FERTILIZER NITROGEN RECOVERY AND ^{15}N AND BROMIDE DISTRIBUTION IN THE SOIL PROFILE AS AFFECTED BY THE TIME OF APPLICATION ON AN IRRIGATED UPLAND COTTON (*Gossypium hirsutum L.*)

by

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As members of the Dissertation Committee, we certify that we have read the dissertation

prepared by *Jose A. Cristobal Navarro-Ainza*

entitled *FERTILIZER NITROGEN RECOVERY AND* ^{15}*N AND BROMIDE DISTRIBUTION IN THE SOIL PROFILE AS AFFECTED BY THE TIME OF APPLICATION ON AN IRRIGATED UPLAND COTTON (Gossypium hirsutum L.)*

and recommend that it be accepted as fulfilling the dissertation requirement for the

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ABSTRACT

The first project involved the evaluation of different times of application on the fertilizer nitrogen recovery (FNR) in the soil-plant system in an irrigated upland cotton system, during two seasons. This was accomplished by using the isotopic dilution technique applying the ^{15}N to microplots. No differences were observed in the total plant FNR, seed and stover, soil and the total FNR among the different ^{15}N application times; however, despite no differences were observed in the plant the FNR value ranged from 30-38%, while the seed exhibited the highest FNR with an average over 50% of the ^{15}N recovered in the plant. In the soil, the obtained average FNR value was over 40% while the total FNR (plant + soil) ranged from 70-80%, being reduced as the ^{15}N application time was delayed.

The second project was conducted during two seasons to examine the ^{15}N and bromide distribution pattern in the soil profile as a function of the time of application. ^{15}Nitrogen and bromide were applied to the soil at three different times in a cotton growth cycle; after that, soil samples were taken at the end of the cotton cycle to a depth of 1.80 m., and ^{15}N and bromide recoveries were determined. Slightly higher FNR were obtained with the intermediate application time. The higher FNR were detected in the surface layer (0-30 cm) with an average of 40%. Below 30 cm depth, low ^{15}N recoveries were obtained and even lower below the 60 cm soil layer. Bromide recovery behavior was related to the water movement in the soil profile: as the Br^- application time was delayed more of the anionic tracer was found in the top of the soil profile, while less Br^- was found in the surface soil for the early Br^- application time.

I. INTRODUCTION

The production of cotton (*Gossypium spp.*) in the United States and around the world is composed basically by the production of Upland cotton (*Gossypium hirsutum L.*), being at least 91% of the worldwide planted area while the Pima cotton (*Gossypium barbadense L.*) only comprises 9% of total area. During 2005 in the world, 35.22 million hectares were planted to cotton with an average yield of 690.52 kg per hectare and a total production of 111.71 million bales. In the case of the United States, the Upland area planted during 2005 was about 5.63 million hectares, representing 98% of the cotton planted, with a production record of 23.1 million bales and an average yield of 923.7 kg per hectare; Texas is the leading cotton-producing state plus 13 states more which comprise 98% of the U.S. cotton planted. Arizona cotton planted in 2005 was 90,355 hectares with an average yield of 1,448 kg per hectare and a total production of 628,000 bales.

In the desert southwest, irrigation and fertilization practices are two important, and sometimes limiting factors, in the cotton production systems. The excessive application of water together with fertilizer sometimes can result in a threat to the groundwater and surface water sources. Regarding the fertilization practices, nitrogen (N) is the nutrient more frequently applied and in higher amounts, despite is quantitatively the most important for plant growth.

Actually, there is a considerable concern about the contamination of groundwater by agrochemicals; in fact, the Environmental Protection Agency (EPA) has established the 10 mg l^{-1} of nitrates as an upper limit for safe drinking water. Nitrate is a common compound found in groundwater, and this anion can have different sources such as fertilizers, animal feedlots, septic tanks, geological origin, among others. So, the use of N fertilizers in agriculture is a common source of nitrates, and can serve as a source of contamination of the groundwater, especially in the case of an irrational use of fertilizer and irrigation water, combined with its use on sandy or very permeable soils.

The use of labeled N is an accurate and effective way to assess the fate of N applied to the crops in the soil-plant system; it provides a direct measure of the N uptake and recovery, N balances and N losses. Despite the benefits obtained by using this technique, however, it has several disadvantages, and some of them are: i) the ^{15}N labeled fertilizers are expensive, ii) it requires detailed field and laboratory methodology, iii) laboratory methods of analysis are expensive, and iv) ^{15}N microplot size is important. Also, there are others anions like bromide which has been used as a tracer for water movement studies, and it is mentioned to behave physically in a similar fashion to nitrate in the soil. Based on this description, bromide has a potential use as a nonbiological tracer together with its very low cost compared to the use of isotopic technique.

The present studies were carried out in a cotton irrigated system during two seasons with the following objectives: i) determine the effect of the time of ^{15}N application on the fertilizer N recovery on upland cotton, and ii) establish how the distribution of ^{15}N and bromide in the soil profile is affected by the time of application.

II. LITERATURE REVIEW

II.1 NITROGEN CYCLING

Nitrogen (N) cycle in the soil is an integral part of the global N cycle. The source of the soil N is the atmosphere, where the stable molecule N_2 is the predominant gas with 79.1% by volume. After carbon, hydrogen and oxygen, N is the other element intimately associated with reactions carried out by living organisms. A key feature of the soil N cycle is the turnover of N through mineralization performed by microorganisms and soil fauna and immobilization carried out by microorganisms (Stevenson and Cole, 1999).

Nitrogen can enter or leave the soil-plant system by more routes than any other nutrient (Olson and Kurtz, 1982). Gains in soil N occur by microbial fixation of molecular N_2 and by addition of ammonia, nitrate and nitrite in rainwater; losses occur through crop removal, leaching and volatilization. Nitrogen is ultimately returned to the atmosphere as molecular N_2 by biological denitrification, thereby completing the cycle (Stevenson, 1982; Stevenson and Cole, 1999). Also, in some situations, small amount of gasses containing N escape to the atmosphere from plant leaves. Nitrogen is also contained in water of guttation excreted from foliage and in exudations from roots (Olson and Kurtz, 1982).

Among all mineral nutrients, N is quantitatively the most important for plant growth. Nitrogen is generally the first element to become deficient in plant production systems in semiarid and arid regions. This is due to the fact that soil organic matter (SOM) is the primary reserve of soil N and most soils in low rainfall areas are low in organic matter (Haggin and Tucker, 1982).

Crop N requirements and the release of available N from SOM reserves are known to depend largely on climate. Optimal crop utilization of applied N fertilizer is necessary also to minimize the risk of environmental pollution. Avoiding unwise use of N fertilizers will have direct effect in reducing pollution of ground and surface water supplies.

II.2 NITROGEN MANAGEMENT IN CROP PRODUCTION SYSTEMS

The timing of fertilizer N applications is probably more critical for dryland farming than for cropping in humid regions or under irrigation, because of the limited water availability. In general, the optimum time to apply fertilizer N from an environmental standpoint integrates at least four factors: (i) crop to be grown, (ii) climate, (iii) soil, and (iv) chemical formulation of the fertilizer (Aldrich, 1984). For example, for the mountain states, including Arizona, N rates range as high as 180 kg N ha^{-1}. Split applications of N are common and this is because the low fertility in soils and the high soil permeability. Split applications can include preplant N, band-applied N at seeding, or broadcasting N before jointing for small grains (Westfall, 1984).

In an experiment to examining the effects of seeding method and time of fertilization on urea-^{15}N recovery on rice (*Oryza sativa* L.) (Westcott et al., 1986), the timing of N application did not significantly affect dry matter production and grain yields. Total N uptake and grain N uptake were not significantly affected by timing the N fertilizer application. Westcott et al. (1986) also indicated that the percent recovery of labeled N in the soil-plant system at harvest was significantly greater with the two treatments receiving topdressed N than with the single preplant or preflood application. This was due to the fact that relatively less N was applied as a topdressing and that plants had at later stage of development, a much greater capacity for uptake, due to its larger root mass. Finally, this greater N recovery with midseason topdressing was not translated into higher grain yields; similar results have been reported for grain sorghum (*Sorghum bicolor* L.) with split N applications (Westcott et al., 1986; Mascagni and Helms, 1989).

Different N recovery results have been obtained in wheat where splitting fertilizer N resulted in both greater yield and grain N concentrations (Alcoz et al., 1993). In relation with an increased N recovery, similar observations have been made in shade tobacco (*Nicotiana tabacum*) (Rathier and Frink, 1986), suggesting that the application of N long in advance of the actual plant needs, such N is subjected to mineralization and loss due to leaching during this period early in the season.

Another study on the effect of time of urea application in calrose rice (Humphreys et al., 1987), the plant recovery of ^{15}N ranged from 3.8% for fertilization at sowing to 41.3% for topdressing at panicle initiation. Approximately two-thirds of the labeled fertilizer N was in the grain. Eighty percent of the fertilizer applied at sowing was not accounted for, compared with 45% and 40% for topdressing after permanent flood (usually established at the fourth to fifth leaf stage) and at panicle initiation, respectively. From 17-27% of the fertilizer N was retained in the soil and roots, and virtually all of this was in immobilized forms (Humphreys et al., 1987; Norman et al., 1989).

In winter wheat (*Triticum aestivum* L.) under a humid region the immobilization of ^{15}N derived from fertilizer in the surface 20 cm averaged 18.5% of the applied fertilizer N. The immobilized ^{15}N in this study represented 95.6% of the residual fertilizer in the surface soil, with the rest being in inorganic forms. Split applications of fertilizer N resulted in 19.5% ^{15}N immobilized being similar to a fall N application with the nitrification inhibitor DCD (dicyandiamide) (Bronson et al., 1991). Over 80% of the amount recovered was located in the top 5 cm of the surface soil. Despite the low soil N availability, the majority of the plant N uptake (73-97%) was derived from the soil, even where the fertilizer was used most efficiently (Humphreys et al., 1987).

In another study in rice using urea as the N source and DCD, Norman et al. (1989) indicated that significantly more fertilizer N was recovered in the rice plants at harvest each year when the fertilizer N was topdressed in three split applications than when the fertilizer N was applied all preplant, broadcast preplant incorporation or subsurface preplant incorporation, with or without DCD. The addition of DCD to the preplant treatment, resulted in significantly greater uptake of the fertilizer N. Total recovery of the fertilizer N in the soil-plant system for the different treatments studied ranged from 28 to 74% in 1984 and 36 to 68% in 1985. The highest percent recoveries of fertilizer N in the soil-plant system were realized when the fertilizer N was topdressed in three split applications or applied all preplant with DCD. Similar results have been obtained with split applications in this crop, with total recovery at maturity ranging from 74.6 to 96.8% of the applied N (Wilson et al., 1989).

Also, Guindo et al. (1994) working with rice found that the fertilizer ^{15}N uptake from the preflood N application increased until 21 days after application, peaking at 62% of the applied N. When fertilizer ^{15}N was applied at midseason, maximum fertilizer ^{15}N uptake (74% of the applied N) was observed seven days after its application. So, when N was applied in split applications, the total N accumulated until 14 days after the midseason N application, and then showed an increasing trend until maturity (Guindo et al., 1994). For winter wheat total recovery of added N has averaged 76.8%, and it is reported not to be affected by time of application or use of DCD (Bronson et al., 1991).

In another experiment in wheat, the proportion of N applied at anthesis recovered in aboveground plant biomass at maturity was always greater than that of N applied at planting. The mean fertilizer N uptake efficiency over year and N rates was 68% for N added at anthesis versus 41% for N applied at preplant. This greater efficiency of late-season vs. preplant fertilizer N may reflect the more extensive root system, better photosynthate supply, and larger sink capacity of the fully established plant community at anthesis (Wuest and Cassman, 1992).

Fertilizer N applied all preplant without DCD always had the lowest percent recoveries for rice (Norman et al., 1989). In wheat the apparent fertilizer N uptake efficiency was increased by increasing the number of split applications. The highest efficiency was achieved by the four times N application treatment (Alcoz et al., 1993).

In irrigated cotton (*Gossypium hirsutum* L.) (cv. Deltapine 61 and Deltapine 90), Constable and Rochester (1988) reported an average of N fertilizer recoveries of 30% across five experiments in Australia. Such recoveries were increased by split N applications in one of the experiments, and in one midseason (before bloom) application. The delay (before flowering) in N application produced no deleterious effects on crop yield, N recovery, nir on fiber quality. Mascagni and Helms (1989) working with grain sorghum evaluated N rates, time of N applications and nitrification inhibitors under two soil moisture conditions using a sprinkler irrigation system, and showed that the split N applications increased plant N concentration above the preplant N application at the early boot growth stage and total N uptake at physiological maturity, for both of the soil moisture treatments under study. Finally, they mentioned that split N applications may be an effective way to improve N efficiency, particularly on poorly drained soils.

Regardless of tillage, much less fall surface-applied N was utilized by corn than when

N was banded in the spring. As with whole plants, ^{15}N recovery in grain was significantly greater for spring-banded than for fall surface-applied N regardless of tillage (Timmons and Cruse, 1990).

II.3 FERTILIZER NITROGEN RECOVERY IN CROPS

In a study on the effects of salt stress on the uptake of ^{15}N by cotton, Pessarakli and Tucker (1985) found that high salinity levels affected more of the ^{15}N uptake at the vegetative than at the reproductive stage of growth. Also, at both stages of growth, ^{15}N concentrations of shoots were significantly increased for salinized plants (-0.8 MPa) compared with the controls. Finally, the total plant N uptake decreased as the culture medium became more saline.

Sanchez and Blackmer (1988) determined that only 13 to 33% of the labeled N was removed from the soil within plots during harvest of the first crop of corn (*Zea mays* L.) after the labeled fertilizer N application. Also, during the second and third crops only small percentages of the labeled N were recovered after application. The same situation has been reported for winter wheat by Bronson et al. (1991), with 1.9% of ^{15}N recovery in the second year in the grain and shoots, representing about 10% mineralization of the ^{15}N immobilized the year before.

Harris and Hesterman (1990) also indicated that an important portion of the labeled N derived from incorporated alfalfa was recovered in the grain (62%) of corn, while only 34% and 3% of labeled N was recovered in the stover and roots of the plants, respectively. The same situation has been observed in wheat (Rao et al., 1991) and in crested wheatgrass (Power and Legg, 1984). In fact, Walters and Malzer (1990a) mentioned that in corn, both grain and stover N concentrations increased with increasing rate of applied N. In the same study, the fertilizer-^{15}N recovery in the plant averaged 47, 51, and 44% of applied N at the 180 kg N ha^{-1} (1980), 90 kg N ha^{-1} (1981), and 180 kg N ha^{-1} (1981) rates, respectively.

Reddy and Reddy (1993) after one growing season of corn, obtained recoveries of N from $^{15}NH_4^+$ (ammonium) and $^{15}NO_3^-$ (nitrate) fertilizer that was in the range of 43 to 56% and from 44 to 57% of added N, respectively. Ma et al. (1995) reported for maize fertilizer N recoveries of 29 and 38%, respectively, for N fertilizer rates of 100 and 200 kg N ha^{-1} with 18 to 26% recovered in the grain. In another experiment, Timmons and Cruse (1990) found that the recovery of ^{15}N by mature corn plants ranged from 2 to 41% during two seasons, for different tillage and fertilization methods. In all the cases, according to Sutherland et al. (1993) the increases in N recovery by the plant reflects the natural ^{15}N abundance of the available soil N pool in a given period of time.

Harris and Hesterman (1990) quantified the ^{15}N contribution from alfalfa (*Medicago sativa* L.) plant material to the soil and two succeeding barley (*Hordeum vulgare* L.) crops in two locations; at both locations, and for all three soil N fractions (total, inorganic and organic N), most of the alfalfa-^{15}N recovered was from the top 30 cm of the soil. Also, recovery of alfalfa-^{15}N by the second year barley crop measured only 1% of the initial input at both locations. In general, the efficiency of N use averaged 21%; this is less than one-half of the fertilizer N use efficiency (recovery) for inorganic fertilizer N, which is generally assumed 50%.

In rice it has been found that deep placement significantly reduced immobilization of initially applied fertilizer N by the organic soil N fraction. Apparently, the results suggest that the lower immobilization seemed related to a concentration effect of the applied N, since the amount of immobilized N decreased when N was band- or point-placed rather than when N was broadcast (Schnier et al., 1988).

Another experiment with conservation tillage including wheat (Sharpe et al., 1988) reported that the fertilizer N was the primary source of N for plant uptake in the first four weeks following fertilization (day 70 to 95 after plantation); about 80% of the fertilizer N was utilized by the plants in this period. During this time, 47.8 kg ha^{-1} was from fertilizer N. After day 95, there was a drastic change in the relationship between plant N derived from the fertilizer and that derived from mineralized soil N.

In a comparison of fertilizer N sources with UAN (urea-ammonium nitrate solution) versus granular urea recovery in rice, Wilson et al. (1994) indicated that the rice plant recovered 72.5% of the N from the granular urea and 52.6% from the UAN solution in the soil. This percentage of UAN solution recovery is in the range reported by others workers (Schnier et al., 1988; Timmons and Baker, 1992); also, reported that N loss (N unaccounted for) from the soil-plant system was higher from $^{15}NO_3^-$ sources than $^{15}NH_4^+$ sources. The N unaccounted for was about threefold higher when fertilizer N application was increased from 100 to 200 kg N ha^{-1}.

In potato (*Solanum tuberosum* L.), the fertilizer N recovery was determined to be 25% and 56% for 1985 and 1986, respectively. The 85% of the fertilizer N was located in the tubers, followed by the leaves, the stem above ground, stems under ground and roots. An important amount (44 and 34%) of labeled N was left in the soil after harvest in both years (Saoud et al., 1992).

Regardless of tillage, much less fall surface-applied N was utilized than was N banded in the spring. As with whole plants, ^{15}N recovery in grain was significantly greater for spring-banded than for fall surface-applied N regardless of tillage (Timmons and Cruse, 1990).

It has been reported that the recovery of applied N in the soil-plant system has been significantly increased by the addition of several nitrification inhibitors (Freney et al., 1993). So, the addition of the wax-coated calcium carbide (nitrification inhibitor) has increased the mean recovery of applied N by 35% in cotton with the greatest effect being observed at the lowest rate of N application. The data obtained in this work suggest that the addition of this inhibitor improved uptake of fertilizer N (Freney et al., 1993). This also was detected in a study under greenhouse conditions where cotton plants had an apparent N recovery of 98% under these conditions, where dicyandiamide, a nitrification inhibitor was used as well as control of the pH to reduce the ammonia volatilization losses (Reeves and Touchton, 1989).

II.4 METHODS OF ESTIMATING RECOVERY OF NITROGEN

There are six known isotopes of N and only those having mass numbers 14 and 15 are stable and occur naturally. The stable N isotopes have been used almost exclusively in biological systems. Their use as tracers is based on the fact that ^{14}N and ^{15}N occur naturally in a ratio of 272:1 and the natural occurring N contains about 0.3663 atom % ^{15}N (Hauck and Bremner, 1976; Buresh et al., 1982). In addition, Bremner and Hauck (1982) mentioned that the ratio of ^{14}N to ^{15}N in natural and synthetic substances is not constant. Slight variations in ^{15}N abundance occur because of isotope effects (effect of nuclear characteristics other than atomic number on the nonnuclear chemical and physical properties of isotopes that lead to variations in the expression of these properties) during the biological or chemical transformations of one N form into another.

Materials with a higher or lower ^{15}N content than the natural abundance of 0.3663 atom % ^{15}N can therefore be used as tracers. The change in N isotope ratios in samples obtained from the biological system permits study of the transformations of the added tracer material. The amount of change in isotopic ratio from the background level (i.e., control plot or nonfertilized plot) permits the calculation of the extent to which the tracer has interacted with and become part of the system (Hauck and Bremner, 1976; Buresh et al., 1982).

There are three fundamental assumptions important to the use of the N isotopes as tracers in biological systems: (i) complex elements (those containing two or more isotopes) in the natural state have a constant isotope composition; (ii) living systems can distinguish one isotope from another of the same element only with difficulty, if at all; and (iii) the chemical identity of isotopes is maintained in biochemical systems Hauck and Bremner (1976). However, according to the same authors, these assumptions are valid for most studies in which ^{15}N compounds are used.

Some of the advantages of using ^{14}N and ^{15}N as tracers are derived from their inherent nonradioactivity: (i) they are stable isotopes and their use in an experimental system is not limited by time (i.e., there is no isotope decay with time); (ii) their use does not pose a health hazard to the experimenter nor cause radiation effects on the biological system under study; (iii) the residues of an experimental system do not create a disposal problem; and (iv) no permit is needed to conduct a stable nitrogen tracer experiment in the field. Among the disadvantages are: (i) ^{15}N is expensive; and (ii) high cost of the equipment needed for ^{15}N analysis (Hauck and Bremner, 1976). Several methods are available for analyzing N isotope ratio, but the most precise and convenient method involves the use of the mass spectrometer. Other methods, such as those using infrared spectroscopy, nuclear magnetic resonance, electron paramagnetic resonance, or microwave spectroscopy, have importance in N research but are not used in routine N isotope-ratio analysis (Hauck, 1982). In this study the mass spectrometer was used for analyzing the N isotope ratios on soil and plant samples.

Plant uptake of applied N can be estimated either directly by the isotope dilution method (IDM) and indirectly by the difference method (DM) (Roberts and Janzen, 1990; Varvel and Peterson, 1990; Rao et al., 1991; Stout, 1995).

The DM assumes that mineralization, immobilization, and other N transformations are the same for both fertilized and unfertilized soils (Varvel and Peterson, 1990). This indirect method commonly gives higher estimates of fertilizer N uptake than the direct tracer method (Humphreys et al., 1987; Roberts and Janzen, 1990; Varvel and Peterson, 1990). The direct tracer method is influenced by pool substitution of ^{15}N for ^{14}N (mineralization-immobilization turnover or MIT), which can result in erroneous N recovery efficiency estimations when substitution is not accounted for quantitatively. The labeled N acts as a substitute for unlabeled soil N; this substitution leaves less ^{15}N available for plant uptake and so N recoveries estimated by this method may be low. This has been confirmed by results obtained in wheat (Roberts and Janzen, 1990; Rao et al., 1991) and corn under monoculture and rotational systems (Varvel and Peterson, 1990).

All the interactions (i.e. priming effect, pool substitution, etc.) that occur when fertilizer N is added to the soil and changes in soil N content within a given pool, constitute Added Nitrogen Interactions (ANI) (Jenkinson et al., 1985). In comparing the direct and the indirect method of N uptake by winter wheat, Roberts and Janzen (1990) found that the differences between the two methods are minimized when yield response to applied N is high (i.e. under conditions of low soil fertility and good soil moisture).

II.5 BROMIDE USE IN ESTIMATING LEACHING POTENTIAL

The fate of fertilizer N applied to agricultural soils is of growing concern due to the potential for groundwater contamination and health risks associated with high nitrate (NO_3^-) levels in groundwater. In addition to the potential environmental problems associated with NO_3^- leaching, the economic impact to the farmer is considerable. Therefore, measurement of NO_3^- leaching is important for the evaluation of N fertilizer practices. However, quantitative measurements of NO_3^- leaching losses in the field are very difficult and often inadequate to fully describe the amount of NO_3^- leached.

It has been suggested that the amount of leaching is affected by the timing of irrigation, or of wetting by rainfall, relative to the progress of hydrolysis and nitrification (Scotter et al., 1984). In fact, there are many factors that affect NO_3^- leaching. These include timing, placement, and source of N in the fertilizer versus the timing, amount, and uniformity of water applications. All of which effect the degree of preferential flow of water and NO_3^- (Miller et al., 1993).

Another option to reduce the NO_3^- leaching is the use of nitrification inhibitors.Workers using lysimeters have reported that N leaching losses increased in each successive year of the experiment and averaged 20, 34.7, and 92.8 kg N ha^{-1} in 1980, 1981, and 1982, respectively, reflecting the leaching of residual N from the previous year's fertilizer application. Such N losses occurred primarily as NO_3^-. The incorporation of N or the use of a nitrification inhibitor did not significantly influence the amount of N leached in any year. Apparent fertilizer N leaching losses indicated that a twofold increase in the N application rate resulted in an average of 3.4 times more N leached over the course of the study (Walters and Malzer, 1990b).

Several techniques are commonly used for estimating soil NO_3^- leaching potentials including chloride (Cl^-) balance (Ramos, 1988), porous cup soil solution samplers (Wagner, 1962), soil sampling below the rooting zone, and vacuum extractors (Shaffer et al., 1979). Soil samples below the rooting zone can be used to accurately measure NO_3^- in the soil at a given point in time, but this is a periodic measurement which may miss much of the total N flux (Schnabel, 1983).

The recent development of an exchange resin that specifically absorbs NO_3^- has the potential for measuring NO_3^- leaching by burying it below the rooting zone. The potential advantage of an exchange resin technique for measuring NO_3^- leaching is its use to measure cummulative movement of NO_3^- without an independent measurement of water flux and without interfering with crop production (Torbert and Elkins, 1992). They also have been used for other purposes. Comparing different types of resins it is reported that approximately 80% of NO_3^- leached through large intact soil cores was retained on anion exchange resin placed at the bottom of the cores. Also, under uniform flow conditions, both resins (Dowex 1-X8 and Ionac SR-6) removed 95% of NO_3^- until the resins were more than half saturated with NO_3^- (Schnabel et al., 1993).

Bromide (Br^-) has been reported to be a good physical analog for NO_3^- in leaching studies because it is similar in size and charge to NO_3^-, is not naturally present in the soil in detectable quantities, and physically it behaves in a similar fashion (Tillman and Scotter, 1991; Jones and Schwab, 1993). Bromide background levels in soils are quite low with values commonly ranging from 5 to 40 mg kg^{-1} (Bowen, 1966; Martin, 1966; Tennyson and Settergren, 1980; Maw and Kempton, 1982).

Bromide is a common and suitable tracer for soil water studies; this tracer is not sorbed by most soils, and is not subject to chemical or biological transformations such as NO_3^-. In addition, Br^- is readily analyzed by several methods. Some new tracers like 0-TFMBA (0-{trifluoromethyl} benzoic acid), PFBA (pentafluoro- benzoic acid), and 2,6-DFBA (2,6-difluorobenzoic acid) have shown a behavior similar to Br^- and suggested as being adequate for soil water, groundwater, and solute movement studies (Bowman, 1984; Owens et al., 1985; Richter and Jury, 1986; Jabro et al., 1991; Agus and Cassel, 1992; Shipitalo and Edwards, 1993). However, Onken et al. (1975) have suggested that the use of Br^- to trace fertilizer NO_3^- should be interpreted only in a qualitative manner if used in the presence of growing plants, due in part to differential uptake of Br^- and N.

In relation to Br^- uptake by plants, Jemison and Fox (1991) published some information about the uptake of Br^- by corn, indicating that in a greenhouse study neither Br^- nor Cl^- had any significant effect on N uptake, although conversely, applied N did significantly reduce plant Br^- and Cl^- concentrations. On the other hand, in the field experiment, plants from the plot with no N applied had significantly higher Br^- concentrations than those in a manure treatment throughout most of the first year. Apparently, this higher Br^- concentration was due to the smaller plant size in the N deficient plants and not because of reduced Br^- uptake in higher N treatments.

Silvertooth et al. (1992) studying the Br^- and NO_3^- movement in a cotton production system mentioned that in general, the Br^- concentration patterns tended to peak at depths in the 0.67- to 1.2-m portion of the profile. However, detectable levels of Br^- were measured at 1.8 m in four of six plots on the last two sampling dates (158 and 178 days after planting). The NO_3^- levels were lower at these dates compared to the first dates of sampling and this was probably a function of N uptake and utilization by the crop and the timing of the N fertilizer applications. Assuming that Br^- behaves in a similar way to NO_3^-, the relatively low Br^- recoveries for the first two dates of sampling indicate a high degree of leaching potential. The later sampling dates showed progressively higher Br^- recovery levels, and therefore lower leaching potentials under these conditions.

It was reported for a wheat experiment that the addition of PPD (Phenylphosphorodiamidate) to urea improved the apparent recovery of urea substantially when urea+PPD was applied after irrigation. The topdressed application of straight urea was recovered at a rate of 52% if it was applied before irrigation, but only 31% was present as ^{15}N in the plant tops if the urea was broadcast on the wet surface after the irrigation event. Approximately 75% of the soil ^{15}N was located in the top 15 cm, for the urea-based sources when applied after irrigation, reflecting little or no leaching evidence. In contrast, when applied before irrigation, ^{15}N derived from urea or urea-DCD moved down to 15- to 30 cm layer (Katyal et al., 1987).

In studying the movement of a Br^- tracer in response to intermittent water flow in the field, Tillman et al. (1991) report an average recovery of Br^- of 112% and 101% for treatment one [sprayed application of five mm of 77 mole m^{-3} potassium bromide (KBr) solution followed immediately by the spray application of 50 mm of water in five mm pulses every 30 minutes] and treatment two (same as treatment one, except that prior the KBr application 20 mm of water was sprayed onto the soil surface, again in five mm pulses); both treatments were applied for a period of 24 hours with a total water applied of 1,200 mm. The application of 20 mm of water (smaller pores filled with water) prior to the Br^- had a marked effect on the ability of the 50 mm of water applied after the Br^- to move it further into the profile. Thus, the capillarity of the dry soil would pull the Br^- solution into the smaller pores within the aggregates in the topsoil. During subsequent irrigation of the wetter soil, flow was mostly through the larger pores, largely bypassing the smaller pores containing Br^-, making it relatively inmune to leaching.

In a second experiment, 10 microplots, 0.5 m^2 total area with no plants were used. On day 1, 5 mm of solution containing 154 mole m^{-3} of KBr was sprayed to each plot. On day 3, 30 mm of distilled water was sprayed to five of the plots (early water). On day 11, 30 mm of water was sprayed to the remaining five plots (late water). The recovery of the applied Br^- was 83% and 82% for the day 3 application (early water) and the day 11 application (late water) treatments, respectively (Tillman et al., 1991).

In another paper, Scotter and Tillman (1991) reported recoveries of 94% and 90% of the Br^- applied for the early- and late-water treatments, respectively. The authors indicate that these results shows that the application of liquid fertilizer N to a relatively dry topsoil will be much less prone to subsequent leaching than if applied to a wet soil, because diffusion in dry soil is much slower than diffusion in moist soil. On the other hand, if this fertilizer is applied to wet soil and the irrigation is delayed for several days, the leaching will be reduced markedly (Tillman et al., 1991).

In a study on the preferential flow of solutes and herbicides in three different soil textures (loam, sandy loam and sandy soil) Rice et al. (1991) indicated that the tracer Br^- velocity was about 2 to 2.5 times greater than the piston flow model velocity indicating preferential or bypass flow (Jabro et al., 1991; Rice et al., 1991; Jones and Schwab, 1993). Also, in the sandy soil the Br^- recoveries averaged 102% for the sprinkler plots and 94% for the flood plots (Rice et al., 1991). In another study, Smith et al. (1995) mentioned a 93% of Br^- recovery in the effluent and soil. Also, they indicated that Br^- concentrates in pore centers and moves with the most rapidly flowing water; little Br^- may be entering smaller pores or pores with constricted openings. Contrary to this, Everts et al. (1989) had mentioned previously that preferential flow during an irrigation event appeared to be of less overall significance in the transport of the conservative tracers, Br^-.

With the aim to reduce soil NO_3^- leaching potential it has been reported that the use of winter cover crops is a good option. In California, Jackson et al. (1993) evaluated different cover crops indicating that nonleguminous cover crops grown during the rainy winter fallow season can succesfully decrease soil NO_3^- and reduce the potential for leaching. In yet another study, it has been found that perennial ryegrass (*Lolium perenne*) crop sown in small monolith lysimeters, at the time of pea (*Pisum sativum*) residue incorporation reduced total NO_3^- leaching by only 15% during the first leaching period (september-april from the first year), whereas the grass crop during the succeeding leaching periods totally eliminated NO_3^- leaching; this confirms the beneficial effect of ryegrass in decreasing soil NO_3^- leaching in arable crop rotations (Jensen, 1994).

In evaluating the mobility of agrochemicals through soil from two tillage systems Levanon et al. (1993) showed that ponded-flow leaching of Br^-, NO_3^-, and pesticides from the surface soil tended to be greater with plow-tillage (PT) than with no-tillage (NT). The greater NO_3^- leaching from PT was explained by a greater net N mineralization in the PT vs. NT soil. The higher organic carbon (C) levels in the NT soil and surface residue may have enhanced immobilization of the surface applied NH_4NO_3 in these soils.

In another study comparing the Br^- transport under tilled and nontilled soil by using two methodologies (solution samplers and soil cores), it was reported that both methodologies showed deeper and more rapid movement of Br^- in the nontilled soil. Also, the mean solute velocity was 35% lower in the tilled soil than in the nontilled soil according to both the solution samplers and the soil cores (Fleming and Butters, 1995).

An experiment evaluating the water percolation in an irrigated field using Br^- as a tracer showed that the first 0.10 m irrigation displaced the tracer peak downward about 0.50 m. The rate of downward movement of the tracer peak was constant at 10.3 mm per day from the 0.50 m to 1.20 m depth, and then increased to 16.1 mm per day from 1.20 to 2.1 m. The increase in tracer velocity corresponded approximately with a change in soil texture. The deep percolation rate, as calculated from the tracer movement, was about five times faster than calculated by water balance and piston flow models (Rice et al., 1986).

III. DISSERTATION FORMAT

In this document are included as Appendix A and B, two manuscripts that will be submitted for publication.

Appendix A contains a manuscript entitled "Influence of Time of Application on Fertilizer Nitrogen Recovery in Upland Cotton (*Gossypium hirsutum* L.)" which will be submitted for publication to Soil Science Society of America Journal.

Appendix B contains a manuscript entitled "^{15}N and Bromide Distribution Pattern in the Soil Profile as Affected by the Time of Application" which will be submitted for publication to Agronomy Journal.

APPENDIX A

INFLUENCE OF TIME OF APPLICATION ON FERTILIZER NITROGEN RECOVERY IN UPLAND COTTON (*GOSSYPIUM HIRSUTUM* L.)

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Influence of Time of Application on Fertilizer Nitrogen Recovery in Upland Cotton (*Gossypium hirsutum* L.)

Abstract

With the aim to evaluate the effect of different times of application on the fertilizer nitrogen (N) recovery (FNR) in an irrigated upland cotton (*Gossypium hirsutum* L.) production system a study was conducted during two consecutive years. The study was carried out at the Marana Agricultural Center of the University of Arizona in southern Arizona, on a Pima clay loam soil (fine-silty, mixed, calcareous thermic Typic Torrifluvent), planted with 'DPL-20' upland cotton and furrow irrigated. Three labeled N application times (based on stage of growth from pinhead square to peak bloom) were evaluated applying a total amount of 168 kg N ha^{-1} in each treatment. Results from the study showed that total plant FNR was very similar among the different ^{15}N application times. At all the ^{15}N application times, in both years, seed exhibited the highest FNR values, compared with stover FNR, with an average of more than 60% of the ^{15}N recovered in the plant. Also, there was 25% more plant FNR during the first year than the second year. In the soil, the early ^{15}N application date (establishment) showed the higher FNR values with 46% ^{15}N recovery, and the lowest at the late ^{15}N application date (peak bloom). Also, there were differences between years in the soil FNR values. The total FNR (plant and soil) in the upland cotton system was higher in the second year. The total FNR value tended to be lower with a delay in the ^{15}N application time.

Introduction

Nitrogen (N) is quantitatively the most important mineral nutrient for plant growth. Also, this nutrient is generally the first element to become deficient in semiarid and arid regions. This is due to the fact that soil organic matter is the major reserve of soil N and most soils in low rainfall areas are low in organic matter (Haggin and Tucker, 1982). Optimal crop utilization of applied N fertilizer is therefore necessary to optimize efficiency of crop uptake and to minimize the risk of environmental pollution. The wise use of N fertilizers will also have a direct effect in reducing the pollution of ground and surface water supplies.

Plant uptake of applied N can be estimated either directly by the Isotope Dilution Method (IDM) and indirectly by the Difference Method (DM) (Roberts and Janzen, 1990; Varvel and Peterson, 1990; Rao et al., 1991; Stout, 1995). Some of the assumptions with the isotopic methods are: (i) isotope compositions in tracers are constant; (ii) living organisms cannot distinguish one isotope from another of the same element; and (iii) chemical identities of isotopes are maintained in biochemical systems. According to Hauck and Bremner (1976), these assumptions are valid for most studies in which ^{15}N compounds are used.

The DM assumes that mineralization, immobilization, and other N transformations are the same for both fertilized and unfertilized soils (Varvel and Peterson, 1990) and usually gives higher estimates of fertilizer N uptake than the direct tracer method (Humphreys et al., 1987; Roberts and Janzen, 1990; Varvel and Peterson, 1990). The direct tracer method is influenced by pool substitution of ^{15}N for ^{14}N (mineralization-immobilization turnover or MIT), which can result in erroneous N recovery efficiency estimations when substitution is not accounted for quantitatively (Varvel and Peterson, 1990). The labeled N is substituted with unlabeled soil N; this substitution leaves less ^{15}N available for plant uptake and so N recoveries estimated by this method may be low.

All interactions (i.e., priming effect, pool substitution, etc.) that occur when fertilizer N is added to the soil and change the soil N content in a given pool constitute what are referred to as the Added Nitrogen Interactions (ANI) (Jenkinson et al., 1985). In comparing the IDM and the DM of N uptake by winter wheat (*Triticum aestivum* L.), Roberts and Janzen (1990) found that the differences between the two methods are minimized when yield response to applied N is high (i.e., under conditions of low soil fertility and adequate soil moisture), and ANI are presumably low. This means that if soil fertility is low, the applied N will be taken up in a high proportion, by the growing crop leading to minimal interactions with the native N, and therefore we will observe low ANI. This situation will be reflected in the amount of N taken up by a crop, and will be similar irrespective of the method used to determine the N uptake efficiency.

There are six known isotopes of N and only those having mass numbers 14 and 15 are stable and occur naturally. The stable N isotopes have been used almost exclusively for research in biological systems. Their use as tracers is based on the fact that ^{14}N and ^{15}N occur naturally in a near constant ratio of 272:1 and natural occurring N (atmospheric N_2) contains about 0.3663 atom % ^{15}N (Hauck and Bremner, 1976; Buresh et al., 1982).

Materials with a higher or lower ^{15}N content than the natural abundance of 0.3663 atom % ^{15}N can therefore be used as tracers. The change in N isotope ratio in samples obtained from a soil-plant system following applications of labeled material (either enriched or depleted N^{15} contents) permits study of the transformations of the added tracer material. The amount of change in isotopic ratio from the background level (i.e., control plot or nonfertilized plot) permits calculation of the extent to which the tracer has interacted with and become part of the system (Hauck and Bremner, 1976; Buresh et al., 1982).

Sanchez and Blackmer (1988) determined that only 13 to 33% of the labeled N was removed by the decomposing plant material from the plots during harvest of the first crop of corn (*Zea mays* L.) in Iowa after the labeled N application in this experiment. During the second and third crops only small percentages of the labeled N were recovered after the initial application (0.3-1.5%). The same pattern has been reported for winter wheat by Bronson et al. (1991), with 1.9% of the ^{15}N recovery found in the second year following ^{15}N labeled fertilizer application, representing about 10% mineralization of the ^{15}N immobilized the year before. Fritschi et al. (2005) reported N recoveries of 5.8 and 2.6% in Acala cotton during the second and third year after labeled N application. The recovery detected in the aboveground material was from 1.4 to 2.6 times greater in the first year than in the second year after application; the N recovered by seed and lint was about 33% in the first year.

In a study addressing the ^{15}N recovery for five years of crested wheatgrass [*Agropyron desertorum* (Fisch. ex Link) Schult.] production in North Dakota, Power and Legg (1984) speculated that one might expect that grass and soils from plots previously receiving no N would initially immobilize more fertilizer N than grass and soils from plots previously receiving a high rate of N fertilization. They found that an average of 32% of the ^{15}N labeled fertilizer was recovered in the top growth the year of fertilization, while an average of 39% of the applied 15N was recovered in the soil, mostly as organic N in the first season (Power and Legg, 1984). In potatoes (*Solanum tuberosum* L.) the fertilizer N recovery was determined to be 25% and 56% for two different seasons, respectively (Saoud et al., 1992). Eighty five percent of the fertilizer N was located in the tubers, followed by the leaves, stems, and roots. A substantial portion (40%) of the labeled N was left in the soil after harvest.

Different N recovery results have been obtained in winter wheat where splitting fertilizer N resulted in both greater yield and grain N concentrations (Alcoz et al., 1993). Similar observations have been made in shade tobacco (*Nicotiana tabacum*) (Rathier and Frink, 1986), where the application of fertilizer N long in advance of the actual plant needs, resulted in a loss of fertilizer N due to leaching early in the season. Halevy et al. (1987) reported that 36% of the N was taken up by first bloom in very high yielding cotton experiments conducted in Israel. This shows that an important part of the N demand by the cotton crop occurs before first bloom and up to peak bloom. In another study where the fertilizer N uptake was estimated for Upland cotton in Arizona during seven seasons, N uptake decreased during the first four years, then increased after the fourth year. This pattern of increased N uptake after four years apparently results due to N mineralization (Navarro et al., 1997a,b). Also, this reflects the importance of considering residual N and the potential mineralization in the soil, where N is cycled and being available to the crop after several seasons.

The fertilizer N uptake efficiency in winter wheat was increased by increasing the number of split applications. The highest efficiency was achieved by four-split treatments (Alcoz et al., 1993). In cotton cv. Deltapine 61 and Deltapine 90, Constable and Rochester (1988) reported an average of N fertilizer recovery of 30% across five experiments. Fertilizer N recovery was increased by split N applications in one of the experiments with one midseason N application. The delay in N application produced no deleterious effects on crop yield, N recovery, or on fiber quality.

Current N management recommendations in many cotton-producing regions like

Arizona include the use of split applications of N fertilizer. Under the irrigated conditions in Arizona and the desert Southwest, fertilizer N applications are recommended to be split between pinhead square and peak bloom which is referred to as "N application window", considering the crop condition and previous amounts of in-seasons N fertilizer applied (Silvertooth and Doerge, 1990; Silvertooth et al., 1990; Silvertooth et al., 1991; Silvertooth et al., 1995; Silvertooth et al., 1996; Unruh and Silvertooth, 1996; Silvertooth et al., 2005). Nitrogen uptake has been shown to be greatest splitting N applications to wheat (Alcoz et al., 1993), and also split applications it is considered to lower the nitrate leaching potential.

This study was conducted to verify current N management guidelines and recommendations for cotton production under irrigated conditions in the desert Southwest. These guidelines recommend split N applications in the "N application window" from pinhead square to peak bloom which extends from about 400 heat units after planting (HUAP) up to 2000 HUAP (Silvertooth et al., 1990; Silvertooth et al., 1991; Silvertooth et al., 1995; Silvertooth et al., 1996; Silvertooth et al., 2005). The objective of this study was to compare the effect of N application times on the FNR on an Upland cotton irrigated system within the N application window that is recommended.

Materials and Methods

A field study was conducted in 1994 and 1995 on a Pima clay loam (fine-silty, mixed, calcareous thermic Typic Torrifluvent) soil located at the University of Arizona Marana Agricultural Center in southern Arizona (600 m elevation). The field was wet planted to 'DPL-20' Upland cotton (*Gossypium hirsutum* L.) on 18 April 1994 and on 24 April 1995 in plots eight rows wide with a row spacing of 1 m. The field was furrow irrigated with macroplots extending the full length of the 183 m irrigation run (north-south orientation). Planting was accomplished with a conventional (John Deere Maximerge; Deere & Co., Moline, IL) planter.

A set of three microplots was established in each macroplot; one at the head, the middle, and the end of the field, each one separated by approximately 48 m (Silvertooth et al., 1997; Silvertooth et al., 1998). The macroplots were set up in the same exact location each year while the microplots were established above (toward the head of the field) the previous year's microplots (about 5.0 m) and were randomized both years. Microplots for ^{15}N applications were placed in the center four rows of eight row macroplots (Figure 1). The macroplots that were used in this experiment had not received fertilizer N applications for seven years. Microplots consisted of four rows 1 m length (4 m^2 total area) (Silvertooth et al., 2001). The applied treatments of ^{15}N labeled fertilizer in this study were carried out at three application times: establishment, between pinhead square PHS and early bloom, and at peak bloom stage (Table 1). The N fertilizer source was $(^{15}NH_4)_2\ SO_4$ with 5 atom % ^{15}N and non-labeled $(NH_4)_2\ SO_4$. The fertilizer N was applied at a rate of 56 kg N ha^{-1} on each date of application, with a total amount of 168 kg N ha^{-1} applied to each treatment. Each treatment received three applications of 56 kg N ha^{-1}. Non-labeled $(NH_4)_2\ SO_4$ was used on the application dates where ^{15}N labeled materials were not prescribed (Table 2). Based on a preplant soil testing analyses no phosphorus (P) or potassium (K) fertilizer was used (Silvertooth et al., 1995; Silvertooth and Norton, 1996).

The N fertilizer applications were made by dissolving the appropriate amounts of ^{15}N labeled and/or non-labeled materials in 500 ml of distilled-deionized water. This solution was evenly distributed by hand in a trench 1 m in length, and approximately 15 cm deep which was cut 15 cm in the west side of the bed simulating a sidedress N application. After the N-solution was applied the trench was immediately covered with soil (Silvertooth et al., 2001). All cultivation, irrigation and pest management practices were conducted in a uniform manner on an as-needed basis. During 1994 a total of five in-seasons irrigations were applied with approximately 130 mm of water per irrigation for a total of 650 mm of water. In 1995 three in-seasons irrigations were applied for a total de 390 mm of water (Table 3). Irrigation water used in this study provided an average of 5 mg/L NO_3^--N that corresponded to about 30 kg NO_3^--N ha^{-1}, considering 60 cm of total water applied to the crop. The rainfall recorded during the growing season was 133.1 mm during 1994 and 138.17 mm for 1995 (Table 3). Irrigation termination dates were on August 28 and August 1 for 1994 and 1995 seasons, respectively.

Harvest was accomplished by hand picking all seedcotton (lint+seed) from the plants located in the central 0.5 m of the two middle rows of each microplot, with a total sample area of 1 m^2 (Figure 2). Stover samples were obtained by clipping plants at the soil surface and collecting the entire aboveground plant material, including leaves found on the surface off the ground in the sample area. This material was dried with a forced-air oven at a temperature of 65°C for 48 hours or until constant weight was obtained. Dry weights were obtained for each of the plant portions following separation. After drying, the samples were chopped and ground first with a hammer mill (1.4 mm mesh screen) then with a Wiley mill to pass a 1 mm mesh screen. The seed was separated from the lint by use of a small gin, and then the seed was ground with a Wiley mill to pass a 1 mm mesh screen. Subsamples of the stover and the seed were further ground to a very fine powder for laboratory analysis.

At the end of each season single soil cores were obtained from a central row in the seed row line on each microplot using a 5 cm diameter auger. Soil samples were obtained in late October in 1994 and mid-October in 1995, after harvest and before disking the soil. The soil sample location was in the center of the row, 0.5 m from the edge of the microplot. Soil samples were collected at 30 cm intervals to a depth of 180 cm (Figure 3). Soil samples were air-dried and ground to pass a 1 mm mesh screen. A subsample from each soil sample was obtained and further ground to a very fine powder for laboratory analysis. Determinations of ^{15}N content were made from isotope ratio analyses for each sample (plant and soil). Total N and isotope ratio analyses were carried out for all soil, stover, and seed samples by use of a Carlo Erba N/A 1500 Elemental Analyzer (CE Elantech, Inc., Lakewood, NJ) and a VG Isomass Mass Spectrometer (Isomass Scientific, Inc., Calgary, AB).

Fertilizer N recovery (FNR) was calculated using the following equation (Hauck and Bremner, 1976):

$$FNR = P(c-b)/f(a-b)$$

where *P* is the total N in plants or soil (kg ha^{-1}); *f* is the labeled N fertilizer applied (kg ha^{-1}); and *a*, *b* and *c* are atom percentages of ^{15}N in fertilizer (*a*), in control plants or soil with non-labeled fertilizer (*b*), and in plants or soil with labeled fertilizer (*c*).

Crop development was monitored in terms of plant height, mainstem node number, fruit retention, and HUAP (Silvertooth et al., 1995). Petiole samples from the youngest fully expanded leaves were taken on biweekly intervals throughout the growing season up to cut-out (end of the primary fruiting cycle), and analyzed for NO_3^- -N levels to monitor the in-season N status of the crop in the study area. These analyses were performed using a NO_3^- -N specific electrode (Silvertooth and Doerge, 1990).

Lint yield, total N uptake, plant fertilizer N recovery (PFNR), soil fertilizer N recovery (SFNR) and total fertilizer N recovery (TFNR) in the soil-plant system were determined and analyzed statistically. The experimental design was a randomized complete block with four replications. Analysis of variance procedures were conducted as outlined by Steel and Torrie (1980) and the SAS Institute (SAS, 1994).

Results and Discussion

Lint Yield and Total N Uptake

The analysis of variance of lint yield showed no significant differences ($P \leq 0.05$) among the ^{15}N application times, indicating no differences in terms of applying N from early PHS up to peak bloom (Figure 4). This supports previous findings from Unruh and Silvertooth (1996) whom found maximum rates of N uptake between 400 and 1600 HUAP (30/13°C thresholds) for irrigated cotton in Arizona, which comprise N applications from PHS up to peak bloom stage of crop growth. In fact, according to Fritschi et al. (2005) cotton plants in California took up to ≈ 70% of the fertilizer-^{15}N recovered from the belowground pool (soil-root) between early PHS and peak bloom with a similar pattern to that of the total plant N uptake.

With respect to the total N uptake during the first season, higher amounts of N were taken up by cotton plants in 1994 with no significant differences among N application times (Figure 5). The average total N uptake in both seasons was close to 150 kg N ha^{-1}. Navarro et al. (1997a,b) reported an estimated total N uptake of 168 kg N ha^{-1}, using the DM for Marana, AZ. Our values in this experiment were lower but close to these previous values, and such a difference could be attributed to the difference of the methods utilized to determine FNR (Carefoot et al., 1993; Harmsen and Moraghan, 1988; Hauck and Bremner, 1976; Norton and Silvertooth, 1998; Rao et al., 1991).

Plant Fertilizer Nitrogen Recovery

Fertilizer N recovery of cotton stover and seed, as a function of the labeled N application time is presented in Table 3. No significant differences in the FNR of stover and seed were found among the three ^{15}N application times in both years. These results agree with those reported by Westcott et al. (1986) on rice (*Oriza sativa*). Only slight differences were observed, although not statistically significant, between years in our experiments and between stover and seed plant parts. In both years, seed exhibited higher ^{15}N recoveries than stover, which is due to the fact that seed commonly takes up more N and other nutrients because of the high N sink demand in seed (Unruh and Silvertooth, 1996).

Similar trends were reported for grain sorghum (*Sorghum vulgare L.*) (Mascagni and Helms, 1989), rice (Humphreys et al., 1987; Norman et al., 1989; Wilson et al., 1989), wheat (Alcoz et al., 1993), and oat (*Avena sativa L.*) (Riga et al., 1980). This has been attributed to the fact that relatively less N was applied as a topdressing and plants had a greater capacity for uptake at later stages of development (Westcott et al., 1986). Rathier and Frink (1986) suggested that the application of N in advance of the actual plant needs expose the N to soil processes such as mineralization and losses due to leaching.

Averaging for both years and ^{15}N application times in this project, ^{15}N recovery in the seed was slightly over 60% of the total ^{15}N recovery (Table 4). Humphreys et al. (1987) reported that in rice approximately two-thirds of the plant ^{15}N recovery was in the grain, which is close to the value obtained in our studies. Ma et al. (1995) indicated that fertilizer N recoveries in the grain were 50% above those recovered in other plant parts in maize. For potatoes (*Solanum tuberosum L.*) an average FNR of 40% has been reported, from which 85% of the fertilizer N was located in the tubers (Saoud et al., 1992).

In our study, the PFNR (stover + seed) by the cotton plant (only aboveground material) exhibited no statistical differences among the ^{15}N application times. Also, the average PFNR for both years was 34.23% for our studies (Table 4).

Soil Fertilizer Nitrogen Recovery

The data from 1994 and 1995 indicate that there were no statistical differences (Table 5) in terms of the SFNR among treatments and both years. The average SFNR values for both years ranged from 45% (at the early ^{15}N application time) to about 35% (at the late ^{15}N application time). These values of N recovered in the soil would be considered high in relation to results from Torbert and Reeves (1994), Freney et al. (1993) and Karlen et al. (1996) where values of 13 to 35% were reported. However, these results do match well with those reported by Fritschi et al. (2004) in California for Upland and Pima (*G. barbadense* L.) cotton. Differences noted could be attributed to plant uptake patterns that may differ among crops and to differences in the sampling of the soil profile to 1.8 m in this study compared to 0.9 m depth or less in the other studies mentioned. Considering the average soil FNR values of both years, there is a trend for lower ^{15}N recovery in the soil as we delayed the ^{15}N application time (Figure 7), although it was not significant.

Total Fertilizer Nitrogen Recovery

The total fertilizer nitrogen recovery (TFNR) in Upland cotton for this project is presented in Table 6. There were no statistical differences among the ^{15}N application times.

Even though treatments were not significantly different, the second season exhibited slightly higher TFNR values compared with the first season. Also, a high degree of variation (coefficient of variation, CV) was found for TFNR for both seasons, with CVs in excess of 20%. When years are pooled, a slight trend (NS) can be observed where the TFNR is reduced as the ^{15}N application time is delayed, with values ranging from approximately 80% at the early ^{15}N application time to about 70% with the late ^{15}N application time (Figure 8). Unaccounted fertilizer N was in the range from 20-30% for both years. These high TFNR values indicate that the fertilizer N losses were small for the treatments used suggesting irrigation was properly managed and fertilizer application were made during periods of rapid uptake of N by the plant. These values are in the normal range of FNR for the soil-plant system as previously reported in other studies (Torbert and Reeves, 1994; Karlen et al., 1996; Rochester et al., 1997). For the N rates used in this study the losses would correspond to about 33-50 kg N ha^{-1} of N fertilizer. It is important to note that N fertilizer remaining in the roots was not accounted for in this study.

Conclusions

The PFNR was similar for all ^{15}N application dates in this study. In both years and at all application times, the seed fraction showed the highest FNR with an average over 60% of the ^{15}N recovered in the plant. Average SFNR values were greater than those obtained in the plant (both years). The TFNR (plant and soil) in this upland cotton system were similar both years but slightly higher in the second year. Trends in this study suggest that as we delay the ^{15}N application time, TFNR declines.

The information obtained from this study indicates that there are no significant differences in recovery potential by applying N between early PHS up to peak bloom: In this range of application times we can realize similar N recovery in the soil-plant system. This work provides evidence to reinforce the current guidelines and recommendations for N application in irrigated cotton production systems in the desert Southwest (Silvertooth and Doerge, 1990) in what is termed the N application window (Silvertooth et al., 2005), where high N recovery and reduced N losses from the soil-plant system can be realized.

Future studies of this type should include a higher number of replications in an effort to reduce the variation found in this study. Also, it would be helpful to evaluate other application times, such as before or after the prescribed N application window.

References

Alcoz, M.M., F.M. Honz, and V.A. Haby. 1993. Nitrogen fertilization timing effect on wheat production, nitrogen uptake efficiency, and residual soil nitrogen. Agron. J. 85:1198-1203.

Bronson, K.F., J.T. Touchton, R.D. Hauck, and K.R. Kelley. 1991. Nitrogen-15 recovery in winter wheat as affected by application timing and dicyandimide. Soil Sci. Soc. Am. J. 55:130-135.

Buresh, R.J., E.R. Austin, and E.T. Craswell. 1982. Analytical methods in ^{15}N research. Fert. Res. 3:37-62.

Carefoot, J.M., J.B. Bole, and R.L. Conner. 1993. Effect of timing of application on the recovery of fertilizer N applied to irrigated soft white wheat. Can. J. Soil Sci. 73: 503-513

Constable, G.A. and I.J. Rochester. 1988. Nitrogen application to cotton on clay soil: timing and soil testing. Agron. J. 80:498-502.

Freney, J.R., D.L. Chen, A.R. Mosier, I.J. Rochester, G.A. Constable, and P.M. Chalk. 1993. Use of nitrification inhibitors to increase fertilizer nitrogen recovery and lint yield in irrigated cotton. Fert. Res. 34:37-44.

Fritschi, F.B., B.A. Roberts, D.W. Rains, R.L. Travis and R.B. Hutmacher. 2004. Fate of nitrogen-15 applied to irrigated acala and pima cotton. Agron. J. 96: 646-655

Fritschi, F.B., B.A. Roberts, D.W. Rains, R.L. Travis and R.B. Hutmacher. 2005. Recovery of residual fertilizer-N and cotton residue-N by acala and pima cotton. Soil Sci. Soc. Am. J. 69:718-728

Hagin, J., and B. Tucker. 1982. Fertilization of dryland and irrigated soils. Springer-Verlag, Berlin, Germany.

Halevy, J., A. Marani, and T. Markovitz. 1987. Growth and NPK uptake of high-yielding cotton grown at different nitrogen levels in a permanent plot experiment. Plant and Soil 103: 39-44.

Harmsen, K. and J.T. Moraghan. 1988. A comparison of the isotope recovery and difference methods for determining nitrogen fertilizer efficiency. Plant and Soil 105: 55-67.

Hauck, R.D. and J.M. Bremner. 1976. Use of tracers for soil and fertilizer nitrogen research. Adv. Agron. 28:219-266.

Humphreys, E., P.M. Chalk, W.A. Muirhead, F.M. Melhuish, and R.J.G. White. 1987. Effects of time of urea application on combine-sown calrose rice in south-east australia. 111. Fertilizer nitrogen recovery, efficiency of fertilization and soil nitrogen supply. Aust. J. Agric. Res. 38:113-127.

Jenkinson, D.S., R.H. Fox, and J.H. Rayner. 1985. Interactions between fertilizer nitrogen and soil nitrogen-the so called ‘priming’ effect. J. Soil Sci. 36:425-444.

Karlen, D.L., P.G. Hunt, and T.A. Matheny. 1996. Fertilizer 15Nitrogen recovery by corn, wheat and cotton grown with and without preplant tillage on Norfolk loamy sand. Crop Sci. 36: 975-981.

Ma, B.L., M.L. Dwyer, B.C. Liang, and E. Gregorich. 1995. Use of nitrogen–15 microplots for field studies of maize nitrogen –use efficiency. Commun. Soil Sci. and Plant Anal. 26:1813-1822.

Mascagni, Jr. H.J., and R.S. Helms. 1989. Effect of nitrogen rate, nitrogen timing and nitrification inhibitors on grain sorghum production in Arkansas. Commun. Soil Sci. and Plant Anal. 20:2117-2136.

Navarro, J.C., J.C. Silvertooth, and A. Galadima. 1997a. Fertilizer nitrogen recovery in irrigated upland cotton. p. 402-407. *In* Cotton: A College of Agriculture report. Series P-108. Univ. of Arizona, Tucson.

Navarro, J.C., J.C. Silvertooth, and A. Galadima. 1997b. Fertilizer nitrogen recovery in irrigated upland cotton. p. 581-583. *In* Proc. Beltwide Cotton Conf., New Orleans, LA. 6-10 Jan., 1997. National Cotton Council, Memphis, TN.

Norman, R.J. , B.R. Wells, and K.A.K. Moldenhauer. 1989. Effect of application method and dicyandiamide on urea-nitrogen-15 recovery in rice. Soil Sci. Soc. Am. J. 53:1269-1274.

Norton, E.R. and J.C. Silvertooth. 1998. Field validation of soil solute profiles in irrigated cotton. Agron. J. 90:623-630.

Power, J.F. and J.O. Legg. 1984. Nitrogen –15 recovery for five years after application of ammonium nitrate to crested wheatgrass. Soil Sci. Soc. Am. J. 48:322-326.

Rao, A.C.S., J.L. Smith, R,I. Papendick, and J.F. Parr.1991. Influence of added nitrogen interaction in estimating recovery efficiency of labeled nitrogen. Soil Sci. Soc. Am. J. 55:1616-1621.

Rathier, T.M. and C.R. Frink. 1986. Efficiency of nitrogen fertilizer us by shade tobacco improved by timed applications. Agron. J. 78:459-464.

Riga, A., V. Fischer, and H.J. van Praag. 1980. Fate of fertilizer nitrogen applied to winter wheat as $Na^{15}NO_3$ and $(^{15}NH_4)_2SO_4$ studied in microplots through a four course rotation : 1. Influence of fertilizer splitting on soil and fertilizer nitrogen. Soil Sci. 130:88-99.

Roberts, T.L. and H.H. Janzen. 1990. Comparison of direct and indirect methods of measuring fertilizer N uptake in winter wheat. Can. J. Soil Sci. 70:119-124.

Rochester, I.J., G.A. Constable, and P.G. Saffigna. 1997. Retention of cotton stubble enhances N fertilizer recovery and lint yield of irrigated cotton. Soil Tillage Res. 41: 75-86.

Sánchez, C.A. and A.M. Blackmer. 1988. Recovery of anydrous ammonia-derived nitrogen–15 during three years of corn production in Iowa. Agron. J. 80: 102-108.

SAS Institute, 1994.The SAS system for Windows. Release 6.10. SAS Inst., Cary, N.C.

Saoud, A.A., O. Van Cleemput, and G. Hofman. 1992. Uptake and balance of labeled fertilizer nitrogen by potatoes. Fert. Res. 31: 351-353.

Silvertooth, J.C., L.J. Clark, E.W. Carpenter, J.E. Malcuit, P.T. Else, and T.A. Doerge. 1990. Nitrogen management in irrigated cotton. Cotton, A College of Agriculture Report. University of Arizona. Series P-81:169-174

Silvertooth, J.C. and T.A. Doerge. 1990. Nitrogen management in Arizona cotton production. College of Agriculture Report 9024. University of Arizona, Tucson.

Silvertooth, J.C., J.E. Watson, J.E. Malcuit, and T.A. Doerge. 1991. Solute movement in an irrigated cotton production system. Soil Sci. Soc. Am. J. 56:548-555.

Silvertooth, J.C., E.R. Norton, B.L. Unruh, J.C. Navarro, L.J. Clark and E.W. Carpenter 1995. Nitrogen management experiments for Upland and Pima cotton, 1994. Cotton, A College of Agriculture Report. University of Arizona. Series P-99:311-326

Silvertooth, J.C., and E.R. Norton. 1996. Nitrogen management experiments for Upland and Pima cotton, 1995. Cotton, A College of Agriculture Report. University of Arizona. Series P-103:317-332

Silvertooth, J.C., E.R. Norton, C.A. Sánchez, J.C. Navarro, and A. Galadima. 1997. Evaluation of a nitrogen–15 microplot design in a furrow irrigated row crop system. 89th annual meeting, Am. Soc. of Agron., Anaheim, CA. Agron. Abstr. p. 241

Silvertooth, J.C., E.R. Norton, J.C. Navarro, A. Galadima, and C.A. Sánchez. 1998. Field plot design evaluation for nitrogen-15 labeled fertilizers in a furrow irrigated system. Proceedings of the Beltwide Cotton Conferences. San Diego, CA.

Silvertooth, J.C., J.C. Navarro, E.R. Norton, and C.A. Sanchez. 2001. Evaluation of a nitrogen-15 microplot design in furrow-irrigated cotton. Soil Sci. Soc. Am. J. 65: 247-250

Silvertooth, J.C., A. Galadima, and E.R. Norton. 2005. Residual soil nitrogen evaluations in irrigated desert soils, 2004. Cotton, A College of Agriculture Report. University of Arizona. Series P-142:152-161.

Steel, R. and J. Torrie. 1980. Principles and procedures of statistics, a biometrial approach McGraw-Hill, Inc. San Francisco, CA.

Stout, W.L. 1995. Evaluating the "added nitrogen interaction" effect in forage grasses. Commun. Soil Sci. and Plant Anal. 26:2829-2841.

Torbert, H.A., and D.W. Reeves. 1994. Fertilizer nitrogen requirements for cotton production as affected by tillage and traffic. Soil Sci. Soc. Am. J. 58: 1416-1423.

Unruh, B.L. and J.C. Silvertooth. 1996. Comparisons between an Upland and a Pima cotton cultivar: ll. Nutrient uptake and partitioning. Agron. J. 88: 589-595

Varvel, G.E. and T.A. Peterson. 1990. Nitrogen fertilizer recovery by corn in monoculture and rotation systems. Agron. J. 82:935-938.

Westcott, M.P., D.M. Brandon, C.W. Lindau, and W.H. Patrick Jr. 1986. Effects of seeding method and time of fertilization on urea-nitrogen-15 recovery in rice. Agron. J. 78:474-478.

Wilson, Jr. C.E., R.J. Norman, and B.R. Wells. 1989. Seasonal uptake patterns of fertilizer nitrogen applied in split applications to rice. Soil Sci. Soc. Am. J. 53:1884-18 87.

Table 1. Description of the labeled N treatments applied to 'DPL-20' Upland cotton, 1994 and 1995.

^{15}Nitrogen application time	Crop growth stage	HUAP†
Early	Establishment (vegetative growth stage)	≈400
Intermediate	PHS-Early bloom	≈1000
Late	Peak bloom	≈2000

†HUAP = Heat Units After Planting (30°/13°C thresholds)

Table 2. Application times and amount of labeled and unlabeled N applied to Upland cotton, 1994 and 1995.

Treatments	------ N application time ------		
	1	2	3
	--------- kg N ha^{-1} ---------		
Early	56†	56	56
Intermediate	56	56†	56
Late	56	56	56†
Total	168	168	168

†^{15}N labeled fertilizer (kg ha^{-1})

Table 3. Effect of the ^{15}N application time on the stover and seed fertilizer N recovery in Upland cotton, 1994 and 1995.

^{15}N application time	FNR (%)			
	1994		1995	
	Stover	Seed	Stover	Seed
Establishment	14.44 a†	19.57a	10.99 a	20.27a
PHS-Early bloom	15.15 a	26.22a	11.51 a	20.02a
Peak Bloom	16.12 a	22.76a	10.13 a	18.19 a

†Means within a column followed by the same letter are not significantly different at the 0.05 probability level according to Fishers LSD

Table 4. Fertilizer N recovery (FNR) of stover and seed and plant fertilizer N recovery (PFNR) in Upland cotton, 1994 and 1995.

^{15}N application time	---------- FNR† ---------- Stover	Seed	PFNR (%)‡
	------------------------ % ------------------------		
Establishment	12.71 a§	19.92 a	32.63 a
PHS-Early bloom	13.33 a	23.12 a	36.45 a
Peak bloom	13.12 a	20.48 a	33.60 a

† Fertilizer N recovery obtained as a 2-years average

‡ PFNR is obtained of the sum of stover and seed plant parts

§ Means within a column followed by the same letter are not significantly different at the 0.05 probability level according to Fishers LSD

Table 5. Influence of the ^{15}N application time on the soil fertilizer N recovery (SFNR), 1994 and 1995.

^{15}N application time	----------------- SFNR --------------------		
	1994	1995	Mean
Establishment	33.19 a†	58.86 a	46.03 a
PHS-Early bloom	38.32 a	39.22 a	38.77 a
Peak bloom	26.22 a	44.58 a	35.40 a

† Means within a column followed by the same letter are not significantly different at the 0.05 probability level according to Fishers LSD

Table 6. Influence of the ^{15}N application time and year on the total fertilizer N recovery (TFNR) in Upland cotton, 1994 and 1995.

^{15}N application time	------------ TFNR -------------		
	1994	1995	Mean
	……………. % ……………..		
Establishment	67.20 a†	90.12 a	78.66 a
PHS-Early bloom	79.69 a	70.75 a	75.22 a
Peak bloom	65.10 a	72.90 a	69.00 a

† Means within a column followed by the same letter are not significantly different at the 0.05 probability level according to Fishers LSD

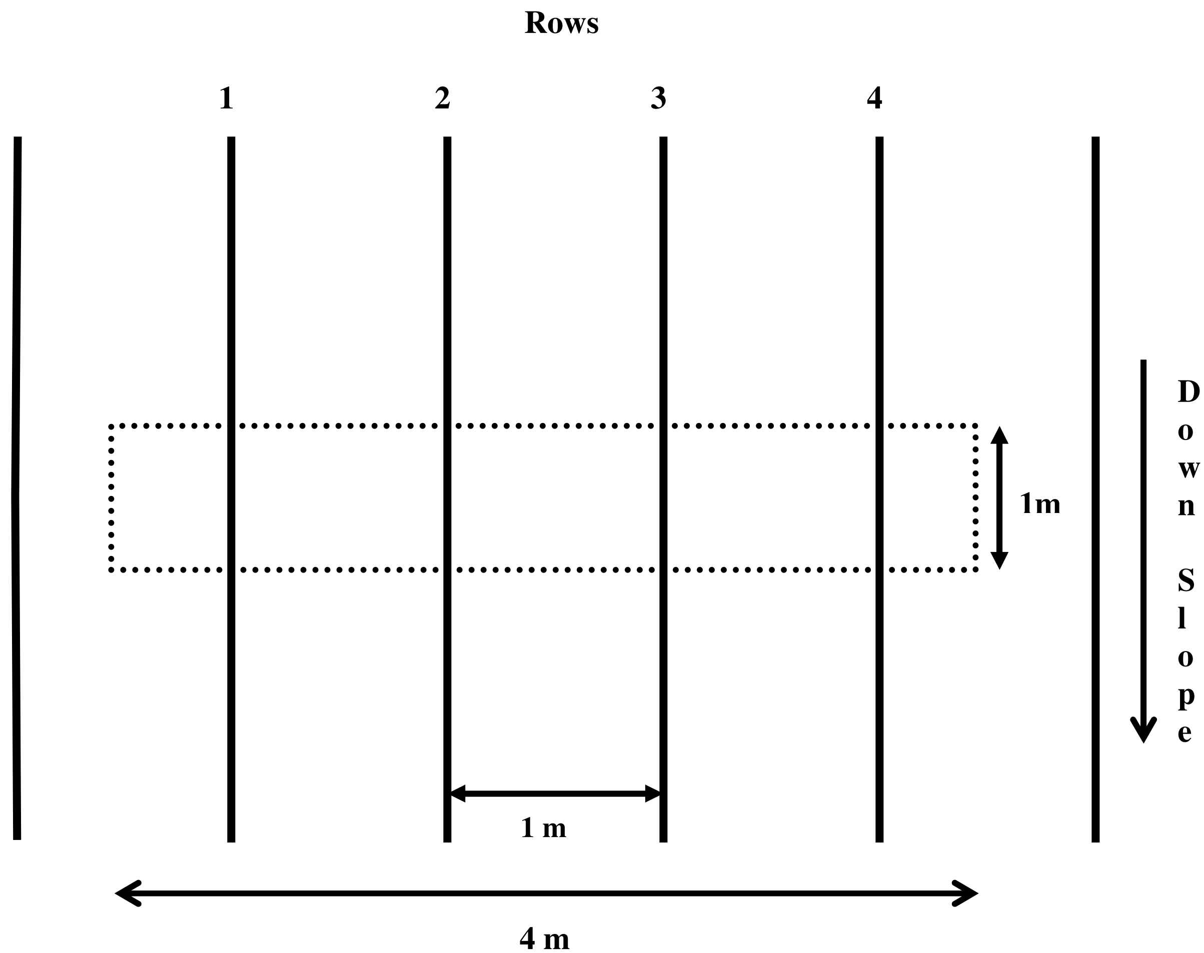

Figure 1. ^{15}N **plot structure for 1994 and 1995**

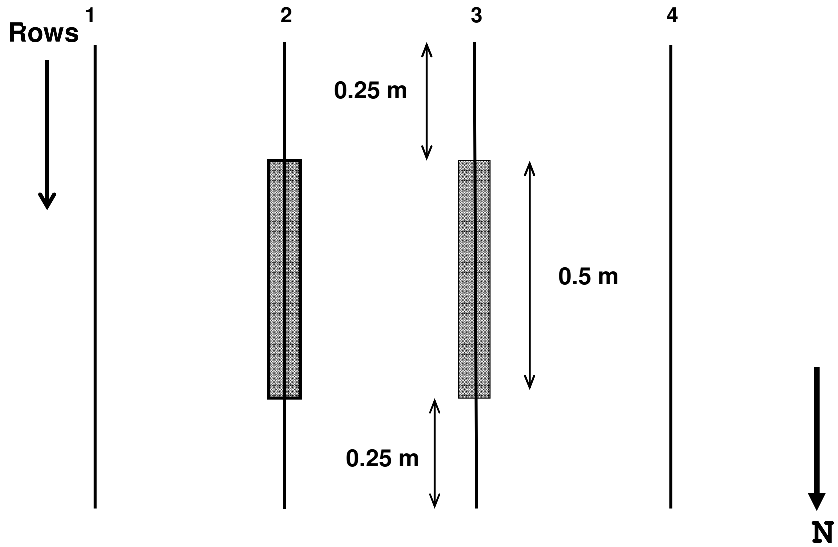

Figure 2. Harvested area and plant sampling scheme (shaded area) in the middle two rows of the microplot for 1994 and 1995.

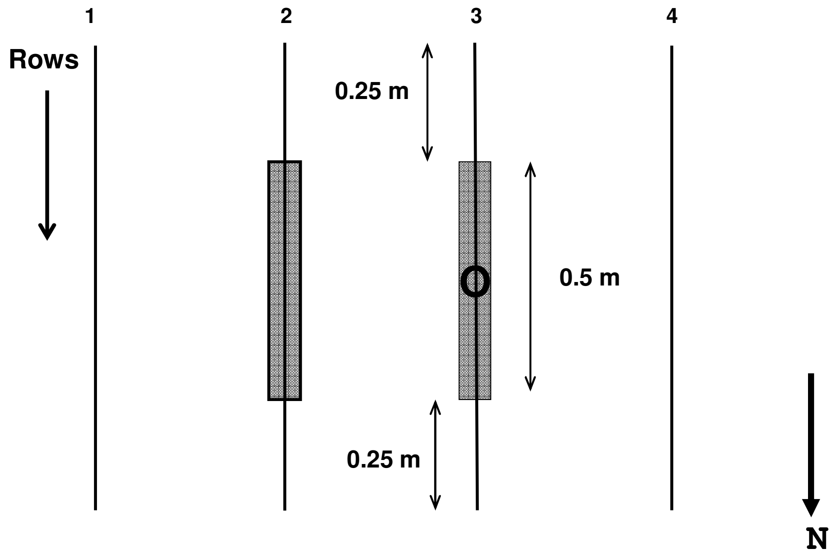

Figure 3. Soil samples taken from the center of a seed row in the microplot area for 1994 and 1995.

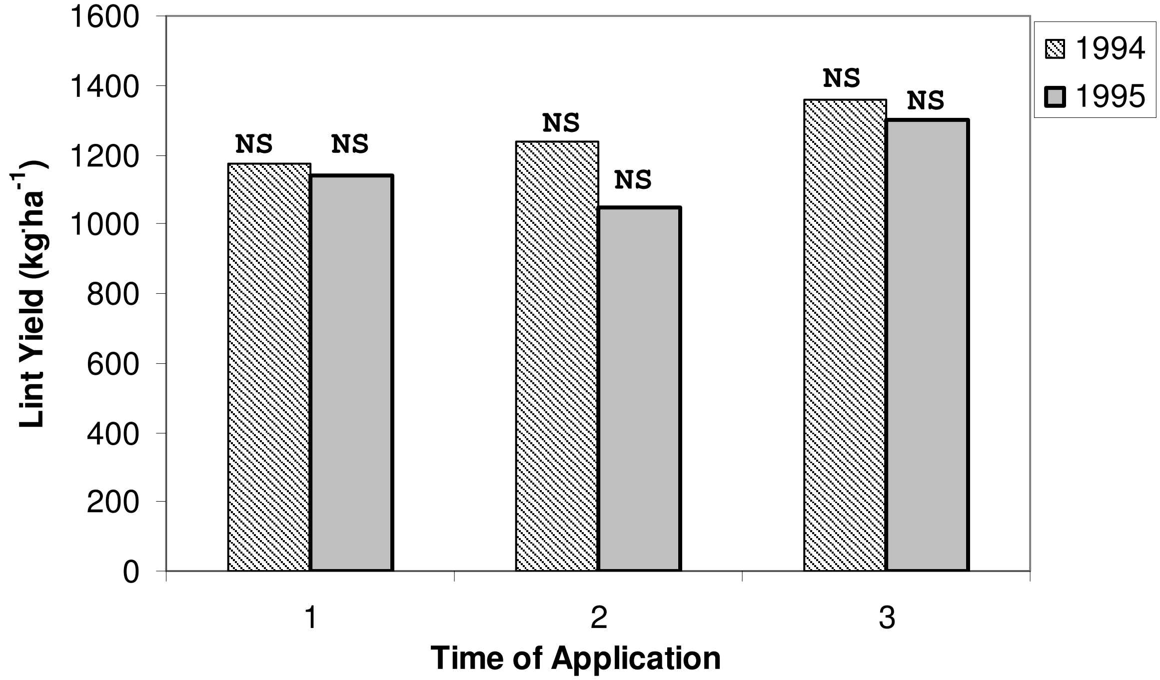

Figure 4. Effect of the N application time on lint yield for 1994 and 1995. NS indicates differences were not significant among treatments within a year ($P<0.05$).

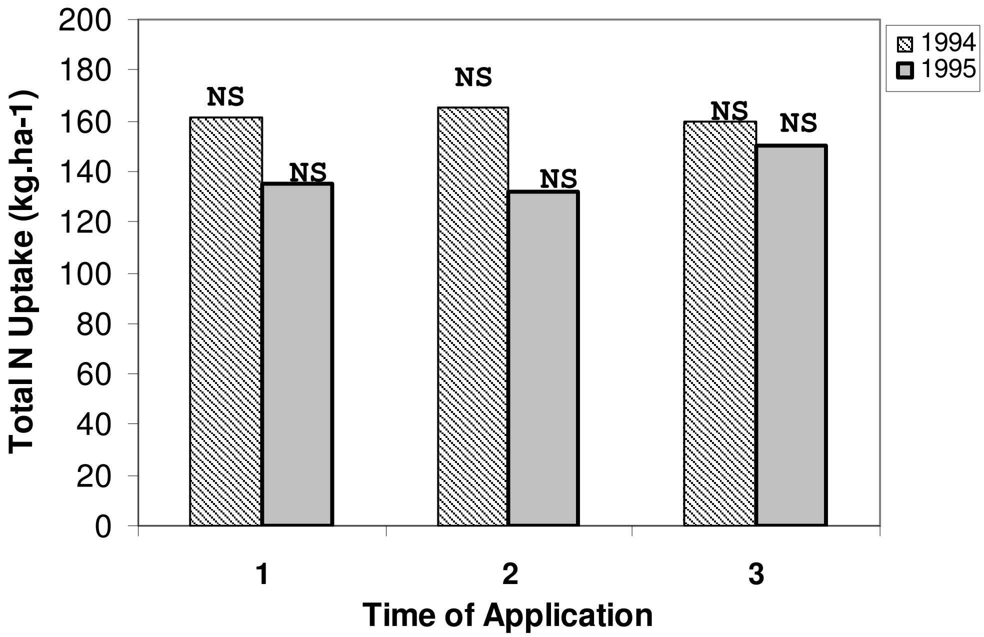

Figure 5. Effect of N application time on the total N uptake in Upland cotton, 1994 and 1995. NS indicates differences were not significant among treatments within a year (P<0.05).

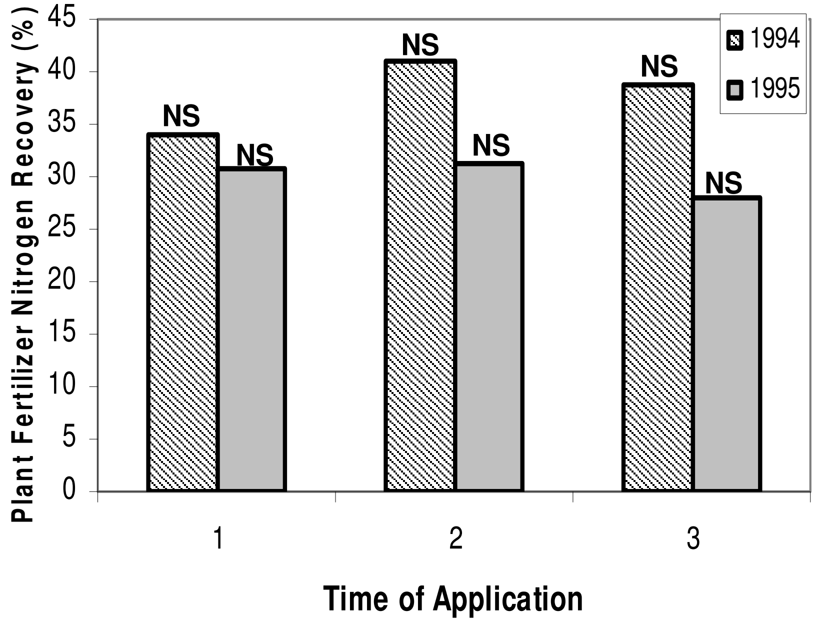

Figure 6. Plant fertilizer N recovery in Upland cotton as affected by the fertilizer N application time, 1994 and 1995. NS indicates differences were not significant among treatments within a year (P<0.05).

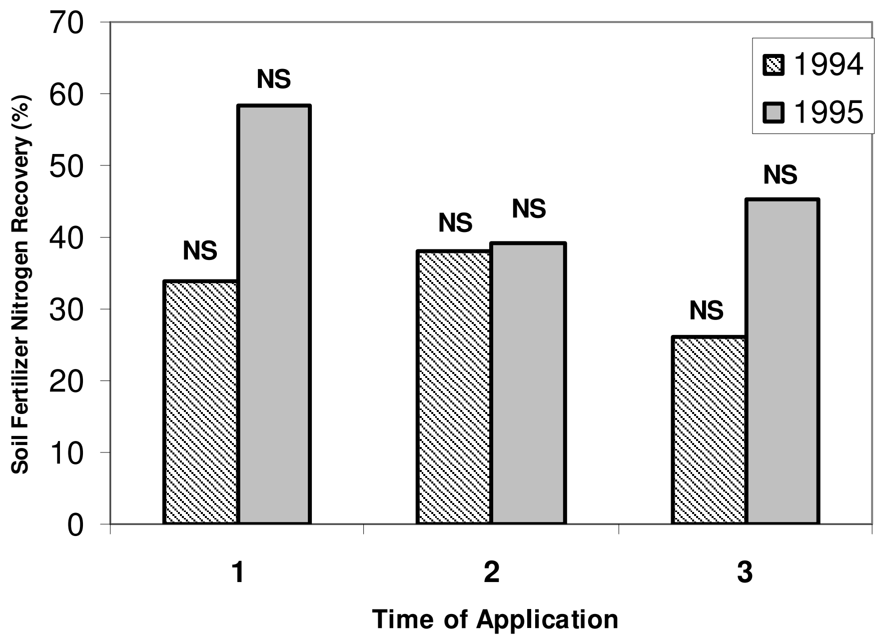

Figure 7. Soil fertilizer N recovery (SFNR) as affected by the time of application, 1994 and 1995. NS indicates differences were not significant among treatments within a year ($P<0.05$).

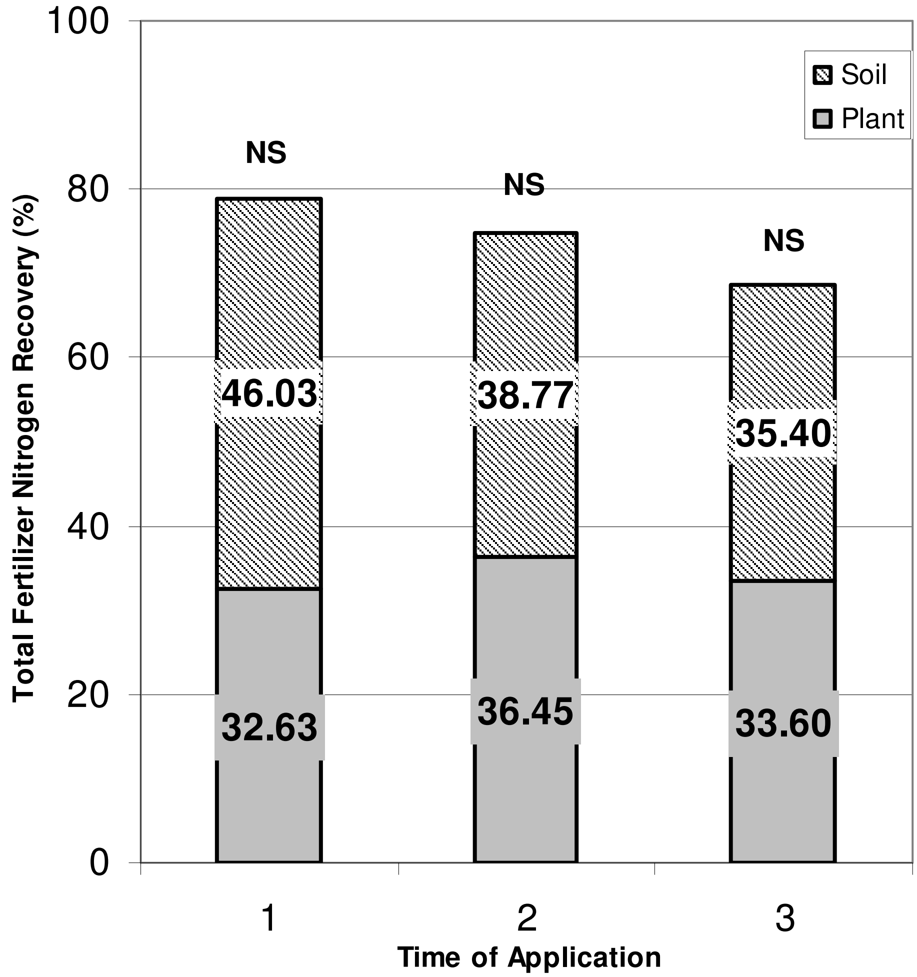

Figure 8. Total fertilizer N recovery and its distribution in the soil and plant. NS indicates total differences were not significant among treatments. Data represent mean values for 1994 and 1995.

APPENDIX B

^{15}N AND BROMIDE DISTRIBUTION PATTERN IN THE SOIL PROFILE AS AFFECTED BY THE TIME OF APPLICATION

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^{15}N and Bromide Distribution Pattern in the Soil Profile as Affected by the Time of Application

Abstract

This study was carried out with the objective to evaluate ^{15}N and Br^- distribution patterns in the soil profile as affected by the time of application in an Upland cotton irrigated system. Microplots were established on a Pima clay loam (fine-silty, mixed, calcareous thermic Typic Torrifluvent) soil, where ^{15}N labeled and unlabeled fertilizer, and Br^- as KBr were applied at three times (establishment, pinhead square to early bloom, and peak bloom stage of growth) during two seasons. Using rates of 168 kg N ha^{-1} and 134 kg Br^- ha^{-1}. In both seasons, the higher fertilizer N recovery was detected in the surface layer (0-30 cm) of the soil with an average of 40% of the fertilizer N recovered. Below a depth of 30 cm, low percentages of N were recovered and even lower at greater depths. High surface recovery rates were apparently related to plant uptake and immobilization processes. Bromide movement in the soil profile appeared to be directly related to the level of exposure to irrigation water. The Br^- applied early was exposed to more irrigation events, and therefore was found deeper in the soil profile compared with late Br^- application times.

Introduction

The fate of fertilizer N applied to agricultural soils is of growing concern due to the potential for leaching and groundwater contamination and health risks associated with high nitrate (NO_3^-) levels in groundwater. In addition to the potential environmental problems associated with NO_3^- leaching, the economic impact to the farmer can be considerable. Therefore, measurement of NO_3^- leaching is important for the evaluation of N fertilizer practices. However, quantitative measurements of NO_3^- leaching losses in the field are very difficult and often inadequate to fully describe the amount of NO_3^- leached.

The anionic tracer Br^- has been reported to be a good physical analog for NO_3^- in leaching studies because it is similar in size and charge to NO_3^-, is not naturally present in the soil in detectable quantities, and behaves physically in a similar fashion (Tillman and Scotter, 1991; Jones and Schwab, 1993). The Br^- background levels in soils are quite low with values commonly ranging from 5 to 40 mg kg^{-1} (Bowen, 1966; Martin, 1966; Tennyson and Settergren, 1980; Maw and Kempton, 1982).

Onken et al. (1975) suggested that the use of Br^- to trace fertilizer NO_3^- should be interpreted only in a qualitative manner if used in the presence of growing plants, due in part to differential uptake of Br^- and N. In relation to Br^- uptake by plants, Jemison and Fox (1991) published some information concerning the uptake of Br^- by corn (*Zea mays* L.), indicating that in a greenhouse study neither Br^- nor chloride (Cl^-) had any significant effect on N uptake, although conversely, applied N did significantly reduce plant Br^- and Cl^- concentrations.

Silvertooth et al. (1992) studying the Br^- and NO_3^- movement in a cotton (*Gossypium hirsutum* L.) production system on a Mohall sandy loam found that in general, the Br^-

concentration patterns tended to peak at depths in the 0.67- to 1.2-m portion of the profile. However, detectable levels of Br^- were measured at 1.8 m in four of six plots on the two later sampling dates (158 and 178 days after planting) that were employed. The NO_3^- levels were lower at these dates compared to the first dates of sampling in the entire soil profile (0-1.80 m); which was probably a function of N uptake and utilization by the crop and the timing of the N fertilizer applications. Considering that Br^- behaves in a similar physical manner to NO_3^-, the relatively low Br^- recoveries for the first two dates of sampling indicate a high degree of leaching potential. The later applications dates showed progressively higher recovery levels, and therefore lower leaching potentials under these conditions.

Many factors affect NO_3^- leaching. These include timing, placement, and source of N fertilizer versus the timing, amount, and uniformity of water applications which affect the degree of preferential flow of water and solute (NO_3^-) movement (Miller et al., 1993).

Tillman et al. (1991) studied the movement of Br^- as a tracer in response to intermittent water flow in the field. Two treatments were applied: treatment one (a 5 mm sprayed application of 77 $mol.m^{-3}$ potassium bromide (KBr) solution followed immediately by the spray application of 50 mm of water in 5 mm pulses every 30 minutes) and treatment two (same as treatment 1, except that prior the KBr application 20 mm of water was sprayed onto the soil surface, again in 5 mm pulses); the treatments were applied for a 24-hour period. Tillman found that wetting soil before Br^- were applied enhanced its movement.

Smith et al. (1995) evaluated the relationship between soil pore size distribution and surface soil compaction on the transport of bromide on undisturbed soil cores. They reported a 93% recovery of Br^- in the effluent and soil. Also, they indicated that Br^- concentrates in pore centers and moves with the most rapidly flowing water; little Br^- may be entering smaller pores or pores with constricted openings, because this anion is repelled from soil surfaces. Contrary to this, Everts et al. (1989) had mentioned previously that preferential flow during an irrigation event appeared to be of less overall significance in the transport of the conservative tracer, Br^-.

The leaching of NO_3^- from N fertilizer applied to no-till corn grown in nonweighing lysimeters was determined for three years after application from point-injection and conventional N management systems. Averaged across three years, annual NO_3^- losses in lysimeter drainage were in the order of PI [(Point-injection) high N rate and split application] > SB (surface banded- 200 kg N ha^{-1}) > KI (knifed-in 200 kg N ha^{-1}) > PI (low N rate and split application). The PI (low/split) management system had the least loss in each year and thus was the most efficient N management system (Baker and Timmons, 1994).

This study evaluated the experimental hypothesis that the distribution patterns of ^{15}N and Br^- in the soil profile will behave differently in an irrigated crop production system due to differences in biological interactions. The objective of this study was to compare how the ^{15}N and Br^- distribution patterns in the soil profile are affected by the time of application in an irrigated upland cotton.

Materials and Methods

A field study was conducted in 1994 and 1995 on a Pima clay loam (fine-silty, mixed, calcareous thermic Typic Torrifluvent) soil located at the University of Arizona Marana Agricultural Center in southern Arizona (600 m elevation). The field was wet planted to 'DPL-20' Upland cotton on 18 April 1994 and on 24 April 1995 in plots eight rows wide with a row spacing of 1 m. The field was furrow irrigated with macroplots extending the full length of the 183 m irrigation run (north-south orientation, head of the field on the north). Planting was accomplished with a conventional (John Deere Maximerge; Deere & Co., Moline, IL) planter.

A set of three microplots was established in each macroplot (which were part of a larger experimental study) area: one at the head, the middle, and the end of the field, each one separated by approximately 48 m (Silvertooth et al., 1997; Silvertooth et al., 1998). The macroplots were set up in the same position each year while the microplots were established within macroplots above (toward the "head" of the field) the previous year's microplots (approximately 5.0 m) and were randomized both years. Microplots for ^{15}N applications were placed in the center four rows of the eight row macroplots (Figure 1). The macroplots were part of a large N study where N rates ranged from 0 to 300 kg N ha^{-1}. The macroplots in this experiment were the 0 N plots and had received no fertilizer N applications for seven years prior to the study. Microplots consisted of four rows 1 m length (4 m^2 total area) (Silvertooth et al., 2001). The ^{15}N labeled fertilizer applications and the three applications of Br^- as KBr (Table 1) were carried out at three stages of crop growth: establishment, between pinhead square (PHS) and early bloom stage, and at peak bloom. The N fertilizer sources were $(^{15}NH_4)_2SO_4$ with 5 atom % ^{15}N and non-labeled $(NH_4)_2SO_4$. The fertilizer N was applied at a rate of 56 kg N ha^{-1} on each application time, with a total amount of 168 kg N ha^{-1} applied to each treatment. Each treatment received three applications of 56 kg N ha^{-1}. Non-labeled $(NH_4)_2SO_4$ was used on the application times where ^{15}N labeled materials were not prescribed (Table 2). Based on preplant soil testing analyses and current UA recommendations no phosphorus (P) or potassium (K) fertilizer were added (Silvertooth et al., 1995; Silvertooth and Norton, 1996).

The fertilizer applications were made by dissolving the appropriate amounts of ^{15}N labeled and/or non-labeled materials in 500 ml of distilled-deionized water. This solution was evenly distributed by hand in a trench 1 m length, approximately 15 cm deep which was cut 15 cm in the west side of the bed simulating a sidedress N application. After the N-solution was applied the trench was immediately covered with soil.

Applications consisted of KBr applied at a rate of 134 kg Br^- ha^{-1} (200 kg KBr ha^{-1}), dissolved in 500 ml of distilled-deionized water. Applications were made by spraying the solution evenly on the soil surface on a 0.5 square meter area in the furrow on both sides immediately adjacent to the row that was treated with ^{15}N labeled fertilizer (Figure 2). To accomplish KBr applications, a CO_2-pressurized sprayer was used with a constant pressure of 10 psi. During 1994 a total of five in-season irrigations were applied with approximately 130 mm of water per irrigation for a total of 650 mm of water. In 1995 three in-season irrigations were applied for a total de 390 mm of water (Table 3 and 4). The lower irrigation numbers in 1995 were due to the additional summer rains received during the growing season at critical times in the growth cycle. The irrigation water used in this study has an average of 5 ppm NO_3^--N that corresponded to about 30 and 20 kg NO_3^--N ha^{-1}, considering the 650 and 390 mm application rates based on the amount of water applied to the crop in 1994 and 1995 (Silvertooth and Doerge, 1990). The rainfall recorded during the growing season was 133.1 mm during 1994 and 138.17 mm for 1995 (Table 3). Irrigation termination dates were on 28 August 1994 and 1 August 1995. All cultivation, irrigation, and pest management practices were conducted in a uniform manner on an as-needed basis.

Harvest was accomplished by hand picking all seedcotton (lint+seed) from the plants located in the central 0.5 m of the two middle rows of each microplot, with a total sample area of 1 m^2 (Figure 3). Stover samples were obtained by clipping plants at the soil surface and collecting the entire aboveground plant material, including leaves found on the surface of the ground in the sample area. All plant material was dried with a forced-air oven at a temperature of 65°C for 48 hours or until a constant weight was obtained. Dry weights were obtained for each of the plant portions. After drying, the samples were chopped and ground first with a hammer mill (1.4 mm mesh screen) and then with a Wiley mill to pass a 1 mm mesh screen. The seed was separated from the lint by use of a small gin and then ground with a Wiley mill to pass a 1 mm mesh screen. A subsample of the stover and the seed were further ground to a very fine powder for laboratory analysis.

At the end of each season single soil cores were obtained in the seed row line from each microplot using a 5 cm diameter auger from a central row. Soil samples were obtained on 31 October 1994 and on 23 October 1995, after harvest and before disking the soil. The sample location was in the center of the row, 0.5 m from the edge. Soil samples were collected at 30 cm intervals to a depth of 180 cm (Figure 4). Soil samples were air-dried and ground to pass a 1 mm mesh screen. Following thorough homogenation a subsample from each soil sample was obtained and further ground to a very fine powder for laboratory analysis. Determinations of ^{15}N content were made by isotope ratio analyses for each sample (plant and soil). Total N and isotope ratio analyses were carried out for all soil, stover and seed samples by use of a Carlo Erba N/A 1500 Elemental Analyzer (CE Elantech, Inc., Lakewood, NJ) and a VG Isomass Mass Spectrometer (Isomass Scientific, Inc., Calgary, AB). For Br^- analysis, soil samples were subjected to a 30 minute 1:1 (soil/distilled-deionized water) extraction. The extract was then analyzed for Br^- concentration by an ion chromatograph (Dionex 22001, Dionex Corp., Sunnyvale, CA).

Fertilizer N recovery (FNR) was calculated using the following equation (Hauck and Bremner, 1976):

$$FNR = P(c-b)/f(a-b)$$

where *P* is the total N in plants or soil (kg ha^{-1}); *f* is the amount of labeled N fertilizer applied (kg ha^{-1}); and *a*, *b*, and *c* are atom percentages of ^{15}N in fertilizer (*a*), in control plants or soil with non-labeled fertilizer (*b*), and in plants or soil with labeled fertilizer (*c*).

Crop development was monitored in terms of plant height, mainstem node number, and fruit retention in relation to heat units accumulated after planting (HUAP) using 30/13°C thresholds (Brown, 1989; Silvertooth et al., 1995). Petiole samples from the youngest fully expanded leaves (approximately four to five nodes below the terminal) were taken on biweekly intervals throughout the growing season up to cut-out (end of primary fruiting cycle), and analyzed for NO_3^--N concentrations to monitor the in-season N status of the crop in the study area (Silvertooth and Doerge, 1990). The analyses were performed using a NO_3^--N specific electrode (Keeney and Nelson, 1982). All statistical analyses were conducted according to procedures outlined by SAS (SAS Institute, 1994) and Steel and Torrie (1980).

Results and Discussion

The fertilizer N recovery in the soil profile (0-180 cm depth) for the three ^{15}N application times during the 1994 and 1995 seasons are shown in Figures 5 and 6. In both years, higher fertilizer N recovery was observed in the top surface layer (0-30 cm depth) with an average value close to 30% of the total fertilizer N recovered in 1994 and 50% in 1995. This also corresponds to a higher fertilizer N recovery by the plant in 1994 (38.09%). In 1995 a lower percentage of fertilizer N was recovered by the plants (30.37%) and a corresponding higher fertilizer N recovery values were found for the soil. The amount of fertilizer N recovery at the 30-60 cm soil layer was rather low (7-8% in both seasons) and even lower below that depth. Low fertilizer N recovery levels at the deepest soil layers would indicate minimal leaching of fertilizer N under these conditions. Finally, the fertilizer N recovery values showed some variation among treatments which could be attributed to the natural variation (heterogeneity) that exists in the soil and/or soil-plant interactions.

The high proportions of fertilizer N recovered in the soil (an average of 40% for both seasons) at the surface soil layer is important to note, it is possible that this fertilizer N was retained by the clay particles in the soil or immobilized. Saoud et al. (1992) working on potatoes established on a clayey soil, found a considerable quantity of applied fertilizer N in the top 10 cm of the soil which was attributed to various factors including the immobilization of fertilizer N, which is somewhat similar to what we have found in our study. High fertilizer N recovery in the surface could also be attributed to immobilization of the labeled fertilizer N. Reddy and Reddy (1993) found that approximately one-half of the residual fertilizer N was recovered from the surface 0-15 cm of the soil in a corn production system, with most of this N immobilized in the organic fraction, resulting in minimal downward movement of inorganic N forms.

The mass balance for the ^{15}N found in the soil shows no significant differences among the times of application (Table 5). The lack of significance could be due to the fact that the application times were similar because the N uptake patterns matched the maximum or optimal uptake periods of the cotton plants.

The Br^- distribution in the soil profile for 1994 and 1995 (Figure 7 and 8) exhibited a distribution pattern related to the Br^- application time. As the Br^- application was delayed more Br^- was recovered from the surface soil layers and less Br^- recovered from deepest soil layers. There were differences between seasons in the amount of Br^- recovered. However, in both seasons a higher amount of Br^- was recovered with the late application times at the surface soil layers and lower amounts for the early Br^- application times. Bromide is a conservative tracer and moves with the water in the soil profile, similar to piston flow. Greater Br^- movement appears to be a direct function of exposure to irrigation water. The amount of Br^- recovered, across application times and all profile depths was 16.63% for 1994 with a total irrigation amount of 650 mm, while for 1995 the average Br^- recovered in the soil profile was 32.49% with a total amount of irrigation of 390 mm. The amount of rainfall was very similar between years, with an average of 135.6 mm. Mass-balance estimates for Br^- in the soil for each of the application times are shown in Table 6. There was not significant differences among the different times of application. The overall average Br^- recovery was 13.5 %. The ^{15}N recovery was far greater than Br^- recovery showing that the use of ^{15}N is a good way to trace N in the soil; however, Br^- is good to be used for estimating the leaching potential.

Conclusions

This results indicate Br^- could be used to evaluate the worst-case scenario for NO_3^- leaching. Bromide is not a good N analog because N is subject to uptake and many interactions (organic and inorganic) in the soil. Thus, Br^- serves as a good estimate for anionic (including NO_3^-) leaching potential.

It would be interesting to conduct this study on a coarser textured soil. We would expect faster NO_3^- and Br^- movement than on a finer textured soil (as used in this study). In any case, these results suggest that it is appropriate to split N applications in order to reduce the amount of NO_3^- leaching potential. The evaluation of other ^{15}N and Br^- application at times in relation to irrigations events and also other methods of application would be additional areas of possible study in the future.

References

Baker, J.L. and D.R. Timmons. 1994. Fertilizer management effects on leaching of labeled nitrogen for no-till corn in field lysimeters. J. Environ. Qual. 23:305-310.

Bowen, H.J.M. 1966. Trace elements in biochemistry. Academic Press. New York.

Brown, P.W. 1989. Heat units. College of Agric. Rep. 8915. Univ. of Arizona, Tucson.

Everts, C.J., R.S. Kanwar, E.C. Alexander Jr., and S.C. Alexander. 1989. Comparison of tracer mobilities under laboratory and field conditions. J. Environ. Qual. 18:491- 498.

Hauck, R.D. and J.M. Bremner. 1976. Use of tracers for soil and fertilizer nitrogen research. Adv. Agron. 28:219-266.

Jemison, Jr. J.M. and R.H. Fox. 1991. Corn uptake of bromide under greenhouse and field conditions. Commun. in Soil Sci. and Plant Anal. 22:283-297.

Jones, R.D. and A.P. Schwab. 1993. Nitrate leaching and nitrite occurrence in a fine-textured soil. Soil Sci. 155:272-282.

Keeney, D.R. and D.W. Nelson. 1982. Nitrogen-inorganic forms. p. 643-698. In A.L. Page, R.H. Miller, and D.R. Keeney (eds), Methods of soil analysis, Part 2. Chemical and microbiological properties-Agronomy Monograph No. 9 (2nd Edition). ASA-SSSA, Madison, Wisconsin.

Martin, J.P. 1966. Bromide. p. 62-64. In H.D. Chapman (ed) Diagnostic criteria for plants and soils.University of California Press, Berkeley, CA.

Maw, G.A. and R.A. Kempton. 1982. Bromine in soils and peats. Plant Soil 65:103-109

Miller, G.D., R.J. Hanks, J.C. Andersen, and G.R. Erikson. 1993. Profits and nitrate leaching under irrigated corn: a simulation. Irrig. Sci. 13:165-170.

Onken, A.B., R.S. Hargrove, C.W. Wendt, and O.C. Wilke. 1975. The use of a specific ion electrode for determination of bromide in soil. Soil. Sci. Soc. Am. J. 39: 1223-1224.

Reddy, G.B. and K.R. Reddy. 1993. Fate of nitrogen –15 enriched ammonium nitrate applied to corn. Soil Sci. Soc. Am. J. 57:111-115.

Saoud, A.A., O. Van Cleemput, and G. Hofman. 1992. Uptake and balance of labeled fertilizer nitrogen by potatoes. Fert. Res. 31: 351-353.

SAS Institute. 1994. The SAS system for Windows. Release 6.10. SAS Inst., Cary, N.C.

Silvertooth, J.C., and T.A. Doerge. 1990. Nitrogen management in Arizona cotton production. College of Agric. Rep. 9024. Univ. of Arizona, Tucson.

Silvertooth, J.C., J.E. Watson, J.E. Malcuit, and T.A. Doerge. 1992. Bromide and nitrate movement in an irrigated cotton production system. Soil Sci. Soc. Am. J. 56:548-555.

Silvertooth, J.C., E.R. Norton, B.L. Unruh, J.C. Navarro, L.J. Clark and E.W. Carpenter 1995. Nitrogen management experiments for Upland and Pima cotton, 1994. Cotton, A College of Agriculture Report. University of Arizona. Series P-99:311-326

Silvertooth, J.C., and E.R. Norton. 1996. Nitrogen management experiments for Upland and Pima cotton, 1995. Cotton, A College of Agriculture Report. University of Arizona. Series P-103:317-332

Silvertooth, J.C., E.R. Norton, C.A. Sánchez, J.C. Navarro, and A. Galadima. 1997. Evaluation of a nitrogen–15 microplot design in a furrow irrigated row crop system. 89th annual meeting, Am. Soc. of Agron., Anaheim, CA. Agron. Abstr. p. 241

Silvertooth, J.C., E.R. Norton, J.C. Navarro, A. Galadima, and C.A. Sánchez. 1998. Field plot design evaluation for nitrogen-15 labeled fertilizers in a furrow irrigated system. Proceedings of the Beltwide Cotton Conferences. San Diego, CA.

Silvertooth, J.C., J.C. Navarro, E.R. Norton, and C.A. Sanchez. 2001. Evaluation of a nitrogen-15 microplot design in furrow-irrigated cotton. Soil Sci. Soc. Am. J. 65: 247-250

Smith, E.A., W.L. Powers, and P.J. Shea. 1995. Relationship of bromide and atrazine movement in soil to pore size distribution, compaction, and saturation cycles. Soil Sci. 159:23-31.

Steel, R. and J. Torrie. 1980. Principles and procedures of statistics, a biometrial approach. McGraw-Hill, Inc. San Francisco, CA.

Tennyson, L.C. and C.D. Settergren. 1980. Percolate water and bromide movement in the root zone of effluent irrigation sites. Water Resour. Bull. 16: 433-437

Tillman, R.W. and D.R. Scotter. 1991. Movement of solutes associated with intermittent soil water flow. 11. Nitrogen and cations. Aust. J. Soil Res. 29 :185-196

Tillman, R.W., D.R. Scotter, B.E. Clothier, and R.E. White. 1991. Solute movement during intermittent water flow in a field soil and some implications for irrigation and fertilizer applications. Agric. Water Management 20:119-133.

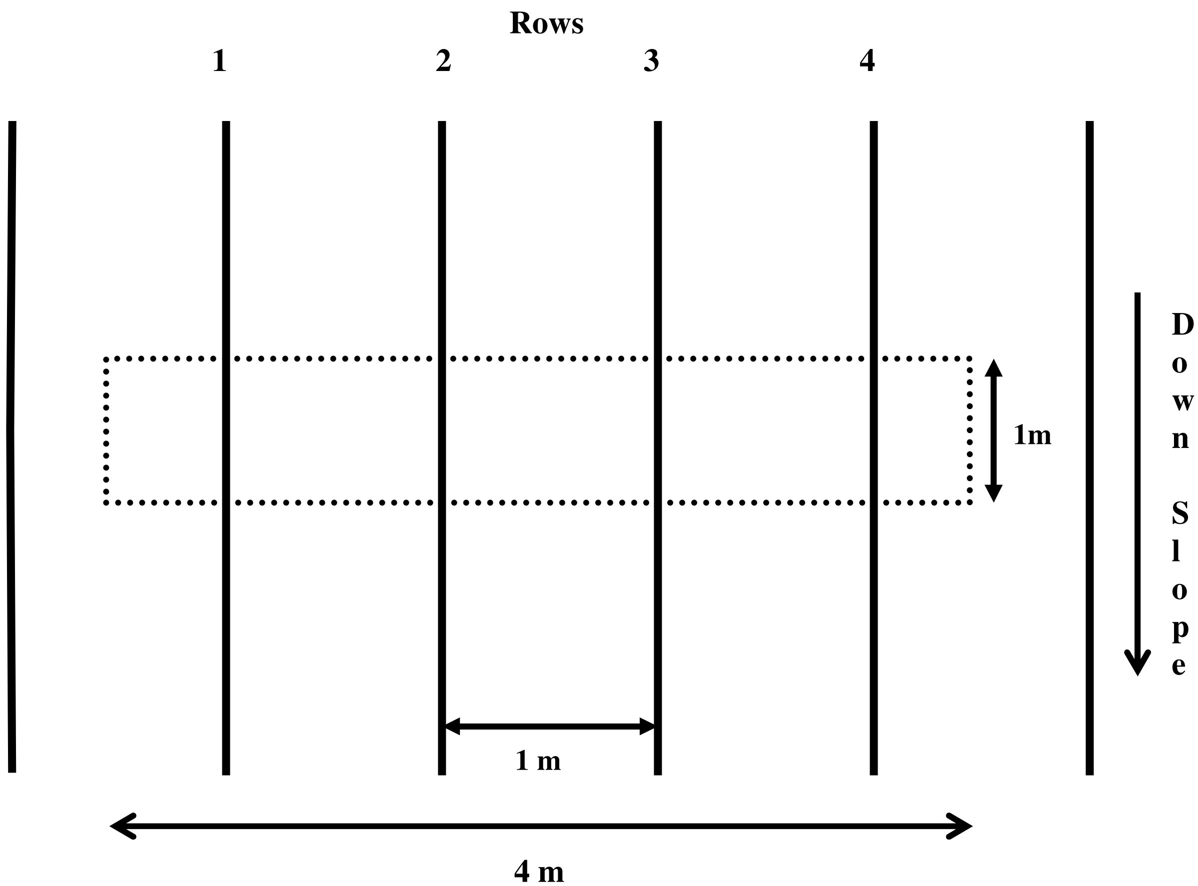

Figure 1. ^{15}N plot structure for 1994 and 1995.

Table 1. Description of the nitrogen and bromide treatments under study on Upland cotton, 1994 and 1995.

Application Time	Crop Growth Stage	HUAP[1]
Early	Establishment (vegetative growth stage)	≈400
Intermediate	PHS-Early Bloom	≈1,000
Late	Peak Bloom	≈2,000

[1]HUAP = Heat Units After Planting (30°/13°C thresholds)

Table 2. Application times and amount of labeled and unlabeled N applied to Upland cotton, 1994 and 1995.

Treatments	------ N Application Time ------		
	1	2	3
Early	56*	56	56
Intermediate	56	56*	56
Late	56	56	56*

*^{15}Nlabeled fertilizer (kg ha^{-1})

Table 3. Dates of irrigation and amount of rainfall on each of the treatments on Upland cotton, 1994 and 1995.

Treatments	Date of irrigation	Rainfall† (mm)
	1994	
1	18 June, 2 July, 26 July, August 10, August 28	133.10
2	2 July, 26 July, August 10, August 28	133.10
3	26 July, August 10, August 28	133.10
	1995	
1	June 25, 7 July, August 1	138.17
2	7 July, August 1	138.17
3	----------	135.12

†Rainfall registered during the entire growing season

Table 4. Dates and amount of irrigation and rainfall applied to the study area on Upland cotton, 1994 and 1995.

Irrigation	Irrigation Date	Irrigation Amount (mm)	Rainfall (mm)
	1994		
1	18 June	130.00	0.00
2	2 July	130.00	0.00
3	26 July	130.00	8.89
4	10 August	130.00	0.00
5	28 August	130.00	74.17
Total		650.00	83.06
	1995		
1	25 June	130.00	0.00
2	7 July	130.00	3.05
3	1 August	130.00	122.17
Total		390.00	125.22

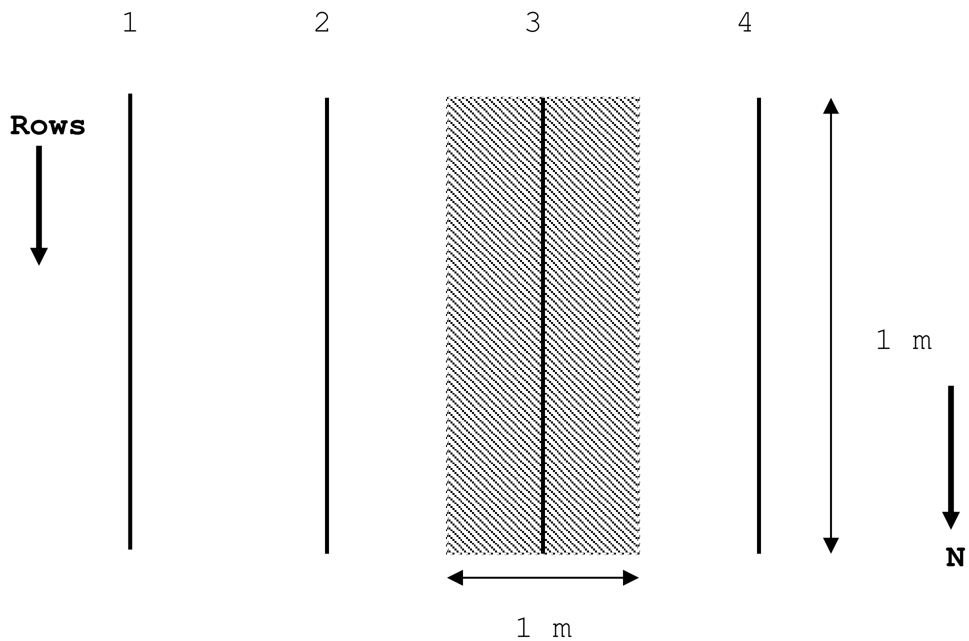

Figure 2. Bromide application area in a seed row in the microplot for 1994 and 1995.

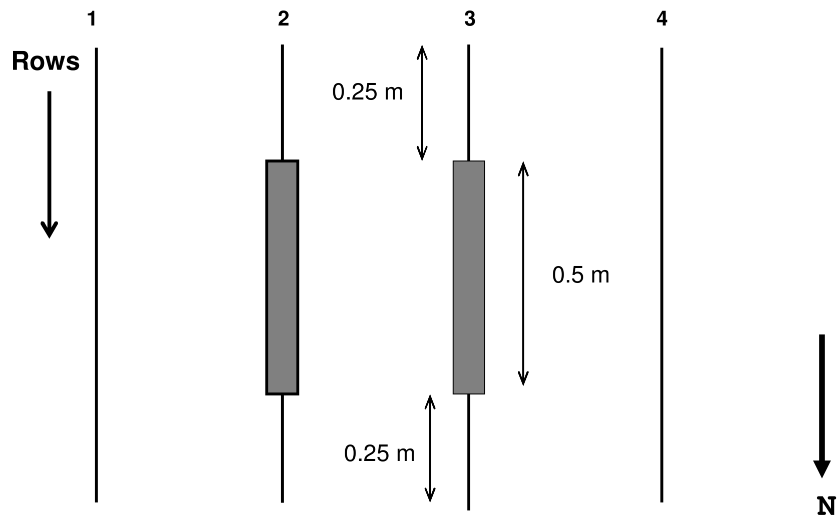

Figure 3. Plant and seedcotton harvesting scheme within each microplot area.

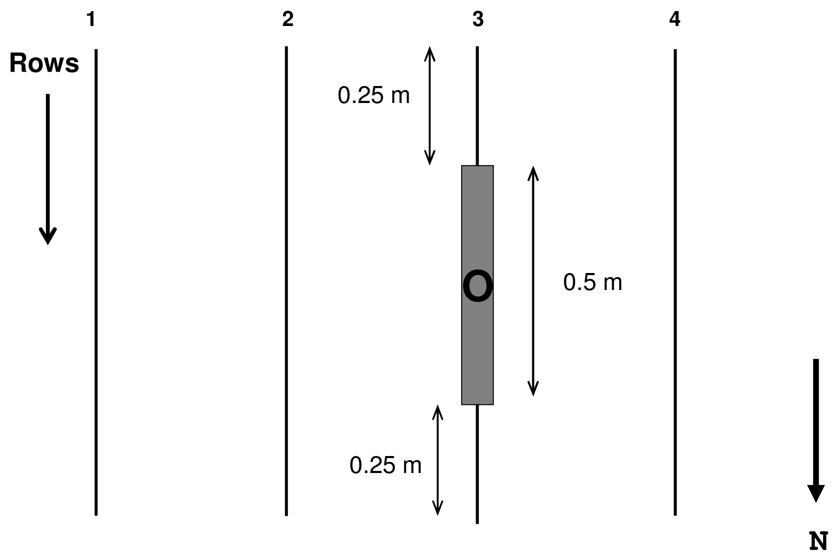

Figure 4. Soil sampling (in the center of a seed row) in the microplot area each season.

Table 5. Mass-balance estimates for ^{15}N in the soil profile at three different times of application on Upland cotton, 1994 and 1995.

	-----^{15}N Soil Recovery (%)-----		
Application Time	1994	1995	Average
Early	41.4 a	71.8 a	56.6 a
Intermediate	60.8 a	49.8 a	55.3 a
Late	33.3 a	56.0 a	44.7 a
C.V. (%)	36.78	33.50	25.19
OSL*	0.2730	0.4150	0.3760

*OSL= Observed Significance Level

Table 6. Mass-balance estimates for Br^- in soil at three different times of application on Upland cotton, 1994 and 1995.

Application Time	-----Br- Soil Recovery (%)----- 1994	1995	Average
Early	11.08 a	11.38 a	11.23 a
Intermediate	15.88 a	10.22 a	16.16 a
Late	25.98 a	10.92 a	18.45 a
C.V. (%)	89.80	26.54	54.51
OSL*	0.9221	0.1657	0.7855

*OSL= Observed Significance Level

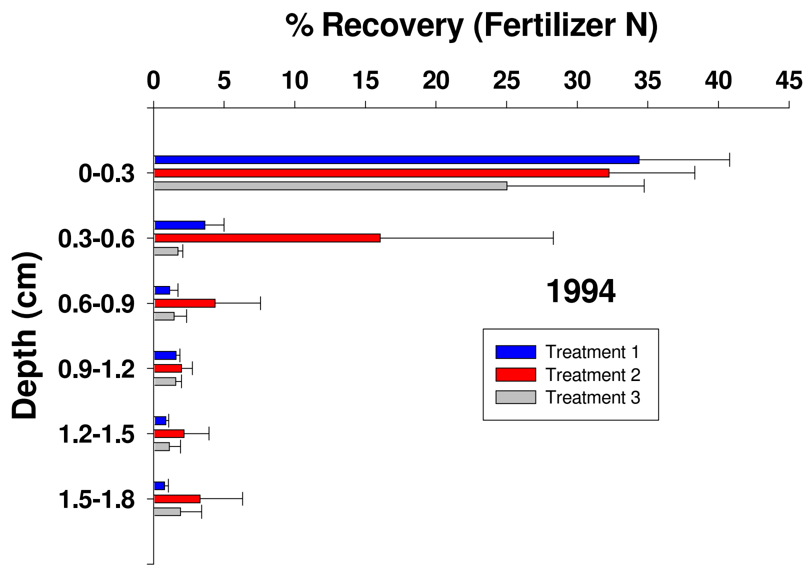

Figure 5. Fertilizer N recovery in the soil profile by depth among treatments for 1994.

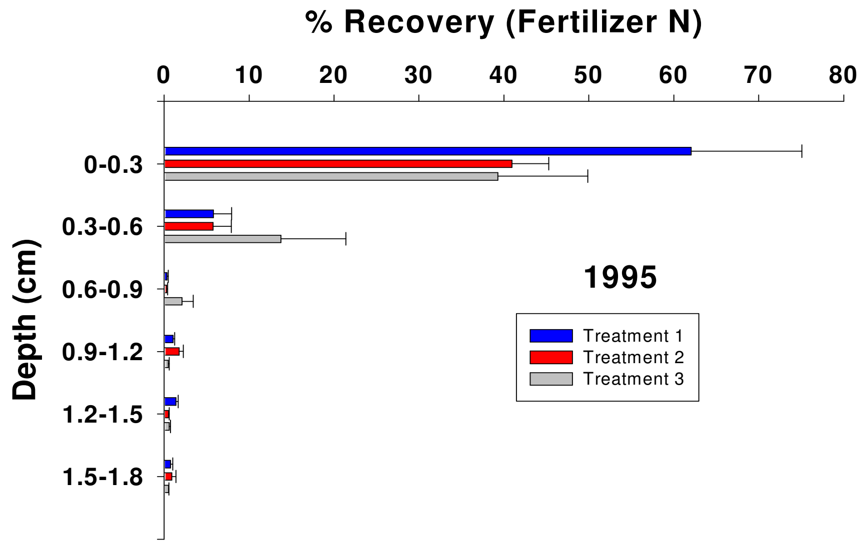

Figure 6. Fertilizer N recovery in the soil profile by depth among treatments for 1995.

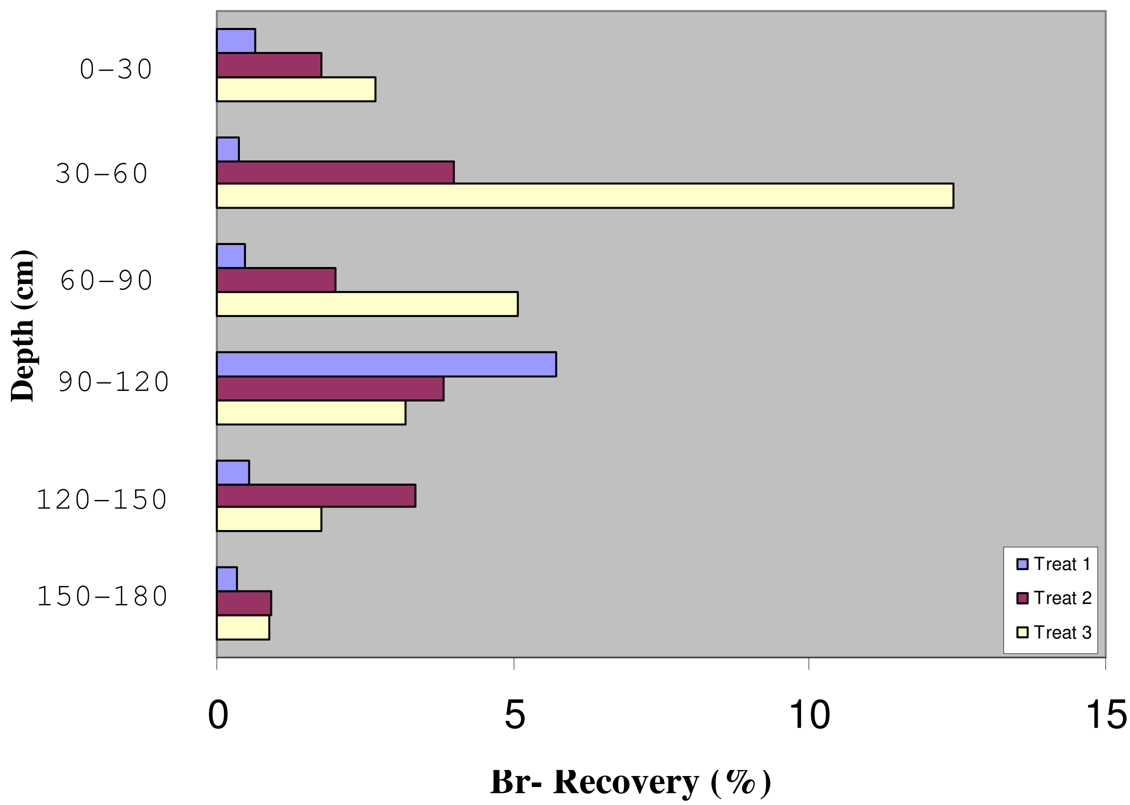

Figure 7. Bromide recovery in the soil profile by depth among treatments for 1994.

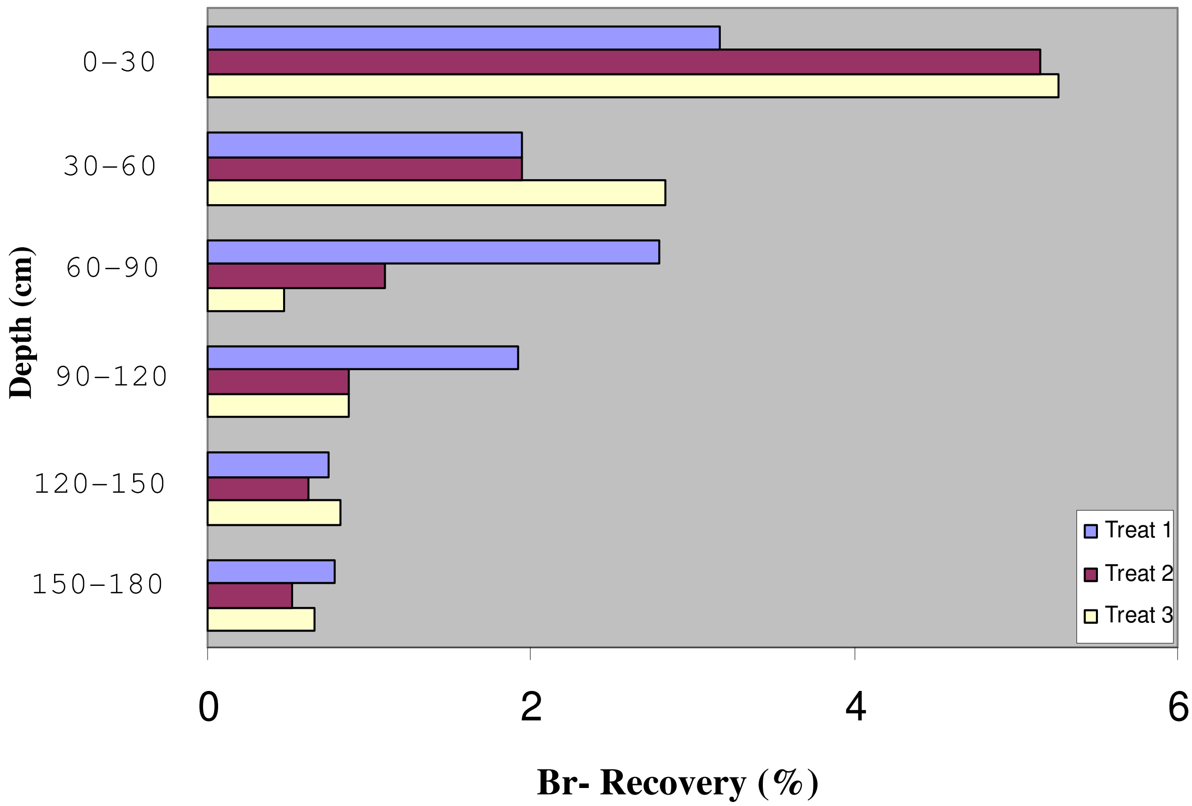

Figure 8. Bromide recovery in the soil profile by depth among treatments for 1995.

REFERENCES

Agus, F. and D.K. Cassel. 1992. Field-scale bromide transport as affected by tillage. Soil Sci. Soc. Am. J. 56:254-260.

Alcoz, M.M., F.M. Honz, and V.A. Haby. 1993. Nitrogen fertilization timing effect on wheat production, nitrogen uptake efficiency, and residual soil nitrogen. Agron. J. 85:1198-1203.

Aldrich, S.R. 1984. Nitrogen management to minimize adverse effects on the environment. p. 663-673. In R.D. Hauck (ed) Nitrogen in crop production. ASA, CSSA, SSSA.

Bowen, H.J.M. 1966. Trace elements in biochemistry. Academic Press. New York.

Bowman, R.S. 1984. Evaluation of some new tracers for soil water studies. Soil Sci. Soc. Am. J. 48:987-993.

Bremner, J.M. and R.D. Hauck. 1982. Advances in methodology for research on nitrogen transformations in soils. p. 467-502. In F.J. Stevenson (ed) Nitrogen in agricultural soils. ASA, CSSA, and SSSA, Madison, Wisconsin.

Bronson, K.F., J.T. Touchton, R.D. Hauck, and K.R. Kelley. 1991. Nitrogen-15 recovery in winter wheat as affected by application timing and dicyandiamide. Soil Sci. Soc. Am. J. 55:130-135.

Buresh, R.J., E.R. Austin, and E.T. Craswell. 1982. Analytical methods in 15N research. Fert. Res. 3:37-62.

Constable, G.A. and I.J. Rochester. 1988. Nitrogen application to cotton on clay soil: timing and soil testing. Agron. J. 80:498-502.

Everts, C.J., R.S. Kanwar, E.C. Alexander Jr., and S.C. Alexander. 1989. Comparison of tracer mobilities under laboratory and field conditions. J. Environ. Qual. 18: 491- 498.

Fleming, J.B., and G.L. Butters. 1995. Bromide transport detection in tilled and nontilled soil: solution samplers vs. soil cores. Soil Sci. Soc. Am. J. 59:1207-1216.

Freney, J.R., D.L. Chen, A.R. Mosier, I.J. Rochester, G.A. Constable, and P.M. Chalk. 1993. Use of nitrification inhibitors to increase fertilizer nitrogen recovery and lint yield in irrigated cotton. Fert. Res. 34:37-44.

Guindo, D., R.J. Norman, and B.R. Wells. 1994. Accumulation of fertilizer nitrogen-15 by rice at different stages of development. Soil Sci. Soc. Am. J. 58:410-415

Hagin, J. and B. Tucker. 1982. Fertilization of dryland and irrigated soils. Springer-Verlag, Berlin, Germany.

Harris, G.H. and O.B. Hesterman. 1990. Quantifying the nitrogen contribution from alfalfa to soil and two succeeding crops using nitrogen-15. Agron. J. 82:129-134.

Hauck, R.D. and J.M. Bremner. 1976. Use of tracers for soil and fertilizer nitrogen research. Adv. Agron. 28:219-266.

Hauck, R.D. 1982. Nitrogen-isotope-ratio analysis. P. 735-780. In A.L. Page, R.H. Miller, and D.R. Keeney (eds) Methods of soil analysis, Part 2. Chemical and microbiological properties-Agronomy Monograph No. 9 (2nd edition). ASA-SSSA, Madison, Wisconsin.

Humphreys, E., P.M. Chalk, W.A. Muirhead, F.M. Melhuish, and R.J.G. White.1987. Effects of time of urea application on combine-sown calrose rice in south-east australia. 111. Fertilizer nitrogen recovery, efficiency of fertilization and soil nitrogen supply. Aust. J. Agric. Res. 38:113-127.

Jabro, J.D., E.G. Lotse, K.E. Simmons, and D.E. Baker. 1991. A field study of macropore flow under saturated conditions using a bromide tracer. J. of Soil and Water Conserv. 46:376-380.

Jackson, L.E., L.J. Wyland, J.A. Klein, R.F. Smith, W.E. Chaney and S.T. Koike. 1993. Winter cover crops can decrease soil nitrate leaching potential. Calif. Agric. 47(5):12-15

Jemison, Jr. J.M. and R.H. Fox. 1991. Corn uptake of bromide under greenhouse and field conditions. Commun. in Soil Sci. and Plant Anal. 22:283-297.

Jenkinson, D.S., R.H. Fox, and J.H. Rayner. 1985. Interactions between fertilizer nitrogen and soil nitrogen-the so called 'priming' effect. J. Soil Sci. 36:425-444

Jensen, E.S. 1994. Leaching in small lysimeters of nitrate derived from nitrogen-15 labeled field pea residues. J. Environ. Qual. 23:1045-1050.

Jones, R.D. and A.P. Schwab. 1993. Nitrate leaching and nitrite occurrence in a fine-textured soil. Soil Sci. 155:272-282.

Katyal, J.C., B. Singh, P.L.G. Vlek, and R.J. Buresh. 1987. Efficient nitrogen use as affected by urea application and irrigation sequence. Soil Sci. Soc. Am. J. 51:366-370.

Levanon, D., E.E. Codling, J.J. Meisinger, and J.L. Starr. 1993. Mobility of agrochemicals through soil from two tillage systems. J. Environ. Qual. 22:155-161.

Ma, B.L., M.L. Dwyer, B.C. Liang, and E. Gregorich. 1995. Use of nitrogen –15 microplots for field studies of maize nitrogen –use efficiency. Commun. Soil Sci. and Plant Anal. 26:1813-1822.

Martin, J.P. 1966. Bromide. p. 62-64. In H.D. Chapman (ed) Diagnostic criteria for plants and soils.University of California Press, Berkeley, CA.

Mascagni, Jr. H.J., and R.S. Helms. 1989. Effect of nitrogen rate, nitrogen timing, and nitrification inhibitors on grain sorghum production in Arkansas. Commun. Soil Sci. and Plant Anal. 20:2117-2136.

Maw, G.A. and R.A. Kempton. 1982. Bromine in soils and peats. Plant Soil 65:103-109.

Miller, G.D., R.J. Hanks, J.C. Andersen, and G.R. Erikson. 1993. Profits and nitrate leaching under irrigated corn: a simulation. Irrig. Sci. 13:165-170.

Norman, R.J., B.R. Wells, and K.A.K. Moldenhauer. 1989. Effect of application method and dicyandiamide on urea-nitrogen-15 recovery in rice. Soil Sci. Soc. Am. J. 53:1269-1274.

Olson, R.A. and L.T. Kurtz. 1982. Crop nitrogen requirements, utilization, and fertilization. p. 567-604. In F.J. Stevenson (ed) Nitrogen in agricultural soils. Agronomy Monograph no. 22. ASA, CSSA, SSSA.

Onken, A.B., R.S. Hargrove, C.W. Wendt, and O.C. Wilke. 1975. The use of a specific ion electrode for determination of bromide in soil. Soil. Sci. Soc. Am. Proc. 39: 1223-1224.

Owens, L.B., R.W. Van Keuren, and W.M. Edwards. 1985. Groundwater quality changes resulting from a surface bromide application to a pasture. J. Environ. Qual. 14: 543-548.

Pessarakli, M. and T.C. Tucker. 1985. Uptake of nitrogen –15 by cotton under salt stress. Soil Sci. Soc. Am. J. 49:149-152.

Power, J.F. and J.O. Legg. 1984. Nitrogen –15 recovery for five years after application of ammonium nitrate to crested wheatgrass. Soil Sci. Soc. Am. J. 48:322-326.

Ramos, C. 1988. The use of chloride balance method for estimating nitrate leaching. p. 256-268. In D.S. Jenkinson and K.A. Smith (ed) Nitrogen efficiency in agricultural soils. Elsevier Science Publ., New York ,NY.

Rao, A.C.S., J.L. Smith, R,I. Papendick, and J.F. Parr.1991. Influence of added nitrogen interaction in estimating recovery efficiency of labeled nitrogen. Soil Sci. Soc. Am. J. 55:1616-1621.

Rathier, T.M. and C.R. Frink. 1986. Efficiency of nitrogen fertilizer us by shade tobacco improved by timed applications. Agron. J. 78:459-464.

Reddy, G.B. and K.R. Reddy. 1993. Fate of nitrogen –15 enriched ammonium nitrate applied to corn. Soil Sci. Soc. Am. J. 57:111-115.

Reeves, D.W. and J.T. Touchton. 1989. Effect of dicyandiamide on growth and nutrient uptake of cotton. Commun. Soil Sci. and Plant Anal. 20:2091-2103.

Rice, R.C., R.S. Bowman, and D.B. Jaynes. 1986. Percolation of water below an irrigated field. Soil Sci. Soc. Am. J. 50:855-859.

Rice, R.C., D.B. Jaynes, and R.S. Bowman. 1991. Preferential flow of solutes and herbicide under irrigated fields. Trans. of the ASAE 34:914-918.

Richter, G. and W.A. Jury. 1986. A microlysimeter field study of solute transport through a structured sandy loam soil. Soil Sci. Soc. Am. J. 50:863-868.

Roberts, T.L. and H.H. Janzen. 1990. Comparison of direct and indirect methods of measuring fertilizer N uptake in winter wheat. Can. J. Soil Sci. 70:119-124.

Sánchez, C.A. and A.M. Blackmer. 1988. Recovery of anydrous ammonia-derived nitrogen –15 during three years of corn production in Iowa. Agron. J. 80:102-108.

Saoud, A.A., O. Van Cleemput, and G. Hofman. 1992. Uptake and balance of labeled fertilizer nitrogen by potatoes. Fert. Res. 31: 351-353.

Schnabel, R.R. 1983. Measuring nitrogen leaching with ion exchange resin: a laboratory assessment. Soil Sci. Soc. Am. J. 47:1041-1042.

Schnabel, R.R., S.R. Messier, and R.F. Purnell. 1993. An evaluation of anion exchange resin used to measure nitrate movement through soil. Commun. Soil Sci. and Plant Anal. 24:863-879.

Schnier, H.F., S.K. de Datta, K. Mengel, E.P. Marqueses, and J.E. Faronilo. 1988. Nitrogen use efficiency, floodwater properties, and nitrogen-15 balance in transplanted lowland rice as affected by liquid urea band placement. Fert. Res. 16: 241-255.

Scotter, D.R., I.H. Mohammed, and P.E.H. Gregg. 1984. The short term fate of urea applied to barley in a humid climate. II. A simple model. Aust. J. Soil Res. 22:181-190.

Scotter, D.R. and R.W. Tillman. 1991. Movement of solutes associated with intermittent soil water flow. 1. Tritium and bromide. Aust. J. Soil Res. 29:175-183.

Shaffer, K.A., D.D. Fritton, and D.E. Baker. 1979. Drainage water sampling in a wet, dual-pore soil system. J. Environ. Qual. 8:241-246

Sharpe, R.R., L.A. Harper, J.E. Giddens, and G.W. Langdale. 1988. Nitrogen use efficiency and nitrogen budget for conservation tilled wheat. Soil Sci. Soc. Am. J. 52:1394-1398.

Shipitalo, M.J. and W.M. Edwards. 1993. Seasonal patterns of water and chemical movement in tilled and no-till column lysimeters. Soil Sci. Soc. Am. J. 57: 218-223.

Silvertooth, J.C., J.E. Watson, J.E. Malcuit, and T.A. Doerge. 1992. Bromide and nitrate movement in an irrigated cotton production system. Soil Sci. Soc. Am. J. 56: 548-555.

Smith, E.A., W.L. Powers, and P.J. Shea. 1995. Relationship of bromide and atrazine movement in soil to pore size distribution, compaction, and saturation cycles. Soil Sci. 159:23-31.

Stevenson, F.J. 1982. Origin and distribution of nitrogen in soil. p. 1-42. In F.J. Stevenson (ed) Nitrogen in agricultural soils. Agronomy Monograph no. 22. ASA, CSSA, SSSA.

Stevenson, F.J. and M.A. Cole. 1999. Cycles of soil. John Wiley & Sons, Inc. Second Edition. New York, N.Y. p. 139-190

Stout, W.L. 1995. Evaluating the “added nitrogen interaction” effect in forage grasses. Commun. Soil Sci. and Plant Anal. 26:2829-2841.

Sutherland, R.A., C. van Kessel, R.E. Farrel, and D.J. Pennock. 1993. Landscape-scale variation in plant and soil nitrogen-15 natural abundance. Soil Sci. Soc. Am. J. 57:169-178.

Tennyson, L.C. and C.D. Settergren. 1980. Percolate water and bromide movement in the root zone of effluent irrigation sites. Water Resour. Bull. 16:433-437.

Tillman, R.W. and D.R. Scotter. 1991. Movement of solutes associated with intermittent soil water flow. 11. Nitrogen and cations. Aust. J. Soil Res. 29 :185-196

Tillman, R.W., D.R. Scotter, B.E. Clothier, and R.E. White. 1991. Solute movement during intermittent water flow in a field soil and some implications for irrigation and fertilizer applications. Agric. Water Manage. 20:119-133.

Timmons, D.R. and R.M. Cruse. 1990. Effect of fertilization method and tillage on nitrogen-15 recovery by corn. Agron. J. 82:777-784

Timmons, D.R. and J.L. Baker 1992. Fertilizer management effect on recovery of labeled nitrogen by continuous no-till. Agron. J. 84:490-496.

Torbert, H.A. and C.B. Elkins. 1992. Determining differential water movement through ion exchange resin for nitrate leaching measurements. Commun. Soil Sci. and Plant Anal. 23:1043-1052.

Varvel, G.E. and T.A. Peterson. 1990. Nitrogen fertilizer recovery by corn in monoculture and rotation systems. Agron. J. 82:935-938.

Wagner, G.H. 1962. Use of porous ceramic cups to sample water within the profile. Soil Sci. 94:379-386.

Walters, D.T. and G.L. Malzer. 1990a. Nitrogen management and nitrification inhibitor effects on nitrogen-15 urea: 1. Yield and fertilizer use efficiency. Soil Sci. Soc. Am. J. 54:115-122.

Walters, D.T. and G.L. Malzer. 1990b. Nitrogen management and nitrification inhibitor effects on nitrogen-15 urea: 11. Nitrogen leaching and balance. Soil Sci. Soc. Am. J. 54:122-130.

Westcott, M.P., D.M. Brandon, C.W. Lindau, and W.H. Patrick Jr. 1986. Effects of seeding method and time of fertilization on urea-nitrogen-15 recovery in rice. Agron. J. 78:474-478.

Westfall, D.G. 1984. Management of nitrogen in the mountain states. p. 751-763. In R.D. Hauck (ed) Nitrogen in crop production. ASA, CSSA, SSSA.

Wilson, Jr. C.E., R.J. Norman, and B.R. Wells. 1989. Seasonal uptake patterns of fertilizer nitrogen applied in split applications to rice. Soil Sci. Soc. Am. J. 53:1884-1887.

Wilson, Jr. C.E., B.R. Wells, and R.J. Norman. 1994. Fertilizer nitrogen uptake by rice from urea-ammonium nitrate solution vs. granular urea. Soil Sci. Soc. Am. J. 58:1825-1828.

Wuest, S.B. and K.G. Cassman. 1992. Fertilizer nitrogen use efficiency of irrigated wheat: 1. Uptake efficiency of preplant versus late-season application. Agron. J. 84:682-688