup>-1</sup> ---			
0	6.0	7.0	7.7
42	5.4	7.5	8.0
94	6.2	7.5	8.2
200	6.6	7.1	7.8

180 mg K kg<sup>-1</sup> as KCl before the first cutting of corn was planted. Five corn plants or six soybean plants were grown in each pot. Twenty-five mg N kg<sup>-1</sup> as NH<sub>4</sub>NO<sub>3</sub> were added at planting for each corn cutting. The corn was allowed to grow 35 days after seeding before harvest while the soybeans were allowed 39 days of growth.

All pots were weighed twice a week and watered up to a moisture content of 21% by weight. Supplemental water was added as needed between weighings. Supplemental light was supplied for 16 hours each day. Soil samples were taken at the harvest of each cutting, air-dried and crushed to pass a 2 mm sieve using a mortar and pestel. The pH of the soil was measured on a stirring 2:1 water:soil suspension. Soils sampled after the first corn cutting, the second soybean cutting, and both alfalfa cuttings were extracted with 1.0 M NH<sub>4</sub>HCO<sub>3</sub> + 0.005 M DTPA (AB-DTPA) as described by Soltanpour et al. (1982) and Tamm's acid ammonium oxalate as described by Reisenauer (1965).

Plant samples were rinsed with deionized distilled water, dried at 65° C for 24 hours, and ground in a Wiley mill through a 40 mesh screen. Digestion of plant material was done by placing 1 gm of tissue in a ceramic crucible and dry ashing at 450° C for 10 hours. The ash was dissolved in 5 ml of 6 M HNO<sub>3</sub> and brought to 10 ml with 2000 mg Li<sup>+</sup> L<sup>-1</sup> as LiCl.

Analysis for Mo was performed on duplicate digest or extracts using direct current argon plasma emission spectroscopy. Emission was measured on the 379.8 nm line. All digests and extracts were analyzed with a final Li<sup>+</sup> concentration of 1000 mg L<sup>-1</sup> acting as an ionization suppressant.

## Results and Discussion

Molybdenum concentrations in plant tissue samples from the factorial arrangement of inorganic Mo treatments are presented in Table 2. Analysis of variance for both cuttings of all crops indicated significant pH, added Mo, and pH by added Mo interaction affects, except for the first cutting of alfalfa which had a nonsignificant interaction affect. This interaction affect had been noted in other work (Gupta, 1969) but has not been investigated at levels of added Mo as high as those used in this study. With the exception of the 60 and 120 mg Mo  $\text{kg}^{-1}$  additions at pH level 2 with the second cutting of soybeans, additions of Mo resulted in significant increases in the Mo concentrations of both cuttings of all crops.

Increasing soil pH's generally resulted in significant increases in Mo concentrations at the zero and 30 mg  $\text{kg}^{-1}$  levels of added Mo. With Mo additions of 60 or 120 mg  $\text{kg}^{-1}$ , soil pH had different effects on Mo concentrations, depending on the crop and cutting. Molybdenum concentrations in at least one and sometimes both of the upper pH levels in both cuttings of corn were significantly greater than pH level 1 except at the 60 and 120 mg Mo  $\text{kg}^{-1}$  additions in the second cutting. Similar results were seen with both cuttings of alfalfa with the exception of the 120 mg Mo  $\text{kg}^{-1}$  additions where no significant differences between pH levels were found.

Significant increases in Mo concentrations with increasing soil pH in the first cutting of soybeans occurred at the zero and 30 mg Mo  $\text{kg}^{-1}$  additions but not at the 60 and 120 mg Mo  $\text{kg}^{-1}$  additions. Molybdenum concentrations obtained for the second cutting of soybeans are

Table 2. Mean Mo concentrations in corn, soybeans and alfalfa tissue grown in soils amended with sodium molybdate.

	<u>Mo additions (mg kg<sup>-1</sup>)</u>							
pH level	0	30	60	120	0	30	60	120
	----- mg kg <sup>-1</sup> -----							
	<u>first cutting</u>				<u>second cutting</u>			
	<u>corn</u>							
1	4.8a <sup>1</sup>	117a	242a	380a	4.3a	227a	457a	681b
2	6.4b	174b	244a	337a	10.9b	347c	457a	511a
3	8.6c	242c	419b	903b	11.1b	272b	478a	607b
	<u>soybeans</u>							
1	12.2a	170a	347a	624b	13.4a	1100b	1470b	2150c
2	18.9b	214b	345a	435a	49.1b	1130b	1560b	1460b
3	20.6b	229b	400a	593b	54.5b	516a	618a	654a
	<u>alfalfa</u>							
1	9.0a	193a	299a	645a	11.0a	447a	581a	1060a
2	19.0b	342b	542b	716a	25.0b	725b	862b	1020a
3	19.0b	370b	480b	646a	27.0b	702b	865b	1070a

<sup>1</sup>Treatment means having the same letter within a column and crop are not significantly different at the 5% level by the least significant difference test. Molybdenum additions resulted in significant increases in Mo concentrations in all instances except the second cutting of soybeans at pH level 2 at 60 and 120 mg Mo kg<sup>-1</sup>.

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contradictory with the results from other cuttings of all cultivars, i.e., plant Mo was significantly decreased as pH increased, except where no Mo was added to the soil. The reasons for this can not be explained, but similar results were obtained for the second soybean cutting from the sludge-amended soils (Table 3). The high pH level in conjunction with Mo additions were apparently not toxic to the soybean growth since the dry matter production per pot was not significantly influenced by either variable.

Molybdenum concentrations in plant tissue samples resulting from the pH adjustments of sludge amended soils are presented in Table 3. Increases in soil pH significantly increased Mo concentrations in both cuttings of corn and alfalfa and in the first cutting of soybeans. Exceptions to increased plant Mo with increases in soil pH are the second cutting of corn and the first cutting of soybeans at the high sludge rate where no increase occurred. Enhanced plant uptake due to increasing soil pH is in agreement with other work where sludge Mo rates were lower (Davis, 1981; Williams and Gogna, 1982). As with inorganic Mo additions, Mo concentrations in second cutting soybean tissue decreased with increasing soil pH.

Molybdenum concentrations in the second cutting of any crop are generally higher than their respective first cuttings (Tables 2 and 3). Molybdenum has been shown to move in the soil primarily through a mass-flow mechanism when the saturation extract of the soil contained more than  $4 \text{ ug L}^{-1}$  Mo (Lavy and Barber, 1964). Since each of the second cuttings was grown in a time period with a longer average daylength compared to that for the first cutting, increased transpiration and greater accumulation of Mo could be expected. For example, the first

Table 3. Mean Mo concentrations in corn, soybean, and alfalfa tissue grown in sludge amended soils.

	Mo additions (mg kg <sup>-1</sup> )					
pH level	20	44	94	20	44	94
	----- mg kg <sup>-1</sup> -----					
	<u>first cutting</u>			<u>second cutting</u>		
	<u>corn</u>					
1	30a <sup>1</sup>	125a	213a	74a	315a	329a
2	142b	310b	265a	260b	403b	383a
3	268c	540c	419b	273b	470c	362a
	<u>soybeans</u>					
1	108a	394a	444a	300a	800b	986b
2	232b	427b	418a	736c	1010c	1070b
3	205b	425b	417a	391b	585a	692a
	<u>alfalfa</u>					
1	82a	289a	336a	201a	486a	659a
2	312c	552b	528b	487b	876b	895b
3	238b	521b	497b	483b	773b	944b

<sup>1</sup>Treatment means having the same letter within a column and crop are not significantly different at the 5% level by the least significant difference test.



soybean cutting was planted on 9 December and harvested on 16 January (a time period having the shortest days of the year) while the second cutting was planted on 28 February and harvested on 6 April. During these growth periods, the average dry matter production per pot was 2.9 g for the first cutting and 4.7 g for the second cutting, a further indication of increased growth due to longer daylengths.

Levels of extractable Mo removed by the AB-DTPA extractant and Tamm's reagent from the soils sampled after the first cutting of corn are shown in Table 4. Increasing soil pH with inorganic Mo additions either significantly reduced extractable Mo or did not have a significant affect with either extractant. Molybdenum extracted from sludge treated soils by AB-DTPA was significantly increased by increasing soil pH, but pH had little effect on Mo extracted by Tamm's reagent. The increase in extractable Mo with increasing soil pH would be expected based on previous work in which the activity of molybdate was shown to increase in the water extract of soils after amendments with CaO (Follett and Barber, 1967). The finding that soil pH did not affect Mo extracted by Tamm's reagent agrees with other studies which failed to find significant differences in oxalate extractable Mo with changes in soil pH (Gupta, 1969; Gupta, 1970).

Multiple regressions were performed with the Mo concentrations in a particular cutting of a crop as the dependant variable and the Mo extracted with one of the two extractants and soil pH as the independant variables (Table 5). Because of differences in the Mo concentrations between the two cuttings of each crop grown, a large reduction in the  $R^2$  values was obtained when the data from both cuttings were combined. Therefore, each cutting will be considered individually.

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Table 4. Extractable levels of Mo from soils sampled after the first cutting of corn.

	<u>inorganic Mo additions</u>					<u>sludge Mo additions</u>		
	----- mg kg <sup>-1</sup>					-----		
pH level	0	30	60	120		20	44	94
	----- mg kg <sup>-1</sup>					-----		
	<u>AB-DTPA</u>							
1	0.1a <sup>1</sup>	15.5a	26.1b	64.5c		7.3a	15.8a	28.5a
2	0.2a	12.6a	19.4a	30.5a		8.0a	18.6b	28.9a
3	0.1a	13.9a	28.2b	44.8b		9.3b	19.6b	30.2b
	<u>Tamm's reagent</u>							
1	1.1a	26.4a	42.8a	89.4b		18.4b	34.9a	75.4a
2	1.2a	27.5a	47.3a	70.7a		15.7a	34.8a	74.4a
3	1.1a	26.0a	51.6a	94.3b		16.5a	35.6a	74.8a

<sup>1</sup>Treatment means having the same letter within a column and extractant are not significantly different at the 5% level by the least significant difference test.

Table 5. Regression coefficients and significant variables from multiple regressions between Mo concentrations in plant tissues and extractable Mo and soil pH.

cultivar	<u>inorganic Mo additions</u>				<u>sludge Mo additions</u>			
	<u>AB-DTPA</u>		<u>Tamm's</u>		<u>AB-DTPA</u>		<u>Tamm's</u>	
	R <sup>2</sup>	S.V. <sup>②</sup>	R <sup>2</sup>	S.V.	R <sup>2</sup>	S.V.	R <sup>2</sup>	S.V.
<u>first cutting</u>								
corn	0.59 <sup>#</sup>	EMo,pH	0.70	EMo,pH	0.77	EMo,pH	0.68	EMo,pH
soybeans	0.78	EMo	0.80	EMo	0.82	EMo	0.72	EMo,pH
alfalfa	0.59	EMo,pH	0.63	EMo,pH	0.75	EMo,pH	0.65	EMo,pH
<u>second cutting</u>								
corn	0.82	EMo	0.73	EMo	0.76	EMo,pH	0.67	EMo,pH
soybeans	0.57	EMo,pH	0.58	EMo,pH	0.66	EMo	0.61	EMo
alfalfa	0.62	EMo,pH	0.66	EMo,pH	0.84	EMo,pH	0.77	EMo,pH

<sup>②</sup>Significant variables (S.V.) in the prediction equation, EMo is the extractable Mo with the indicated extractant.

<sup>#</sup>All regression coefficients are significant at the 95% probability level.

The arithmetic means of regression coefficients for each extractant and Mo source across both cuttings of all crops are 0.66 and 0.77 for the AB-DTPA extractant and 0.68 and 0.68 for Tamm's reagent for inorganic and sludge Mo, respectively. These means indicate that, (i) the AB-DTPA extractant, individually or in combination with soil pH, generally performed better than Tamm's reagent with the sludge Mo and, (ii) the AB-DTPA extractant worked better with sludge Mo than with inorganic Mo amendments. Further testing of this extractant as a Mo availability index is warranted.

Soil pH was a significant variable in 17 out of 24 prediction equations indicating that soil pH should be considered when assessing the potential for Mo accumulation in crops. Soil pH is less effective in predicting Mo concentrations in soybean tissue as compared to corn or alfalfa tissues with soil pH being significant in only 3 out of 8 equations for soybeans.

Molybdenum application rates from the sludge and sodium molybdate overlap, presenting the opportunity to compare the availability of Mo to plants from the two sources. The data does not indicate that one source has a much greater availability than the other. However, given the influence that soil pH has on plant available Mo and the influence that sludge loading has on soil pH, a direct comparison is not possible because similar pH levels for both sources at one application rate is difficult to obtain.

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## APPENDIX

The following appendix tables contain either information on Mo analysis that did not need to be given in the results and discussion sections of the preceeding chapters or information on various elements obtained during routing multielement analysis of plants and soils. Explanations of the treatments or samplings for tables A-1 to A-15 or tables A-16 to A-32 can be found in chapters III and IV, respectively.



## APPENDIX TABLES

### Field Studies

Table A-1. Concentrations of selected elements in the sludge applied to the field plots in 1982.

Table A-1. Concentrations of selected elements in the sludge applied to the field plots in 1982.

Sample <sup>1</sup>	Zn	Cd	Cu	Mn	Ni	Pb	Cr
	----- mg kg <sup>-1</sup> -----						
spring 1982	951+42	10.0+0.5	564+37	830+35	47.3+4.7	402+16	775+64
fall 1982	906+73	9.8+0.7	550+36	825+130	47.4+3.4	385+33	785+110
all samples	929+60	9.9+0.6	557+36	828+81	47.4+4.1	394+24	780+86

Sample	Al	Fe	P	Mg	Ca	K
	----- % -----					
spring 1982	2.0+0.3	2.2+0.2	1.3+0.1	1.3+0.2	7.1+0.2	1.4+0.1
fall 1982	2.0+0.1	2.0+0.2	1.3+0.2	1.4+0.03	7.0+0.4	1.4+0.1
all samples	2.0+0.2	2.1+0.2	1.3+0.2	1.4+0.2	7.1+0.3	1.4+0.1

<sup>1</sup>Means from the spring and fall samplings reflect four sludge samples; "all" values are mean of all eight samples.

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Table A-2. Concentrations of selected elements in total soil digests from the fall 1984 sampling of corn plots in Experiment 1.

Sludge Treatment	Zn	Cd	B	P	Cu
- Mg ha <sup>-1</sup> -	----- mg kg <sup>-1</sup> -----				
0	38.3±2.6	1.3±0.3	5.5±1.0	700±100	7.9±0.9
42	55.0±7.5	1.5±0.4	5.0±0.9	1200±500	15.9±2.2
94	70.3±4.5	1.8±0.1	5.1±0.4	1500±300	25.2±1.8

Sludge Treatment	Mn	Ni	Pb	Cr
- Mg ha <sup>-1</sup> -	----- mg kg <sup>-1</sup> -----			
0	318±50	12.1±0.7	24.1±1.9	27.1±3.0
42	337±74	12.0±1.3	27.8±3.2	36.6±3.2
94	340±47	13.0±0.5	36.2±1.9	53.8±7.9

Table A-3. Levels of extractable Mo from soils collected in fall 1982 and 1983.

Sludge Treatment	AB-DTPA		NaOAc		Tamm's		DAHP	
	f82	f83	f82	f83	f82	f83	f82	f83
- Mg ha <sup>-1</sup> -	----- mg kg <sup>-1</sup> -----							
	<u>Experiment 1</u>							
	<u>Corn</u>							
0	0.2	0.2	0.1	0.2	1.6	0.4	0.1	0.1
42	5.7	8.6	0.5	0.9	10.5	17.2	2.7	5.4
94	10.6	17.0	0.6	1.6	19.0	36.6	5.5	11.0
LSD <sup>1</sup>	2.4	4.0	0.6	0.4	2.7	3.3	1.0	1.5
	<u>Soybeans</u>							
0	0.2	0.3	0.1	0.2	1.8	1.2	0.1	0.1
42	5.9	7.8	0.6	1.0	12.6	18.2	3.6	5.2
94	8.4	18.5	0.8	1.0	18.4	38.9	4.7	11.6
LSD	2.0	2.2	0.17	0.17	4.2	3.6	1.5	1.5
	<u>Experiment 2</u>							
	<u>corn</u>							
194	22.4	35.0	2.2	3.5	45.6	81.9	12.5	19.6
200 <sup>2</sup>	0.8	29.6	0.2	3.0	2.6	57.6	0.4	17.9
LSD	5.0	n.s.	0.4	n.s.	10.4	n.s.	1.6	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.<sup>2</sup>Sludge application made after f82 sampling.

**Table A-4. Concentrations of selected elements in soybeans grown in 1982.**

[illegible]





Table A-4 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
- Mg ha <sup>-1</sup> -		mg kg <sup>-1</sup>		%				
Whole Plant	0	2.9+0.2	0.8+0.1	34.8+2.3	0.36+0.02	0.54+0.03	3.0+0.1	1.7+0.2
	42	3.0+0.2	0.9+0.06	33.8+1.8	0.35+0.01	0.60+0.06	2.9+0.1	2.0+0.1
	94	3.1+0.03	1.0+0.03	32.4+0.4	0.35+0.01	0.67+0.02	2.9+0.2	2.2+0.1
	LSD	n.s.	0.1	1.9	n.s.	0.17	n.s.	0.2
Diagnostic Tissue	0	2.3+0.4	0.5+0.02	36.4+1.3	0.40+0.02	0.40+0.00	2.9+0.1	1.1+0.05
	42	2.3+0.05	0.6+0.01	37.4+2.4	0.40+0.01	0.39+0.05	2.7+0.2	1.1+0.05
	94	2.3+0.06	0.6+0.03	36.5+1.4	0.40+0.01	0.41+0.05	3.0+0.2	1.2+0.05
	LSD	n.s.	0.03	n.s.	n.s.	n.s.	0.2	0.1
Grain	0	1.3+1.3	0.1+0.01	24.0+0.9	0.54+0.02	0.26+0.01	1.8+0.04	0.24+0.03
	42	0.8+0.1	0.2+0.02	24.9+0.3	0.54+0.02	0.27+0.01	1.8+0.03	0.25+0.02
	94	0.6+0.03	0.1+0.01	25.3+4.2	0.54+0.02	0.27+0.00	1.8+0.04	0.25+0.01
	LSD	n.s.	0.1	1.1	n.s.	n.s.	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.

**Table A-5. Concentrations of selected elements in soybeans grown in 1983.**

[illegible]

Table A-5 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
- Mg ha <sup>-1</sup> -		----- mg kg <sup>-1</sup> -----		----- % -----		-----		
Whole Plant	0	3.1+0.3	1.1+0.1	41.0+2.7	0.36+0.01	0.61+0.07	2.7+0.2	1.9+0.3
	42	3.7+0.3	1.4+0.3	39.5+1.9	0.37+0.02	0.63+0.02	2.6+0.1	2.0+0.1
	94	3.8+0.2	1.4+0.1	39.6+1.5	0.36+0.01	0.60+0.3	2.7+0.3	1.8+0.5
	LSD	0.4	0.2	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	0	2.8+1.3	0.5+0.07	44.4+0.8	0.41+0.02	0.38+0.03	2.7+0.1	0.91+0.03
	42	2.5+0.1	0.5+0.07	42.2+5.9	0.45+0.05	0.40+0.03	2.7+0.1	0.94+0.03
	94	3.0+1.5	0.5+0.08	44.1+3.1	0.42+0.03	0.41+0.02	2.2+0.1	1.0+0.09
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	0.2	n.s.
Grain	0	0.7+0.05	0.2+0.05	28.3+1.0	0.53+0.03	0.25+0.01	1.9+0.05	0.3+0.01
	42	0.9+0.4	0.4+0.09	28.7+1.16	0.53+0.03	0.25+0.01	1.9+0.05	0.3+0.01
	94	0.8+0.05	0.8+0.05	29.0+1.5	0.52+0.01	0.25+0.01	1.9+0.05	0.3+0.01
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.

Table A-6. Concentrations of selected elements in soybeans grown in 1984.

Sample	Sludge Treatment	Al	Zn	Cd	Fe	Cu	Mn	Ni
		----- mg kg <sup>-1</sup> -----						
Whole Plant		- Mg ha <sup>-1</sup> -						
	0	238+21	n.a. <sup>1</sup>	0.9+0.1	323+44	12.1+1.3	214+58	3.9+0.5
	42	214+29	n.a.	0.7+0.1	308+27	13.9+1.5	111+17	2.6+0.4
	94	187+35	n.a.	0.7+0.2	298+39	13.4+1.1	103+9	2.7+0.1
	LSD <sup>2</sup>	n.s.		n.s.	n.s.	n.s.	49.6	0.6
Diagnostic Tissue	0	85.8+14.8	40.8+5.5	0.8+0.1	163+17	15.1+1.1	208+89	6.2+2.1
	42	82.7+9.2	46.0+9.8	0.7+0.2	167+15	20.3+7.0	83.6+21	3.3+1.2
	94	66.8+12.9	41.3+0.9	0.5+0.04	148+15	17.8+1.4	64.5+4.6	2.8+0.3
	LSD	n.s.	n.s.	0.1	n.s.	n.s.	79.0	2.2
Grain	0	4.7+1.2	42.2+3.5	0.4+0.1	63.8+2.7	12.6+0.7	37.2+6.2	5.7+1.7
	42	4.8+0.4	44.0+4.3	0.4+0.1	68.3+3.5	15.0+1.1	27.8+3.6	3.7+1.1
	94	5.1+0.4	44.0+3.2	0.3+0.1	65.0+3.4	13.5+0.4	23.7+0.7	2.8+0.2
	LSD	n.s.	n.s.	n.s.	3.5	1.1	4.1	1.1

<sup>1</sup>Not available.

Table A-6 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
- Mg ha <sup>-1</sup> -		----- mg kg <sup>-1</sup> -----		----- % -----				
Whole Plant	0	3.2+0.1	1.1+0.1	47.7+4.8	0.35+0.02	0.75+0.06	1.8+0.2	1.6+0.04
	42	3.8+0.2	1.3+0.2	38.9+3.9	0.33+0.01	0.76+0.07	1.7+0.4	1.9+0.1
	94	4.2+0.5	1.3+0.3	38.9+2.0	0.33+0.01	0.79+0.04	1.7+0.2	2.0+0.1
	LSD	0.6	n.s.	4.7	n.s.	n.s.	n.s.	0.2
Diagnostic Tissue	0	2.5+0.1	0.3+0.06	45.7+2.7	0.34+0.02	0.46+0.02	2.3+0.2	0.84+0.06
	42	3.2+0.5	0.4+0.05	43.4+2.4	0.37+0.03	0.50+0.02	2.1+0.2	1.1+0.1
	94	2.9+0.2	0.4+0.07	39.5+1.0	0.38+0.02	0.46+0.02	2.1+0.2	1.0+0.09
	LSD	0.5	n.s.	1.8	n.s.	n.s.	n.s.	0.3
Grain	0	0.6+0.3	0.1+0.06	24.1+0.4	0.48+0.01	0.19+0.01	1.6+0.02	0.13+0.01
	42	0.6+0.2	0.3+0.2	24.3+1.1	0.51+0.04	0.18+0.01	1.5+0.1	0.13+0.02
	94	0.5+0.2	0.1+0.03	24.4+0.8	0.47+0.02	0.18+0.01	1.5+0.05	0.13+0.01
	LSD	n.s.	n.s.	n.s.	0.02	n.s.	n.s.	n.s.

<sup>2</sup>Least significant difference at the 95% probability level.

**Table A-7. Concentrations of selected elements in corn grown on Experiment 1 in 1982.**

[illegible]

Table A-7 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> -----			----- % -----			
Whole Plant	0	2.2+0.3	1.3+0.3	10.8+3.0	0.52+0.04	0.16+0.01	3.0+0.1	0.29+0.01
	42	2.3+0.3	1.0+0.1	8.6+0.4	0.49+0.03	0.17+0.02	2.8+0.2	0.35+0.03
	94	2.4+0.3	1.3+0.06	9.2+0.6	0.49+0.04	0.19+0.03	2.8+0.2	0.37+0.1
	LSD	n.s.	0.2	0.7	n.s.	n.s.	n.s.	0.04
Diagnostic Tissue	0	2.5+0.2	0.8+0.4	7.6+1.8	0.42+0.01	0.11+0.01	1.9+0.05	0.39+0.02
	42	2.9+0.3	0.7+0.2	7.8+1.3	0.32+0.04	0.14+0.01	1.9+0.05	0.50+0.03
	94	2.5+0.1	0.6+0.04	7.8+0.4	0.42+0.01	0.16+0.03	1.8+0.08	0.48+0.02
	LSD	n.s.	n.s.	n.s.	n.s.	0.02	0.1	0.05
Grain	0	0.4+0.1	0.09+0.04	1.9+0.2	0.26+0.02	0.09+0.01	0.44+0.04	0.08+0.01
	42	0.4+0.1	0.06+0.03	2.0+0.1	0.27+0.02	0.09+0.01	0.46+0.05	0.07+0.03
	94	0.3+0.1	0.06+0.03	2.0+0.1	0.25+0.01	0.09+0.01	0.42+0.01	0.07+0.01
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.

Table A-8. Concentrations of selected elements in corn grown on Experiment 2 in 1982<sup>1</sup>.

Sample	Sample Treatment	Al	Zn	Cd	Fe	Cu	Mn	Ni
		----- mg kg <sup>-1</sup> -----						
		- Mg ha <sup>-1</sup> -						
Whole Plant	194	64.6+12	66.0+6.9	0.5+0.1	232+17	16.2+2.9	101+18	1.2+0.2
	200	84.6+11	44.5+2.6	0.5+0.2	284+33	15.9+0.6	118+15	1.3+1.3
	LSD <sup>2</sup>	17.1	8.0	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	194	11.6+3.0	54.6+5.4	0.3+0.06	144+20	17.0+1.6	150+30.3	0.5+0.1
	200	12.1+0.4	44.9+4.9	0.3+0.04	153+9	18.0+0.9	188+37.6	0.5+0.09
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Grain	194	4.6+0.4	24.5+1.6	0.5+0.04	28.4+2.2	4.2+1.4	6.5+0.7	0.5+0.1
	200	5.4+0.4	20.6+0.4	0.5+0.1	29.1+3.4	3.7+0.4	7.3+0.6	0.3+0.02
	LSD	n.s.	0.2	n.s.	n.s.	n.s.	n.s.	0.16

<sup>1</sup>The 200 Mg ha<sup>-1</sup> sludge application was made after this corn crop.





Table A-8 (continued)

Sample	Sample Treatment	Pb	Cr	B	P	Mg	K	Ca
	-- Mg ha <sup>-1</sup> --	----- mg kg <sup>-1</sup> -----	----- % -----					
Whole Plant	194	2.2+0.1	1.2+0.3	8.6+0.3	0.44+0.1	0.15+0.01	2.7+0.2	0.33+0.01
	200	2.8+1.1	1.2+0.4	9.3+0.8	0.47+0.03	0.15+0.02	2.8+0.03	0.30+0.01
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	194	2.9+0.2	1.3+1.5	7.2+1.2	0.43+0.03	0.15+0.03	1.9+0.2	0.54+0.08
	200	2.8+0.3	0.7+0.1	7.3+1.0	0.44+0.03	0.13+0.02	2.0+0.1	0.47+0.02
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Grain	194	0.3+0.06	0.1+0.02	2.2+0.1	0.28+0.01	0.09+0.01	0.42+0.01	0.06+0.0
	200	0.4+0.09	0.1+0.02	2.0+0.02	0.26+0.01	0.08+0.01	0.41+0.02	0.06+0.0
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

<sup>2</sup>Least significant difference at the 95% probability level.



Table A-9. Concentrations of selected elements in corn grown on Experiment 1 in 1983.

Sample	Sludge Treatment	Al	Zn	Cd	Fe	Cu	Mn	Ni
		----- mg kg <sup>-1</sup> -----						
		- Mg ha <sup>-1</sup> -						
Whole Plant	0	201+40	73.1+4.9	0.4+0.04	304+53	20.4+2.9	207+7	1.8+0.3
	42	165+37	80.6+9.7	0.4+0.04	275+44	20.3+1.0	113+13	1.2+0.2
	94	175+43	81.5+10	0.4+0.06	286+45	20.8+2.7	123+39	1.8+0.7
	LSD <sup>1</sup>	n.s.	n.s.	n.s.	n.s.	n.s.	47	n.s.
Diagnostic Tissue	0	21.9+5.0	27.7+3.3	0.1+0.02	229+19	13.1+1.8	135+25	n.a. <sup>2</sup>
	42	24.7+2.7	37.8+7.5	0.2+0.02	296+36	12.1+2.4	111+18	n.a.
	94	23.9+1.7	41.9+4.5	0.2+0.02	304+14	13.9+1.3	112+22	n.a.
	LSD	n.s.	8.7	n.s.	n.s.	n.s.	n.s.	
Grain	0	8.7+6.5	26.8+3.9	0.4+0.1	89.5+16	7.8+7.4	10.1+0.6	0.4+0.2
	42	5.3+0.7	27.3+1.9	0.4+0.1	72.8+8.2	3.7+0.4	7.7+0.7	0.4+0.07
	94	5.1+0.4	27.2+3.0	0.5+0.1	71.7+8.9	3.4+0.8	7.9+0.7	0.5+0.2
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	0.6	n.s.

Table A-9 (continued)

Sludge					
Cr	B	P	Mg	K	Ca

Table A-9 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
		mg kg <sup>-1</sup>			%			
Whole Plant	0	2.0+0.2	1.3+0.2	7.8+0.2	0.67+0.1	0.35+0.1	5.1+0.2	0.51+0.1
	42	2.5+0.2	1.4+0.3	8.0+0.2	0.65+0.04	0.41+0.1	5.1+0.3	0.61+0.1
	94	2.6+0.4	1.4+0.2	8.6+0.7	0.61+0.5	0.43+0.1	5.2+0.3	0.70+0.1
	LSD	0.4	n.s.	n.s.	n.s.	n.s.	n.s.	0.04
Diagnostic Tissue	0	1.5+0.2	0.1+0.01	8.4+2.3	0.39+0.04	0.21+0.04	2.4+0.04	0.40+0.03
	42	1.9+0.04	0.2+0.04	7.9+0.4	0.37+0.2	0.30+0.03	2.4+0.5	0.58+0.04
	94	2.1+0.03	0.2+0.02	9.0+0.2	0.35+0.02	0.38+0.1	2.3+0.04	0.73+0.1
	LSD	0.17	0.05	n.s.	n.s.	0.06	n.s.	0.04
Grain	0	0.2+0.03	n.d. <sup>2</sup>	1.9+0.2	0.31+0.01	0.14+0.01	0.46+0.03	0.06+0.01
	42	0.3+0.03	n.d.	2.2+0.2	0.32+0.02	0.15+0.01	0.48+0.02	0.06+0.01
	94	0.5+0.5	n.d.	2.2+0.1	0.33+0.02	0.16+0.01	0.51+0.1	0.06+0.01
	LSD	n.s.		0.2	n.s.	0.01	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.<sup>2</sup>Not available.<sup>3</sup>Not detectable, <0.1 mg kg<sup>-1</sup>.



Table A-10. Concentrations of selected elements in corn grown on Experiment 2 in 1983.

Sample	Sludge Treatment	Al	Zn	Cd	Fe	Cu	Mn	Ni
		----- mg kg <sup>-1</sup> -----						
		- Mg ha <sup>-1</sup> -						
Whole Plant	194	188+37	79.4+3.0	0.4+0.03	328+60	21.7+0.6	136+48	1.6+0.3
	200	245+34	78.5+7.0	0.4+0.03	375+50	22.2+2.2	106+12	2.3+1.2
	LSD <sup>1</sup>	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	194	20.7+0.5	48.2+3.1	0.2+0.03	298+6	15.6+0.9	160+18	0.4+0.04
	200	22.4+0.7	49.1+7.6	0.2+0.02	307+29	16.1+2.7	104+22	0.4+0.03
	LSD	1.7	n.s.	n.s.	n.s.	n.s.	40.5	n.s.
Grain	194	6.5+1.9	34.2+4.9	0.6+0.1	93.9+13	9.4+3.6	8.3+0.5	0.5+0.1
	200	5.2+0.3	30.1+6.5	0.6+0.07	71.2+10	3.5+0.4	7.2+0.4	0.5+0.04
	LSD	n.s.	n.s.	n.s.	9.3	n.s.	1.1	n.s.



Table A-10 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
- Mg ha <sup>-1</sup> -		mg kg <sup>-1</sup>		%				
Whole Plant	194	2.6+0.1	1.3+0.2	8.8+0.4	0.57+0.03	0.45+0.09	4.6+0.7	0.76+0.07
	200	3.4+0.3	2.4+0.4	8.5+0.7	0.60+0.04	0.43+0.07	4.8+0.2	0.75+0.07
	LSD <sup>1</sup>	0.6	0.8	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	194	2.0+0.1	0.2+0.03	13.9+2.8	0.38+0.02	0.70+0.1	2.1+0.1	0.70+0.1
	200	2.1+0.1	0.3+0.1	10.3+1.1	0.37+0.02	0.75+0.03	2.1+0.1	0.75+0.04
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Grain	194	0.3+0.1	n.d. <sup>2</sup>	2.1+0.2	0.32+0.02	0.17+0.01	0.47+0.05	0.06+0.01
	200	0.3+0.04	n.d.	2.2+0.1	0.32+0.01	0.16+0.01	0.45+0.03	0.06+0.01
	LSD	n.s.		n.s.	n.s.	n.s.	n.s.	n.s.

<sup>1</sup>Least significant difference at the 95% probability level.<sup>2</sup>Not detectable, <0.1 mg kg<sup>-1</sup>.



Table A-11. Concentrations of selected elements in corn grown on Experiment 1 in 1984.

Sample	Sludge Treatment	Al	Zn	Cd	Fe	Cu	Mn	Ni
		----- mg kg <sup>-1</sup> -----						
		- Mg ha <sup>-1</sup> -						
Whole Plant	0	32.5+8.8	39.6+3.2	0.2+0.03	292+23	13.7+1.3	107+10	0.6+0.1
	42	35.5+9.4	46.4+0.6	0.2+0.02	293+38	15.1+1.0	54.0+8.7	0.6+0.1
	94	37.2+8.8	48.6+3.0	0.2+0.04	274+27	14.7+0.9	44.1+6.8	0.7+0.2
	LSD <sup>1</sup>	n.s.	4.0	n.s.	n.s.	n.s.	16.9	0.16
Diagnostic Tissue	0	32.2+4.6	37.5+2.5	0.3+0.1	295+8	15.1+1.2	184+37	1.1+0.3
	42	37.3+3.2	49.1+2.5	0.3+0.04	285+16	12.5+1.1	92.6+15	0.9+0.4
	94	40.0+2.3	51.9+3.5	0.3+0.04	294+9	13.3+1.1	82.6+14	0.7+0.1
	LSD	4.2	5.1	n.s.	n.s.	2.0	40.6	n.s.
Grain	0	7.2+4.6	21.9+2.1	0.5+0.2	32.4+4.9	7.2+5.3	9.0+1.3	0.5+0.3
	42	4.0+0.7	24.6+1.3	0.5+0.1	27.5+2.8	4.5+1.3	7.3+0.6	0.4+0.1
	94	6.4+4.5	25.4+3.8	0.6+0.2	31.6+8.5	7.9+7.1	6.8+0.6	0.5+0.2
	LSD	n.s.	n.s.	n.s.	n.s.	n.s.	1.6	n.s.

Table A-11 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
- Mg ha <sup>-1</sup> -		mg kg <sup>-1</sup> -----		----- %		-----		
Whole Plant	0	1.7+0.1	0.3+0.1	6.5+0.2	0.53+0.03	0.34+0.02	4.0+0.4	0.33+0.01
	42	2.0+0.12	0.4+0.1	6.5+0.4	0.52+0.03	0.35+0.03	3.7+0.2	0.44+0.04
	94	2.2+0.2	0.6+0.1	6.4+0.33	0.46+0.03	0.37+0.05	3.3+0.6	0.48+0.04
	LSD <sup>1</sup>	0.2	0.2	n.s.	0.04	n.s.	n.s.	0.06
Diagnostic Tissue	0	4.1+1.3	0.3+0.1	12.1+0.7	0.36+0.01	0.28+0.02	1.8+0.1	0.48+0.04
	42	4.9+1.5	0.4+0.1	10.8+0.7	0.38+0.03	0.33+0.03	1.9+0.02	0.61+0.1
	94	4.8+1.3	0.5+0.04	11.2+0.8	0.36+0.03	0.35+0.03	1.8+0.1	0.67+0.1
	LSD	n.s.	0.1	n.s.	n.s.	n.s.	n.s.	n.s.
Grain	0	0.1+0.1	n.d. <sup>2</sup>	1.6+0.2	0.27+0.01	0.09+0.03	0.34+0.12	n.a. <sup>3</sup>
	42	0.1+0.02	n.d.	1.8+0.2	0.29+0.01	0.09+0.03	0.39+0.09	n.a.
	94	0.2+0.1	n.d.	1.7+0.1	0.28+0.01	0.11+0.01	0.47+0.04	n.a.
	LSD	n.s.		n.s.	n.s.	n.s.	n.s.	

<sup>1</sup>Least significant difference at the 95% probability level; <sup>2</sup>not detectable, <0.1 mg kg<sup>-1</sup>; <sup>3</sup>not available.



Table A-12 (continued)

Sample	Sludge Treatment	Pb	Cr	B	P	Mg	K	Ca
	- Mg ha <sup>-1</sup> -	----- mg kg <sup>-1</sup> -----	----- % -----					
Whole Plant	194	2.5±0.3	0.6±0.1	7.2±0.6	0.46±0.02	0.34±0.02	3.2±0.2	0.51±0.03
	200	2.8±0.1	0.8±0.1	7.1±0.3	0.44±0.03	0.36±0.03	3.1±0.5	0.60±0.03
	LSD	n.s.	0.2	n.s.	n.s.	n.s.	n.s.	n.s.
Diagnostic Tissue	194	4.1±1.2	0.7±1.0	13.5±1.2	0.37±0.01	0.37±0.02	1.7±0.1	0.77±0.02
	200	3.6±1.0	0.7±0.1	13.3±0.6	0.38±0.01	0.39±0.03	1.8±0.2	0.76±0.1
	LSD	0.5	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Grain	194	0.4±0.2	n.d. <sup>2</sup>	1.7±0.2	0.3±0.01	0.09±0.02	0.39±0.04	n.a. <sup>3</sup>
	200	0.4±0.4	n.d.	1.8±0.1	0.3±0.02	0.1±0.02	0.40±0.03	n.a.
	LSD	n.s.		n.s.	n.s.	n.s.	n.s.	

<sup>1</sup>Least significant difference at the 95% probability level; <sup>2</sup>not detectable, <0.1 mg kg<sup>-1</sup>; <sup>3</sup>not available.

Table A-13. Metals extracted with AB-DTPA from various soil samplings of corn plots in Experiment 1.

Sludge

Table A-13. Metals extracted with AB-DTPA from various soil samplings of corn plots in Experiment 1.

Sample	Sludge Treatment	Zn	Cd	Fe	P	Cu	Mn	Ni	Pb	Cr
		- Mg ha <sup>-1</sup> ----- mg kg <sup>-1</sup> -----								
fall (1982)	0	1.3±0.2	0.1±0.01	62.0±8.5	35.3±3.8	0.9±0.1	38.7±8.8	0.3±0.1	2.1±1.1	0.2±0.02
	42	4.0±1.0	0.1±0.01	54.6±5.7	34.1±2.2	3.1±0.7	28.0±6.4	0.5±0.1	3.2±0.4	0.2±0.03
	94	5.9±1.2	0.1±0.02	46.9±4.9	31.9±2.9	5.4±1.8	27.2±6.6	1.8±0.4	3.4±0.2	0.1±0.01
spring (1983)	0	1.3±0.1	0.1±0.01	62.6±4.6	38.4±6.5	1.0±0.1	37.1±4.3	0.4±0.03	2.6±0.1	0.2±0.03
	42	5.5±0.7	0.1±0.01	58.0±3.3	40.1±5.1	4.7±0.6	27.9±2.0	0.6±0.03	3.6±0.2	0.2±0.03
	94	10.1±0.5	0.2±0.01	53.6±3.8	41.1±3.8	8.2±0.3	22.9±2.8	0.7±0.05	4.5±0.3	0.2±0.01
fall (1983)	0	1.3±0.1	0.1±0.01	64.1±6.6	35.6±6.1	1.0±0.1	23.0±6.6	0.6±0.2	2.7±0.1	0.2±0.03
	42	6.1±1.4	0.1±0.02	52.2±3.1	37.6±4.5	5.3±1.0	11.0±2.4	0.6±0.2	3.8±0.6	0.2±0.02
	94	11.6±3.9	0.2±0.05	45.9±5.3	38.5±3.9	4.4±2.8	8.8±1.7	0.7±0.2	4.8±1.0	0.2±0.02



Table A-14. Metals extracted with AB-DTPA from soil samplings of soybean plots in Experiment 1.

Sample	Sludge Treatment	Zn	Cd	Fe	P	Cu	Mn	Ni	Pb	Cr
		- Mg ha <sup>-1</sup> ----- mg kg <sup>-1</sup> -----								
fall (1982)	0	1.2±0.1	0.1±0.01	63.3±6.6	36.2±5.6	0.9±0.1	33.2±3.7	0.6±0.1	2.7±0.2	0.2±0.04
	42	4.7±0.9	0.1±0.01	62.2±3.8	38.7±5.2	3.3±0.5	26.4±3.6	1.8±0.3	3.6±0.3	0.2±0.03
	94	6.6±0.6	0.1±0.02	56.8±4.1	37.3±3.8	5.0±0.6	20.5±2.3	2.3±0.1	4.0±0.1	0.2±0.02
spring (1983)	0	1.4±0.2	0.1±0.01	65.2±10.7	38.0±6.8	1.1±0.1	41.7±9.7	0.4±0.04	2.6±0.2	0.2±0.03
	42	6.1±0.9	0.1±0.01	55.7±7.1	37.8±2.6	5.1±0.6	30.8±8.1	0.6±0.01	3.8±0.4	0.2±0.02
	94	13.3±2.6	0.2±0.03	53.6±6.2	41.1±5.4	11.0±2.0	25.0±5.2	0.7±0.1	5.0±0.5	0.2±0.01

Table A-15. Metals extracted with AB-DTPA from soil samplings of plots in Experiment 2.

Sample	Sludge Treatment	Zn	Cd	Fe	P	Cu	Mn	Ni	Pb	Cr
		- Mg ha <sup>-1</sup> - ----- mg kg <sup>-1</sup> -----								
fall (1982)	194	13.4+1.7	0.2+0.02	55.5+5.5	39.7+2.8	10.7+1.5	21.0+3.8	0.7+0.1	6.4+0.9	0.2+0.01
	200 <sup>1</sup>	1.3+0.3	0.1+0.02	49.0+2.6	25.6+2.3	1.1+0.3	27.0+1.1	2.3+0.3	2.7+0.2	0.1+0.01
spring (1983)	194	20.4+4.4	0.3+0.03	53.4+2.8	45.0+1.4	17.3+3.0	22.1+3.8	0.9+0.03	6.6+0.7	0.2+0.02
	200	23.0+6.3	0.3+0.03	62.2+3.7	47.0+6.8	15.7+4.3	33.6+3.7	0.9+0.2	6.6+1.2	0.2+0.02
fall (1983)	194	23.9+3.9	0.3+0.04	58.9+1.6	52.7+2.9	21.4+2.3	11.5+1.6	0.9+0.1	8.3+0.6	0.2+0.04
	200	18.8+6.0	0.2+0.04	62.7+5.5	43.9+7.7	14.5+4.6	15.4+1.6	0.8+0.1	5.9+0.5	0.2+0.03

<sup>1</sup>Sludge application made after fall, 1982 sampling.

## APPENDIX TABLES

### Greenhouse Studies

Table A-16. Extractable levels of Mo from soils sampled after the second cutting of soybeans.

pH level <sup>1</sup>	Mo additions (mg kg <sup>-1</sup> )						
	<u>inorganic</u>				<u>sludge</u>		
	0	30	60	120	20	44	94
	----- mg kg <sup>-1</sup> -----						
	<u>AB-DTPA</u>						
1	0.4c <sup>2</sup>	11.8a	24.5b	50.0b	7.0a	15.1a	25.8a
2	0.3b	10.0a	19.2a	31.3a	6.1b	15.3a	25.8a
3	0.2a	11.7a	22.6a	43.9b	8.0c	17.4b	28.2a
	<u>Tamm's reagent</u>						
1	1.1a	23.4b	46.8a	93.5a	15.7c	30.6a	68.2a
2	1.1a	21.0a	46.0a	92.1a	12.1a	30.0a	67.0a
3	1.0a	20.5a	43.0a	93.7a	13.2b	30.7a	67.7a

<sup>1</sup>See page 55, Table 1, for actual pH values.

<sup>2</sup>Means having the same letter within a column and extractant are not significantly different at the 95% probability level.

Table A-17. Extractable levels of Mo from soils sampled after the first cutting of alfalfa.

pH level <sup>1</sup>	Mo additions (mg kg <sup>-1</sup> )						
	<u>inorganic</u>				<u>sludge</u>		
	0	30	60	120	20	44	94
----- mg kg <sup>-1</sup> -----							
<u>AB-DTPA</u>							
1	0.3a <sup>2</sup>	18.7b	36.2b	53.9b	6.7a	16.8a	29.4a
2	0.4a	11.0a	24.1a	34.9a	7.6b	18.2a	29.0a
3	0.3a	14.3a	16.6a	42.1a	8.5c	17.7a	29.3a
<u>Tamm's reagent</u>							
1	1.1a	34.2b	66.2b	84.3a	16.2b	33.7bc	74.8b
2	1.1a	23.6a	59.3b	90.3a	14.4a	32.3ac	68.0a
3	1.1a	31.7b	38.8a	92.4a	13.9a	30.0a	68.7a

<sup>1</sup>See page 55, Table 1, for actual pH values.

<sup>2</sup>Means having the same letter within a column and extractant are not significantly different at the 95% probability level.

Table A-18. Extractable levels of Mo from soils sampled after the second cutting of alfalfa.

pH level <sup>1</sup>	Mo additions (mg kg <sup>-1</sup> )						
	<u>inorganic</u>				<u>sludge</u>		
	0	30	60	120	20	44	94
	----- mg kg <sup>-1</sup> -----						
	<u>AB-DTPA</u>						
1	0.3a <sup>1</sup>	7.3a	25.3bc	57.7b	5.9a	14.2a	27.1a
2	0.3a	8.9ac	16.0ac	29.2a	6.4a	16.4a	26.3a
3	0.3a	10.3bc	15.7a	28.0a	7.3a	16.2a	28.3a
	<u>Tamm's reagent</u>						
1	1.1a	15.7a	45.9a	85.4a	16.5b	31.8a	67.2a
2	1.2a	19.9ac	37.0a	85.3a	14.4a	31.8a	66.9a
3	1.1a	23.8bc	36.1a	74.8a	13.9a	29.6a	68.8a

<sup>1</sup>See page 55, Table 1, for actual pH values.

<sup>2</sup>Means having the same letter within a column and extractant are not significantly different at the 95% probability level.

Table A-19. Dry matter production for corn, soybeans, and alfalfa grown in soils amended with sodium molybdate.

pH level	Mo additions (mg kg <sup>-1</sup> )					g/pot				
	0	30	60	120	0	30	60	120		
-----										
<u>first cutting</u>					<u>second cutting</u>					
-----										
<u>corn</u>										
1	3.8 <sub>±</sub> 0.5	4.0 <sub>±</sub> 0.3	4.0 <sub>±</sub> 0.3	4.0 <sub>±</sub> 0.4	6.1 <sub>±</sub> 0.6	6.0 <sub>±</sub> 0.7	5.8 <sub>±</sub> 0.3	4.7 <sub>±</sub> 0.2		
2	4.1 <sub>±</sub> 0.5	4.2 <sub>±</sub> 0.3	3.6 <sub>±</sub> 0.3	3.5 <sub>±</sub> 0.2	4.9 <sub>±</sub> 0.3	5.3 <sub>±</sub> 0.2	5.2 <sub>±</sub> 0.7	4.2 <sub>±</sub> 0.6		
3	3.7 <sub>±</sub> 0.4	3.3 <sub>±</sub> 0.2	2.7 <sub>±</sub> 0.5	1.5 <sub>±</sub> 0.7	4.4 <sub>±</sub> 0.2	4.5 <sub>±</sub> 0.2	3.5 <sub>±</sub> 0.2	2.4 <sub>±</sub> 0.3		
<u>soybeans</u>										
1	3.1 <sub>±</sub> 0.1	3.0 <sub>±</sub> 0.3	2.9 <sub>±</sub> 0.3	2.7 <sub>±</sub> 0.4	5.0 <sub>±</sub> 0.3	4.7 <sub>±</sub> 0.3	4.4 <sub>±</sub> 0.2	4.0 <sub>±</sub> 0.1		
2	3.3 <sub>±</sub> 0.2	3.1 <sub>±</sub> 0.4	3.2 <sub>±</sub> 0.4	2.7 <sub>±</sub> 0.4	5.1 <sub>±</sub> 0.3	5.0 <sub>±</sub> 0.5	5.0 <sub>±</sub> 0.3	4.7 <sub>±</sub> 0.4		
3	3.5 <sub>±</sub> 0.1	2.8 <sub>±</sub> 0.4	2.5 <sub>±</sub> 0.1	2.0 <sub>±</sub> 0.3	4.4 <sub>±</sub> 0.7	4.7 <sub>±</sub> 0.7	4.9 <sub>±</sub> 0.2	4.5 <sub>±</sub> 0.4		
<u>alfalfa</u>										
1	6.5 <sub>±</sub> 0.5	6.1 <sub>±</sub> 0.7	6.0 <sub>±</sub> 0.7	6.3 <sub>±</sub> 0.7	9.6 <sub>±</sub> 0.6	9.8 <sub>±</sub> 0.8	8.9 <sub>±</sub> 0.7	8.1 <sub>±</sub> 0.5		
2	7.3 <sub>±</sub> 0.4	7.2 <sub>±</sub> 0.6	7.1 <sub>±</sub> 0.6	7.0 <sub>±</sub> 0.8	10.4 <sub>±</sub> 0.5	9.6 <sub>±</sub> 1.3	9.1 <sub>±</sub> 1.2	6.7 <sub>±</sub> 0.2		
3	6.5 <sub>±</sub> 0.6	6.1 <sub>±</sub> 1.1	5.3 <sub>±</sub> 1.5	3.6 <sub>±</sub> 1.5	9.4 <sub>±</sub> 0.4	8.5 <sub>±</sub> 0.2	7.8 <sub>±</sub> 1.2	6.3 <sub>±</sub> 0.7		

Table A-20. Dry matter production for corn, soybeans, and alfalfa grown in sludge amended soils.

pH level	Mo additions (mg kg <sup>-1</sup> )				g/pot		
	20	44	94	20		44	94
-----							
	<u>first cutting</u>		<u>corn</u>		<u>second cutting</u>		
1	5.4+0.5	6.1+0.7	6.5+0.5	6.3+0.4	6.0+0.4	6.2+0.4	
2	5.2+0.4	4.2+0.7	4.9+0.5	5.8+0.5	4.8+0.3	5.4+0.5	
3	3.4+0.4	2.4+0.1	2.7+0.1	4.2+0.2	3.3+0.5	4.2+0.6	
-----							
			<u>soybeans</u>				
1	2.9+0.3	3.2+0.3	3.5+0.3	5.2+0.2	4.9+0.3	5.1+0.2	
2	3.5+0.1	3.1+0.2	3.5+0.1	5.8+0.1	5.0+0.2	5.2+0.3	
3	2.8+0.2	2.4+0.3	2.6+0.5	5.5+0.7	5.0+0.2	5.6+0.5	
-----							
			<u>alfalfa</u>				
1	6.8+0.9	7.1+1.0	8.5+0.8	9.7+1.0	10.5+0.9	11.5+0.8	
2	7.5+0.5	8.1+0.6	8.0+0.8	11.0+1.4	11.2+0.2	11.4+1.3	
3	6.2+0.7	6.7+0.7	7.7+1.5	10.7+0.3	10.3+0.8	11.8+0.9	



**Table A-21. Concentrations of selected elements in the first cutting of alfalfa grown in soils amended with sodium molybdate.**

[illegible]

Table A-21 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> ----- % -----					
0	1	4.5+0.5	1.4+1.7	0.21+0.01	0.2+0.1	2.7+0.1	1.7+0.1
	2	4.8+0.7	0.6+0.1	0.23+0.03	0.2+0.2	2.8+0.2	1.8+0.1
	3	4.9+0.8	0.7+0.1	0.21+0.04	0.2+0.03	2.7+0.2	2.2+0.4
30	1	5.1+1.3	0.6+0.1	0.20+0.01	0.2+0.02	2.7+0.1	1.7+0.1
	2	4.6+0.4	0.6+0.1	0.22+0.02	0.2+0.02	2.9+0.2	1.8+0.2
	3	4.7+0.7	0.7+0.1	0.23+0.02	0.2+0.01	3.0+0.1	2.2+0.2
60	1	4.3+0.5	0.5+0.1	0.21+0.02	0.2+0.02	2.7+0.2	1.6+0.2
	2	4.0+0.3	0.5+0.1	0.21+0.02	0.2+0.01	2.6+0.2	1.6+0.1
	3	5.9+1.3	0.9+0.1	0.20+0.02	0.2+0.03	2.7+0.1	2.3+0.3
120	1	4.7+0.9	0.6+0.1	0.19+0.02	0.2+0.02	2.6+0.2	1.6+0.1
	2	4.2+0.5	0.6+0.1	0.19+0.02	0.2+0.03	2.9+0.3	1.6+0.1
	3	5.3+0.2	0.9+0.1	0.14+0.03	0.2+0.1	2.4+0.6	2.1+0.5

Table A-22. Concentrations of selected elements in the first cutting of alfalfa grown in sludge amended soils.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
					----- mg kg <sup>-1</sup> -----			
20	1	24.5+5.7	0.9+0.1	86.7+10	71.0+8.0	15.2+1.6	165+32	1.7+0.4
	2	28.3+6.9	0.5+0.1	87.8+11	43.6+3.7	19.1+4.0	85.8+7.8	0.8+0.1
	3	27.2+6.1	0.4+0.1	81.2+12	36.7+2.8	17.2+1.8	80.5+2.2	0.9+0.2
44	1	27.3+7.6	0.6+0.1	83.7+12	65.1+15	17.2+2.5	87.2+5.2	1.5+0.4
	2	28.6+6.0	0.5+0.1	77.3+11	44.4+4.1	17.9+2.9	64.4+2.3	0.9+0.1
	3	28.6+6.0	0.4+0.03	83.3+10	36.7+3.8	18.9+1.3	66.9+7.0	1.1+0.5
94	1	29.5+13	0.7+0.1	79.5+15	58.3+7.7	17.6+2.6	98.5+8.8	0.6+0.3
	2	28.3+7.3	0.6+0.2	82.7+14	52.9+9.5	18.2+3.3	94.3+8.8	0.7+0.2
	3	27.4+7.1	0.6+0.1	75.7+14	43.8+8.7	17.9+1.8	71.3+19	0.6+0.1

Table A-22 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> ----- % -----					
20	1	4.2+0.5	0.5+0.1	0.23+0.02	0.3+0.03	2.8+0.3	1.6+0.1
	2	5.0+0.8	0.7+0.1	0.26+0.01	0.2+0.01	2.8+0.2	1.7+0.1
	3	5.4+1.3	0.7+0.3	0.19+0.03	0.2+0.01	2.8+0.1	2.2+0.2
44	1	4.4+1.0	0.5+0.1	0.24+0.02	0.3+0.03	3.0+0.2	1.6+0.2
	2	4.2+0.7	0.6+0.1	0.23+0.02	0.2+0.01	2.8+0.1	1.7+0.1
	3	5.3+0.7	0.7+0.1	0.20+0.02	0.2+0.02	2.9+0.2	1.8+0.3
94	1	3.9+0.6	0.6+0.3	0.20+0.02	0.2+0.04	2.9+0.3	1.6+0.2
	2	5.1+1.7	0.7+0.2	0.23+0.02	0.2+0.1	2.9+0.2	1.7+0.2
	3	4.4+0.6	0.6+0.1	0.20+0.02	0.2+0.02	2.8+0.2	1.8+0.06

Table A-23. Concentrations of selected elements in the second cutting of alfalfa grown in soils amended

Table A-23. Concentrations of selected elements in the second cutting of alfalfa grown in soils amended with sodium molybdate.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
	1	39.5+7.8	0.7+0.1	95.5+16	61.7+6.1	14.7+1.1	85.0+6.7	1.6+0.1
0	2	41.9+13	0.6+0.1	98.7+15	54.5+2.3	16.2+2.7	74.6+6.2	1.4+0.5
	3	42.7+14	0.5+0.1	94.5+19	52.2+5.1	13.6+1.4	84.6+7.9	1.3+0.3
	1	41.3+14	0.8+0.01	96.7+21	57.3+6.4	14.0+1.0	95.4+17	1.6+0.2
30	2	44.7+12	0.6+0.1	92.8+11	48.3+6.8	14.6+0.3	85.0+13	1.8+1.2
	3	52.9+22	0.5+0.01	79.3+12	41.8+7.3	14.6+1.4	79.3+13	1.2+0.1
	1	39.6+4.5	0.7+0.1	94.9+12	56.7+1.9	14.8+2.6	94.1+6.1	2.0+0.9
60	2	41.3+6.9	0.6+0.04	89.0+18	47.7+8.1	13.6+1.1	83.7+10	1.2+0.2
	3	44.4+9.3	0.5+0.1	84.9+15	38.1+4.4	14.8+1.2	83.3+3.8	1.3+0.3
	1	42.0+9.8	0.6+0.1	91.8+4	51.2+2.9	13.8+0.9	103+3	1.9+0.9
120	2	45.4+19	0.7+0.5	80.9+12	41.4+5.2	13.6+0.8	82.8+9.4	1.2+0.2
	3	46.4+9.9	0.5+0.1	94.0+15	37.9+6.3	13.8+2.3	84.5+13	1.7+1.3

Table A-23 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> -----	----- % -----				
0	1	5.8+0.3	0.8+0.03	0.18+0.02	0.18+0.02	2.8+0.3	1.5+0.2
	2	6.0+0.3	0.9+0.1	0.28+0.03	0.20+0.01	2.6+0.1	1.5+0.1
	3	6.9+1.1	1.1+0.2	0.28+0.02	0.18+0.01	2.5+0.1	2.0+0.3
30	1	5.7+0.6	0.8+0.1	0.23+0.01	0.18+0.02	2.8+0.3	1.4+0.2
	2	5.9+0.6	0.9+0.1	0.26+0.01	0.16+0.01	2.7+0.3	1.5+0.3
	3	6.6+0.6	1.1+0.1	0.27+0.02	0.16+0.02	2.5+0.1	1.8+0.3
60	1	6.2+1.7	0.9+0.1	0.24+0.02	0.18+0.02	2.8+0.2	1.4+0.1
	2	6.4+0.8	0.9+0.1	0.26+0.03	0.16+0.01	2.6+0.1	1.5+0.2
	3	7.9+1.1	1.3+0.2	0.28+0.03	0.17+0.01	2.6+0.1	1.8+0.3
120	1	6.3+2.1	0.8+0.3	0.23+0.02	0.18+0.01	2.8+0.2	1.2+0.1
	2	5.4+0.7	0.8+0.1	0.25+0.03	0.16+0.01	2.5+0.2	1.2+0.1
	3	6.1+0.6	1.1+0.4	0.25+0.03	0.19+0.03	2.7+0.1	1.7+0.2

Table A-24. Concentrations of selected elements in the second cutting of alfalfa grown in sludge amended soils.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni



Table A-24 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> ----- % -----					
20	1	5.9+1.0	0.9+0.2	0.29+0.01	0.22+0.03	2.9+0.2	1.2+0.1
	2	6.9+1.8	1.2+0.5	0.28+0.04	0.15+0.01	2.5+0.2	1.6+0.2
	3	6.9+0.9	1.1+0.2	0.26+0.02	0.16+0.02	2.6+0.2	1.7+0.2
44	1	9.1+3.4	1.5+0.5	0.31+0.01	0.21+0.02	2.8+0.2	1.3+0.1
	2	6.3+1.5	1.0+0.3	0.27+0.02	0.16+0.01	2.5+0.2	1.5+0.1
	3	6.0+0.2	1.0+0.02	0.27+0.02	0.16+0.02	2.6+0.2	1.8+0.2
94	1	6.4+0.5	1.0+0.1	0.31+0.01	0.22+0.02	2.8+0.03	1.4+0.2
	2	7.3+0.3	1.2+0.1	0.29+0.03	0.18+0.02	2.6+0.2	1.5+0.2
	3	7.2+1.1	1.2+0.1	0.28+0.02	0.16+0.02	2.6+0.2	1.7+0.2

Table A-25. Concentrations of selected elements in the first cutting of corn grown in soil amended with sodium molybdate.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
----- mg kg <sup>-1</sup> -----								
0	1	42.8+3.2	1.3+0.6	104+15	9.2+1.3	12.8+2.2	78.7+15	0.7+0.1
	2	34.5+3.2	1.1+0.3	155+2	5.7+1.0	11.7+0.2	86.9+1.9	1.1+0.7
	3	37.5+5.4	0.6+0.2	225+31	6.1+0.8	12.6+1.3	133+12	1.4+1.3
30	1	47.0+6.1	1.0+0.3	108+6	9.9+1.3	13.5+1.4	98.2+5.7	0.7+0.1
	2	35.8+2.1	1.4+0.6	137+6	6.4+0.4	12.0+1.1	95.0+3.7	0.6+0.1
	3	35.1+2.9	0.7+0.1	200+41	6.4+1.2	12.5+1.7	123+8	1.4+1.0
60	1	44.5+1.7	0.9+0.1	154+15	10.1+1.3	14.3+2.8	93.1+6.8	0.6+0.1
	2	35.0+1.8	1.0+0.3	130+19	6.1+0.8	10.5+0.9	88.9+4.7	0.9+0.6
	3	39.0+10.3	0.6+0.2	187+63	6.2+1.9	11.7+2.5	116+12	0.9+0.4
120	1	42.6+3.2	0.9+0.2	96.5+21	8.8+1.4	11.3+1.0	83.5+8.9	0.9+0.4
	2	35.3+2.6	1.2+0.4	187+20	6.3+0.9	13.0+1.4	84.6+5.7	1.8+1.4
	3	37.8+12	1.2+0.4	148+53	4.0+1.8	11.4+3.5	85.1+10	1.2+0.2

Table A-25 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> -----	-----	-----	-----	-----	-----
0	1	1.8+0.7	n.a. <sup>1</sup>	0.06+0.01	0.2+0.02	5.4+0.4	0.6+0.04
	2	1.9+1.1	n.a.	0.07+0.01	0.2+0.02	5.3+0.6	0.7+0.1
	3	1.9+0.6	n.a.	0.09+0.01	0.2+0.01	5.6+0.5	0.8+0.1
30	1	1.3+0.7	n.a.	0.06+0.01	0.2+0.03	5.3+0.1	0.6+0.04
	2	1.8+0.8	n.a.	0.07+0.01	0.2+0.01	5.3+0.1	0.7+0.04
	3	2.0+0.7	n.a.	0.08+0.01	0.2+0.01	5.4+0.2	0.9+0.03
60	1	1.7+0.9	n.a.	0.07+0.01	0.2+0.02	5.3+0.2	0.6+0.04
	2	2.1+0.7	n.a.	0.07+0.01	0.2+0.02	5.3+0.5	0.7+0.1
	3	2.1+0.9	n.a.	0.08+0.01	0.2+0.03	5.1+0.3	0.8+0.2
120	1	1.6+0.9	n.a.	0.06+0.01	0.2+0.02	5.1+0.4	0.6+0.1
	2	2.2+0.9	n.a.	0.07+0.01	0.2+0.02	5.1+0.3	0.7+0.03
	3	3.9+0.6	n.a.	0.07+0.01	0.2+0.02	5.0+0.2	1.0+0.1

<sup>1</sup> Not available.

Table A-26. Concentrations of selected elements in the first cutting of corn grown in sludge amended soils.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
----- mg kg <sup>-1</sup> -----								
20	1	55.3±7.9	1.2±0.5	110±11	11.4±1.7	17.3±0.9	83.8±2.6	1.2±0.6
	2	35.4±6.1	0.9±0.2	88.7±16	7.4±0.9	13.8±1.2	57.0±5.5	2.3±0.4
	3	41.1±6.7	0.9±0.1	126±42	9.1±1.2	17.1±1.9	109±10	2.6±0.7
44	1	51.2±5.7	1.0±0.3	103±8	10.9±2.1	19.3±2.1	45.3±3.6	2.4±0.7
	2	34.2±5.5	0.8±0.3	86.7±16	9.5±0.7	16.2±3.6	50.4±4.1	1.8±0.9
	3	35.5±12	0.8±0.3	99.4±36	8.9±1.9	15.7±4.1	72.9±13	2.7±0.8
94	1	44.7±8.3	1.1±0.3	109±28	11.2±1.0	18.2±1.3	53.0±1.8	1.3±0.9
	2	35.2±13	0.8±0.2	128±41	8.9±3.5	16.2±5.8	51.1±13	1.1±0.6
	3	34.4±2.0	1.1±0.4	89.7±9.8	9.3±1.5	15.1±0.9	71.0±17	1.7±0.9

Table A-26 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> -----				----- % -----	
	1	1.5 $\pm$ 0.6	n.d. <sup>1</sup>	0.09 $\pm$ 0.01	0.3 $\pm$ 0.02	5.2 $\pm$ 0.2	0.5 $\pm$ 0.1
20	2	2.3 $\pm$ 0.4	n.d.	0.08 $\pm$ 0.01	0.2 $\pm$ 0.02	5.2 $\pm$ 0.3	0.7 $\pm$ 0.1
	3	2.6 $\pm$ 0.7	n.d.	0.08 $\pm$ 0.01	0.2 $\pm$ 0.02	5.4 $\pm$ 0.3	1.0 $\pm$ 0.1
	1	2.1 $\pm$ 1.5	n.d.	0.08 $\pm$ 0.01	0.3 $\pm$ 0.03	5.0 $\pm$ 0.3	0.5 $\pm$ 0.1
44	2	1.8 $\pm$ 0.9	n.d.	0.08 $\pm$ 0.01	0.2 $\pm$ 0.01	5.1 $\pm$ 0.3	0.7 $\pm$ 0.1
	3	2.7 $\pm$ 0.8	n.d.	0.08 $\pm$ 0.01	0.2 $\pm$ 0.02	5.0 $\pm$ 0.9	0.9 $\pm$ 0.2
	1	2.4 $\pm$ 0.7	n.d.	0.08 $\pm$ 0.01	0.3 $\pm$ 0.1	4.8 $\pm$ 0.7	0.7 $\pm$ 0.04
94	2	2.6 $\pm$ 1.1	n.d.	0.08 $\pm$ 0.01	0.2 $\pm$ 0.1	4.9 $\pm$ 0.8	0.8 $\pm$ 0.1
	3	2.7 $\pm$ 0.4	n.d.	0.07 $\pm$ 0.01	0.2 $\pm$ 0.02	4.8 $\pm$ 0.7	0.9 $\pm$ 0.1

<sup>1</sup>Not detectable.

**Table A-27. Concentrations of selected elements in the second cutting of corn grown in soils amended with sodium molybdate.**

[illegible]

Table A-27 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	mg kg <sup>-1</sup>						%			
		Pb	Cr	P	Mg	K	Ca				
0	1	1.9+0.8	0.3+0.2	0.09+0.01	0.2+0.1	4.7+1.5	0.5+0.2				
	2	2.1+0.4	0.3+0.1	0.07+0.01	0.2+0.01	4.3+0.2	0.6+0.03				
	3	2.6+0.1	0.4+0.2	0.06+0.01	0.1+0.01	4.4+0.1	0.7+0.1				
30	1	2.0+0.8	0.2+0.1	0.09+0.01	0.2+0.1	5.0+2.3	0.5+0.03				
	2	2.3+0.2	0.3+0.1	0.07+0.01	0.2+0.01	4.2+0.3	0.6+0.04				
	3	2.9+0.5	0.5+0.1	0.06+0.01	0.1+0.01	4.3+0.3	0.7+0.1				
60	1	2.1+0.3	0.3+0.1	0.08+0.01	0.2+0.02	4.0+0.1	0.4+0.1				
	2	2.4+0.6	0.3+0.1	0.05+0.01	0.2+0.01	4.3+0.3	0.6+0.03				
	3	2.3+0.9	0.3+0.1	0.05+0.01	0.1+0.04	4.1+1.2	0.7+0.2				
120	1	2.0+0.1	0.3+0.1	0.06+0.01	0.2+0.01	4.4+0.2	0.5+0.03				
	2	2.6+0.7	0.5+0.2	0.05+0.01	0.2+0.01	4.7+0.2	0.7+0.1				
	3	2.3+0.6	0.5+0.3	0.05+0.01	0.1+0.04	3.1+1.2	0.6+0.2				

Table A-28. Concentrations of selected elements in the second cutting of corn grown in the sludge amended soils.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
	1	43.2+9.3	0.6+0.2	62.3+9.9	11.7+1.5	14.2+0.9	71.1+4.9	0.9+0.4
20	2	35.5+4.4	0.6+0.2	72.4+10	9.8+1.9	11.4+0.8	43.4+3.8	1.6+1.2
	3	35.0+4.3	0.5+0.1	88.5+17	8.9+1.4	13.2+1.1	40.0+4.4	1.8+1.1
	1	39.8+7.4	0.7+0.2	58.4+4.2	13.3+1.5	14.7+2.8	44.4+3.4	0.9+0.4
44	2	35.4+6.1	0.6+0.2	78.2+12	11.5+2.4	13.0+1.4	45.7+4.2	1.6+1.1
	3	33.3+3.5	0.4+0.4	98.6+11	10.8+1.4	12.6+0.5	40.3+3.4	1.6+0.7
	1	38.7+3.8	0.6+0.2	67.2+6.1	15.3+3.5	14.5+0.5	64.3+5.9	1.0+0.4
94	2	38.5+6.3	0.4+0.2	75.6+3.1	12.1+2.1	14.7+1.9	54.5+7.1	1.8+1.0
	3	35.6+5.4	0.6+0.3	77.5+6.8	10.5+1.6	14.6+2.3	47.0+5.1	1.3+0.7



Table A-28 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	----- mg kg <sup>-1</sup> ----- % -----					
		Pb	Cr	P	Mg	K	Ca
20	1	1.9±0.7	0.3±0.2	0.09±0.01	0.3±0.02	3.4±0.3	0.4±0.04
	2	2.8±0.8	0.4±0.2	0.08±0.01	0.2±0.01	3.4±0.2	0.6±0.1
	3	3.4±1.1	0.5±0.3	0.07±0.01	0.2±0.01	4.3±0.2	0.9±0.1
44	1	2.5±1.4	0.3±0.3	0.09±0.01	0.3±0.01	3.1±0.1	0.5±0.1
	2	3.6±1.6	0.4±0.2	0.07±0.01	0.3±0.01	4.1±0.3	0.7±0.03
	3	3.9±1.9	0.7±0.4	0.06±0.01	0.2±0.05	4.2±1.1	0.8±0.3
94	1	2.5±0.6	0.4±0.2	0.09±0.01	0.3±0.02	3.0±0.2	0.3±0.02
	2	2.8±0.9	0.4±0.2	0.07±0.01	0.3±0.03	3.7±0.1	0.3±0.03
	3	3.1±1.0	0.4±0.1	0.06±0.01	0.2±0.01	4.6±0.2	0.2±0.01



Table A-29. Concentrations of selected elements in the first cutting of soybeans grown in soils amended with sodium molybdate.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
----- mg kg <sup>-1</sup> -----								
0	1	19.5+8.6	1.1+0.4	198+91	28.4+1.1	21.2+2.7	115+6	2.3+0.1
	2	18.7+8.6	1.2+0.2	271+120	20.4+1.8	21.9+1.1	117+10	1.4+0.3
	3	20.8+5.3	1.3+0.2	216+42	16.5+1.8	21.6+6.4	110+5	1.5+0.7
30	1	17.8+3.5	1.4+0.4	200+65	25.5+3.7	23.2+3.5	131+15	3.0+1.1
	2	16.4+2.7	1.3+0.4	208+87	19.0+1.8	18.9+3.6	122+8	1.5+0.5
	3	24.4+4.9	1.2+0.2	209+83	15.1+2.1	21.9+3.1	109+13	1.4+0.2
60	1	20.8+1.8	1.2+0.3	242+120	25.2+2.3	21.6+2.4	130+6	2.7+0.3
	2	20.0+1.8	1.4+0.2	182+110	17.6+0.9	21.2+3.1	117+17	1.5+0.3
	3	25.2+5.8	1.0+0.2	216+42	14.2+2.1	20.1+1.7	100+7	1.4+0.3
120	1	36.7+5.4	1.3+0.4	187+28	23.4+1.3	22.0+4.4	134+7	3.0+1.0
	2	23.5+6.2	1.4+0.3	220+84	16.8+1.8	20.8+1.7	116+13	1.5+0.1
	3	26.8+7.9	1.2+0.2	236+180	14.1+0.9	22.0+2.7	72.5+13	1.6+0.2



Table A-29 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	mg kg <sup>-1</sup>					
		Pb	Cr	P	Mg	K	Ca
0	1	5.0+1.0	0.7+0.04	0.2+0.02	0.5+0.3	1.6+0.9	2.3+1.3
	2	5.0+0.8	0.8+0.2	0.1+0.01	0.3+0.1	1.2+0.3	1.7+0.4
	3	5.2+0.6	0.9+0.2	0.1+0.02	0.2+0.1	1.2+0.3	1.9+0.5
30	1	5.5+0.9	0.8+0.2	0.2+0.01	0.4+0.1	1.2+0.3	1.8+0.4
	2	6.1+0.8	0.7+0.1	0.1+0.01	0.3+0.1	1.1+0.2	1.7+0.4
	3	5.4+0.6	0.9+0.1	0.1+0.01	0.2+0.1	1.2+0.3	1.9+0.5
60	1	5.7+1.2	0.9+0.1	0.2+0.01	0.4+0.1	1.2+0.3	1.9+0.4
	2	4.9+0.7	0.8+0.2	0.1+0.01	0.3+0.04	1.1+0.2	1.6+0.3
	3	5.8+1.6	1.0+0.2	0.1+0.01	0.2+0.1	1.3+0.3	2.0+0.5
120	1	5.2+0.3	0.9+0.1	0.2+0.02	0.4+0.1	1.3+0.3	1.8+0.3
	2	5.4+0.8	0.9+0.2	0.1+0.01	0.3+0.01	1.2+0.3	1.7+0.4
	3	6.6+1.0	1.2+0.3	0.1+0.01	0.1+0.1	0.9+0.4	1.4+0.7

Table A-30. Concentrations of selected elements in the first cutting of soybeans grown in sludge amended soils.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
	1	20.4+1.4	1.7+0.5	260+100	33.0+3.5	22.3+2.7	126+7	2.4+0.3
20	2	21.4+5.8	1.3+0.1	225+84	23.4+2.1	27.4+6.4	88.1+8.3	1.7+0.3
	3	23.4+6.6	1.4+0.5	234+95	18.9+1.4	30.0+10	85.6+9.0	1.7+0.6
	1	23.2+7.8	1.4+0.4	119+88	31.2+2.5	25.8+2.8	92.6+10	2.3+0.1
44	2	20.9+1.0	1.4+0.2	200+98	24.2+1.6	27.3+2.2	77.8+3.9	1.6+0.3
	3	28.9+5.9	1.1+0.1	197+130	19.9+1.4	25.4+5.5	68.4+5.4	1.5+0.1
	1	25.6+8.7	1.5+0.6	240+140	23.4+8.9	27.0+10	102+21	2.3+0.6
94	2	22.2+3.6	1.7+0.7	217+84	23.2+5.8	24.4+9.2	110+18	2.3+0.4
	3	27.8+6.6	1.3+0.2	238+52	22.2+1.2	28.1+2.3	79.0+7.6	2.1+0.3

Table A-30 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	mg kg <sup>-1</sup>			%		
				P	Mg	K	Ca		
20	1	5.6+0.8	1.0+0.1	0.2+0.02	0.5+0.1	1.4+0.1	1.8+0.3		
	2	5.8+0.9	0.8+0.1	0.2+0.01	0.3+0.1	1.3+0.3	1.6+0.4		
	3	5.9+0.9	1.0+0.1	0.1+0.01	0.2+0.1	1.0+0.5	1.6+0.7		
44	1	5.3+0.9	0.9+0.04	0.2+0.01	0.5+0.1	1.2+0.3	1.8+0.4		
	2	5.1+0.9	0.8+0.1	0.2+0.01	0.3+0.1	1.4+0.4	1.6+0.4		
	3	5.5+1.2	1.0+0.2	0.1+0.01	0.3+0.1	1.3+0.3	2.0+0.5		
94	1	5.4+1.5	0.9+0.3	0.2+0.01	0.4+0.1	1.3+0.3	1.6+0.5		
	2	5.5+1.5	1.0+0.3	0.2+0.01	0.3+0.1	1.5+0.4	1.5+0.3		
	3	6.0+1.7	1.1+0.3	0.2+0.01	0.3+0.1	1.3+0.3	1.8+0.4		

Table A-31. Concentrations of selected elements in the second cutting of soybeans grown in soil amended with sodium molybdate.

Mo additions (mg kg <sup>-1</sup> )	pH level	Al	Cd	Fe	B	Cu	Mn	Ni
----- mg kg <sup>-1</sup> -----								
0	1	31.9+2.2	1.1+0.1	129+7	45.3+1.2	25.2+2.9	122+14	2.6+0.9
	2	31.9+1.5	1.0+0.1	121+4	38.4+5.0	27.9+2.6	104+10	1.9+0.7
	3	34.0+1.6	0.8+0.2	109+16	32.2+3.8	18.5+2.7	117+55	1.3+0.2
30	1	38.2+6.5	1.2+0.2	136+8	37.7+1.9	25.0+3.3	127+20	2.7+0.2
	2	32.0+4.3	0.8+0.1	114+18	36.1+7.1	24.9+6.0	126+11	1.7+0.6
	3	30.0+5.0	0.8+0.2	93.0+10	27.4+1.7	20.0+7.0	95.2+9	1.4+0.2
60	1	37.3+5.1	1.2+0.4	108+9	35.6+2.5	25.3+5.5	132+16	2.1+0.4
	2	35.6+4.1	1.0+0.2	124+29	36.1+8.7	29.4+2.4	133+13	1.9+0.6
	3	33.4+3.7	0.8+0.1	105+26	26.0+2.8	25.1+5.8	98.6+7	1.1+0.1
120	1	35.4+6.8	1.1+0.4	99.0+19	33.0+3.8	25.7+5.3	127+18	2.3+0.8
	2	36.4+3.0	1.1+0.2	100+6	31.1+6.7	28.5+4.3	143+35	2.3+1.0
	3	30.5+2.7	0.8+0.3	94.0+23	24.5+3.4	22.3+9.6	97.4+4	1.2+0.2



Table A-31 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> ----- % -----					
0	1	7.4+1.1	1.0+0.1	0.3+0.1	0.4+0.1	1.6+0.1	1.9+0.1
	2	7.1+0.7	1.0+0.1	0.3+0.1	0.4+0.03	1.5+0.1	2.0+0.2
	3	7.6+1.4	1.1+0.1	0.3+0.1	0.2+0.03	1.6+0.2	2.2+0.2
30	1	8.0+1.4	1.0+0.1	0.3+0.03	0.4+0.1	1.7+0.1	1.8+0.1
	2	7.3+1.1	1.0+0.1	0.3+0.03	0.4+0.1	1.7+0.2	1.9+0.2
	3	6.9+2.0	1.0+0.1	0.2+0.04	0.2+0.02	1.5+0.1	2.0+0.2
60	1	6.8+1.3	0.9+0.1	0.3+0.04	0.4+0.1	1.7+0.2	1.7+0.1
	2	7.4+1.0	1.0+0.1	0.3+0.1	0.4+0.1	1.8+0.1	1.8+0.1
	3	6.6+0.9	1.0+0.1	0.2+0.01	0.2+0.01	1.4+0.1	2.0+0.2
120	1	7.0+1.4	0.9+0.1	0.3+0.1	0.4+0.1	1.7+0.3	1.5+0.1
	2	7.8+2.0	1.0+0.1	0.3+0.1	0.4+0.02	1.8+0.1	1.8+0.2
	3	6.9+0.9	1.1+0.1	0.1+0.01	0.3+0.01	1.5+0.1	2.2+0.1

**Table A-32.** Concentrations of selected elements in the second cutting of soybeans grown in sludge amended soils.

[illegible]

Table A-32 (continued)

Mo additions (mg kg <sup>-1</sup> )	pH level	Pb	Cr	P	Mg	K	Ca
		----- mg kg <sup>-1</sup> -----	-----	-----	----- % -----	-----	-----
20	1	8.6+1.3	1.0+0.1	0.4+0.02	0.7+0.03	1.3+0.1	1.7+0.1
	2	8.2+1.7	1.0+0.03	0.4+0.01	0.5+0.1	1.4+0.2	1.9+0.2
	3	7.4+0.9	1.0+0.03	0.2+0.1	0.3+0.03	1.6+0.1	2.0+0.1
44	1	7.6+1.3	1.0+0.03	0.4+0.01	0.6+0.1	1.4+0.2	1.6+0.1
	2	8.3+2.6	1.1+0.1	0.4+0.02	0.4+0.04	1.8+0.1	1.7+0.1
	3	7.1+1.0	1.1+0.02	0.2+0.1	0.4+0.04	1.7+0.1	2.1+0.1
94	1	7.5+1.1	1.0+0.1	0.4+0.02	0.6+0.1	1.4+0.2	1.6+0.2
	2	7.8+1.4	1.1+0.1	0.3+0.1	0.4+0.03	1.7+0.1	1.9+0.3
	3	7.5+1.5	1.1+0.1	0.3+0.1	0.4+0.02	1.7+0.1	2.0+0.2

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