

Distribution of ^{15}N -Labeled Fertilizer Applied to Pecan: A Case Study

Rebecca A. Kraimer, William C. Lindemann¹, and Esteban A. Herrera
 Department of Agronomy and Horticulture, MSC 3Q, New Mexico State University, P.O. Box 30003, Las Cruces, NM 88003-8001

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Abstract. From March through June 1996, ^{15}N -labeled fertilizer was applied to mature pecan trees [*Carya illinoensis* (Wangenh.) K. Koch] in a commercial orchard to determine the fate of fertilizer-N in the tree and in the soil directly surrounding the tree. The concentrations of ^{15}N and total N were determined within various tissue components and within the soil profile to a depth of 270 cm. By Nov. 1996, elevated levels of ^{15}N were greatest at depths just above the water table (280 cm), suggesting a substantial loss of fertilizer-N to leaching. Recoveries of ^{15}N from tissue and soil at the end of 1996 were 19.5% and 35.4%, respectively. Harvest removed 4.0% of the fertilizer-N applied, while 6.5% was recycled with leaf and shuck drop. In 1997, with no additional application of labeled fertilizer, the tissue components continued to exhibit ^{15}N enrichment. By the end of the 1997 growing season, ^{15}N levels decreased throughout the soil profile, with the most pronounced reduction at depths immediately above the water table. Estimated recoveries of ^{15}N from pecan tissue (excluding root) and soil at the end of 1997 were 8.4% and 12.5%, respectively. In 1996 and 1997, ^{15}N determinations indicated an accumulation of fertilizer-N in the tissues and a loss of fertilizer-N to the groundwater. Early spring growth, flowering, and embryo development used fertilizer-N applied the previous year, as well as that applied during the current year.

Although N is a primary component of fertility management in pecan (Hunter and Lewis, 1942; Malstrom et al., 1983), it can produce dichotomous results in pecan production. Storey et al. (1986) found that excessive N application ($436 \text{ kg}\cdot\text{ha}^{-1}$) dramatically reduced yield while increasing both the nut size and the amount of abnormal shuck dehiscence. Worley (1974) suggested that high N application rates (90 and $134 \text{ kg}\cdot\text{ha}^{-1}$) may keep trees more vegetative for longer periods, causing a delay in kernel maturity. Although Smith et al. (1985) found that an annual application of N ($224 \text{ kg}\cdot\text{ha}^{-1}$) improved tree appearance, shoot growth, and the number of new shoots produced, Worley (1990) observed a reduction in nut size and a lower percentage of fancy grade kernels with the same N application rate. In addition, this rate of application generally reduced the leaf concentrations of elemental K, P, and Zn (Smith et al., 1985). Clearly, the amount of fertilizer-N considered optimal or excessive will depend on specific orchard conditions. Although Worley (1990) used the previous year's leaf N concentration to determine N fertilizer application rates for pecan, most researchers (Kilby, 1982; McEachern, 1995; Sparks, 1968) addressed N

requirements for the current year and did not consider the N status of both the soil and tree in the previous year (Conradie, 1983; Oland, 1959).

Leaching of fertilizer-N as NO_3^- is often the most important cause of N loss from the soil/plant system (Cameron, 1983), but Broadbent and Carlton (1978) found that very little NO_3^- derived from fertilizer escaped from the root zone unless fertilizer rates exceeded crop needs. Citrus growers in southern California adjusted the fertilizer-N input to a level that, when combined with good water management for maintenance of low salt levels in soils, minimized NO_3^- in the drainage water (Pratt et al., 1972).

The use of ^{15}N -labeled fertilizer can provide quantitative information about residual fertilizer-N from one growing season to the next, and is the only universal method that completely accounts for the added N, other than that lost in gaseous forms (Allison, 1966). Although ^{15}N has been used successfully to study the movement and fate of fertilizer-N in other woody perennial crops such as apple [*Malus sylvestris* (L.) Mill. var. *domestica* (Borkh.) Mansf.] (Grasmanis and Nicholas, 1971), grape [*Vitis vinifera* L.] (Conradie, 1983), and almond [*Prunus dulcis* (Mill.) D.A. Webb] (Weinbaum and Muraoka, 1986), no reports are known in which ^{15}N has been applied to pecan.

A better understanding of the movement of fertilizer-N in the soil and tree is needed to maximize production while protecting the groundwater from fertilizer- NO_3^- . The objectives of this study were to quantify the distribution of fertilizer-N in the soil and in the tissues of pecan during both the year of fertilizer application and the following year.

The study was initiated in Mar. 1996 in a commercial orchard $\approx 12 \text{ km}$ south of Las Cruces, N.M. The 15-year-old 'Western' trees were growing on a level, well-drained Agua loam [fine-loamy over sandy, mixed (calcareous), thermic Typic Torrifluent] that formed in mixed alluvium in the Rio Grande flood plain. The loam surface graded into a fine loamy sand at a depth of $\approx 45 \text{ cm}$. The orchard was flood-irrigated at a rate of 151.9 cm per year and received 11.4 and 19.3 cm of rainfall in 1996 and 1997, respectively. This amount of flood irrigation is the typical application rate for pecan in the southern Rio Grande Valley. The water table was $\approx 280 \text{ cm}$ below the surface (determined by augering).

One tree was selected in each of three rows, and was representative of other trees in the orchard with respect to canopy cover, trunk diameter, and height ($\approx 81.0 \text{ m}^2$, 33.6 cm , and 14.3 m , respectively). The three trees were managed as were those in the rest of the orchard except that ^{15}N -labeled ammonium sulfate ($221 \text{ kg}\cdot\text{ha}^{-1}$) was applied instead of commercial ammonium sulfate. The trees were hedged in 1993 from north to south at 3 m from the trunk and a height of 12 m . Each of the trees selected was located in the middle of a designated area ($\approx 4100 \text{ m}^2$) where N fertilizer application was managed by the researchers, and each was the center of a $9 \times 9\text{-m}$ plot. Before each ^{15}N application, the three plots were rototilled to roughen the surface and to ensure quick entry of water and fertilizer. The ^{15}N was uniformly hand-applied in the $9 \times 9\text{-m}$ plots; rates and timing of both fertilizer application and irrigation are given in Table 1. The labeled fertilizer was immediately raked in, and the orchard was flood-irrigated within 24 h to minimize ammonia volatilization. A border 1.5 m wide, to which no N fertilizer was applied, surrounded the perimeter of each plot. The remainder of the designated area received nonlabeled $(\text{NH}_4)_2\text{SO}_4$ by hand application at the grower's fertilization rate (N at $373 \text{ kg}\cdot\text{ha}^{-1}$) and schedule. The recommended N application rate for pecan in New Mexico is 168 to $224 \text{ kg}\cdot\text{ha}^{-1}$ (Herrera, 1995). The ^{15}N -labeled ammonium sulfate (ISOTEC, Miamisburg, Ohio) had a total N content of 20.9% and a ^{15}N content of 10.4 atom percent.

Background concentrations of ^{15}N for soil and tissue samples were randomly collected within the same orchard but outside the designated 4100-m^2 research area. Prior to the start of the experiment and three times during the 1996 and 1997 growing seasons, background soil samples were collected and similar depths composited; concentrations of ^{15}N were averaged at each depth for use in calculations. Background leaf samples were collected 15 times during the course of the experiment and their ^{15}N concentrations were averaged for use in calculations. Background ^{15}N concentrations for other tissues (catkin, shoot, shuck, shell, kernel, wood, and root) were collected when the respective tissue was sampled from the three research trees.

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¹To whom reprints requests should be addressed. E-mail address: wlindema@nmsu.edu

Soil samples were collected with a 3.8-cm bucket auger at depths of 0–30, 30–60, 60–90, 90–180, and 180–270 cm within 10 d of irrigation. Three sample holes were augered within each research plot at random distances (ranging from 1 m to 4 m) from each tree. Same depth samples from each of the three holes were composited.

Tissue sampling time depended on the type of tissue sampled. Leaf samples were collected from the middle pair of leaflets at the middle position of the compound leaf of current-year growth on 12 dates in 1996 and seven dates in 1997. Shoots from the last 3 years of growth and catkin samples were collected in the spring (2 May 1996 and 26 Apr. 1997). Shuck, shell, and kernel samples were collected at fruit maturity. The wood (including bark) sample was collected (20 Jan. 1998) by coring to the center of the trunk during dormancy. Roots were removed from soil samples after augering at the end of the growing season (30 Oct. 1996) and brushed clean of soil particles before grinding. Root samples of varying sizes were collected at various depths (0 to 270 cm) from each tree; no attempt was made to distinguish roots collected from different depths.

Soil samples were air-dried for 48 h, then ground to pass a 150- μ m sieve. Tissue samples were oven-dried (65 °C) for 48 h, then ground to pass a 180- μ m sieve. Ground soil and tissue samples were sent to the U.S. Dept. of Agriculture–Agricultural Research Service (USDA–ARS) laboratory at the Univ. of Nebraska, Lincoln. Atom percent of ^{15}N and total N were analyzed with the Tracer Mass Stable Isotope Mass Spectrometer (Europa Scientific, Crewe, Cheshire, U.K.).

Recovery of fertilizer- ^{15}N in the soil and tissue samples is described by the following equations.

$$\% \text{ Recovery}_{\text{soil}} = \frac{(\text{total N}) ({}^{15}\text{N}_{\text{sample}} - {}^{15}\text{N}_{\text{BG}}) (D_B) (\text{Volume}) (100)}{{}^{15}\text{N}_{\text{applied}}}$$

$$\% \text{ Recovery}_{\text{tissue component}} = \frac{(\text{total N}) ({}^{15}\text{N}_{\text{sample}} - {}^{15}\text{N}_{\text{BG}}) (\text{Biomass}) (100)}{{}^{15}\text{N}_{\text{applied}}}$$

where total N = mean total N of three research trees (%); ${}^{15}\text{N}_{\text{sample}}$ = mean ^{15}N of three research trees (atom %); ${}^{15}\text{N}_{\text{BG}}$ = mean ^{15}N of background samples (atom %); D_B = bulk density ($\text{kg}\cdot\text{m}^{-3}$); Volume = soil volume of research plot (m^3/plot); ${}^{15}\text{N}_{\text{applied}}$ = ^{15}N application rate (kg/plot); Biomass = mean mass of each tissue component of three research trees (kg/plot).

The estimation of aboveground biomass was needed to determine the biomass of each aboveground tissue component. The following model (King and Schnell, 1972) for black oak (*Quercus velutina* Lam.) was used to estimate above- and belowground biomass.

Total biomass:

$$\text{LOG wt} = 1.00005 + 2.10621 \text{ LOG dbh};$$

Root and stump biomass:

$$\text{LOG wt} = 0.38000 + 2.12094 \text{ LOG dbh};$$

where dbh = diameter of trunk at breast height (1.37 m aboveground) in inches; wt = oven-dried weight in pounds.

To confirm the validity of this model for pecan, a tree similar in size to the research trees was sacrificed and all aboveground tissues were collected on a component basis. The leaf and shuck components were dried (65 °C) and weighed directly. The shell and kernel components were also weighed directly. To determine the dry biomass of the wood tissue, representative samples of trunk, large branches (>7.6 cm diameter), medium branches (2.5 to 7.6 cm), small branches (<2.5 cm), and twigs (<1.3 cm) were dried (65 °C) and weighed. The measured aboveground biomass of the sacrificial tree was 8.6% less than its calculated biomass when the dbh of the sacrificed tree was substituted into the model equations and the aboveground biomass determined by difference.

The dbh of each research tree was used to calculate its aboveground biomass. The proportions of leaf, shuck, and wood tissue of the sacrificial tree were applied to the calculated value of the mean aboveground biomass of the three research trees as described in the following example for the leaf component.

$$\text{Biomass}_{\text{leaf}(\text{research})} =$$

$$(\text{AG Biomass}_{\text{model}(\text{research})}) (\text{Biomass}_{\text{leaf}(\text{sacrificial})})$$

where $\text{Biomass}_{\text{leaf}(\text{research})}$ = mean estimated leaf biomass of three research trees (kg); $(\text{AG Biomass}_{\text{model}(\text{research})})$ = mean aboveground biomass of three research trees (kg); $(\text{Biomass}_{\text{leaf}(\text{sacrificial})})$ = leaf tissue in aboveground biomass of sacrificial tree (%). The mean aboveground biomass was calculated by substituting the mean dbh of the three research trees into the model equations and determining the aboveground biomass by difference. Estimations of root biomass relied solely on the model equation. The kernel and shell components of the research trees were directly weighed.

Results and Discussion

Following the first application (25 Mar. 1996) of ^{15}N -labeled ammonium sulfate (116.6 $\text{kg}\cdot\text{ha}^{-1}$), the orchard was flood-irrigated with 9.9 cm of water (Table 1). Trace amounts of ^{15}N had clearly moved to a depth just above the water table (280 cm) by 10 d after application (Fig. 1A), but the highest concentration remained in the upper soil profile. The ^{15}N recovery in the entire profile was 98.6%, with 85.8% and 82.5% remaining in the upper 90 and 60 cm, respectively. The ^{15}N recovery of 98.6% fell within the range of those in previous reports (Chen et al., 1995).

Table 1. Dates and rates of application of ^{15}N -labeled $(\text{NH}_4)_2\text{SO}_4$ to pecan trees and quantity of irrigation water applied immediately following fertilization, 1996.

Date	Application rate ($\text{kg}\cdot\text{ha}^{-1}$)			
	$(\text{NH}_4)_2\text{SO}_4$	N	^{15}N	Water applied (cm)
25 Mar.	116.6	24.3	2.53	9.9
23 Apr.	343.7	71.7	7.48	7.6
13 May	151.0	31.5	3.28	8.4
24 May	151.0	31.5	3.28	8.3
3 June	148.4	31.0	3.23	9.3
15 June	148.4	31.0	3.23	11.2
Total	1059.1	221.0	23.03	54.7 ^z

^zTotal irrigation water applied during 1996 = 151.9 cm.

At the end of the growing season (6 Nov. 1996), after labeled $(\text{NH}_4)_2\text{SO}_4$ (1059.1 $\text{kg}\cdot\text{ha}^{-1}$) had been applied (March through June), $\approx 35.4\%$ of the ^{15}N applied was recovered in soil to a depth of 270 cm (Fig. 1B). Enrichment was most pronounced at the greater depths. The presence of elevated ^{15}N levels directly above the water table indicates a loss of fertilizer-N to the groundwater by leaching. Root penetration in a clay soil is often twice the depth of that in a sandy soil (Gustafson, 1983), and the micropores of a well-aggregated clay soil inhibit leaching while water easily percolates through the macropores of a sandy soil (Duynisveld et al., 1988). This was substantiated when Weinbaum et al. (1984a) observed that the crop recovery of fertilizer-N on a heavy-textured soil was nearly 50% greater than on a light-textured soil.

During the following winter, the orchard was irrigated with 11.3 cm of water and received an additional 1.5 cm of precipitation. Between 6 Nov. 1996 (Fig. 1B) and 3 Mar. 1997 (Fig. 2A), 13.0% of the ^{15}N applied in 1996 was lost from the soil profile, again suggesting leaching of fertilizer-N into the groundwater. Broadbent and Carlton (1978) found that excess irrigation leached residual fertilizer- ^{15}N from the soil profile below the rooting zone. The ^{15}N accumulation in the 0–90 cm depth may have resulted from the replenishment of ^{15}N at the soil surface by the recycling of 1996 leaves and shucks. Adams and Attiwell (1986) also observed the importance of nutrient recycling by litterfall in Australian eucalypt [*Eucalyptus pauciflora* (Sieber) Sprengel and *E. delegatensis* (R.T. Baker)] forests and found that $\approx 80\%$ of the N cycled from plant to soil is in the form of litter.

A comparison of the 3 Mar. 1997 (Fig. 2A) and 21 Oct. 1997 (Fig. 2B) soil profiles showed diminished ^{15}N concentrations at all depths in October. This suggests additional loss of fertilizer-N from the soil/plant system by leaching. The accumulation of ^{15}N near the surface may have resulted from the recycling of 1996 leaves and shucks. The ^{15}N concentration approached background ^{15}N levels with increasing depth below 90 cm. After two growing seasons, 22% of the ^{15}N -labeled fertilizer applied in 1996 remained in the soil profile.

^{15}N Nitrogen excess (atom %) refers to the concentration of the ^{15}N isotope in the total N above the background level. Total N remained

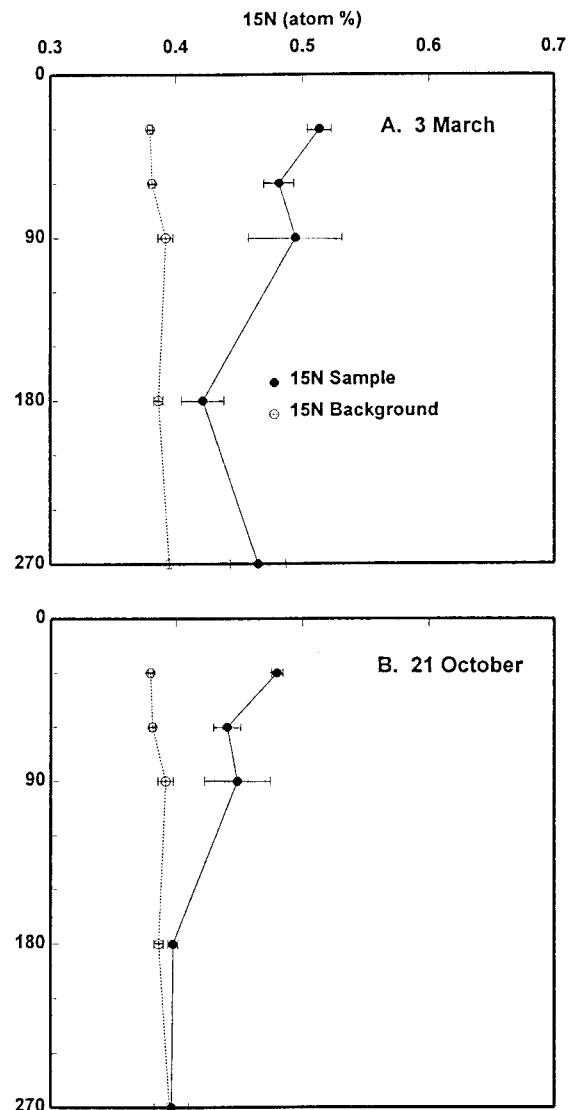
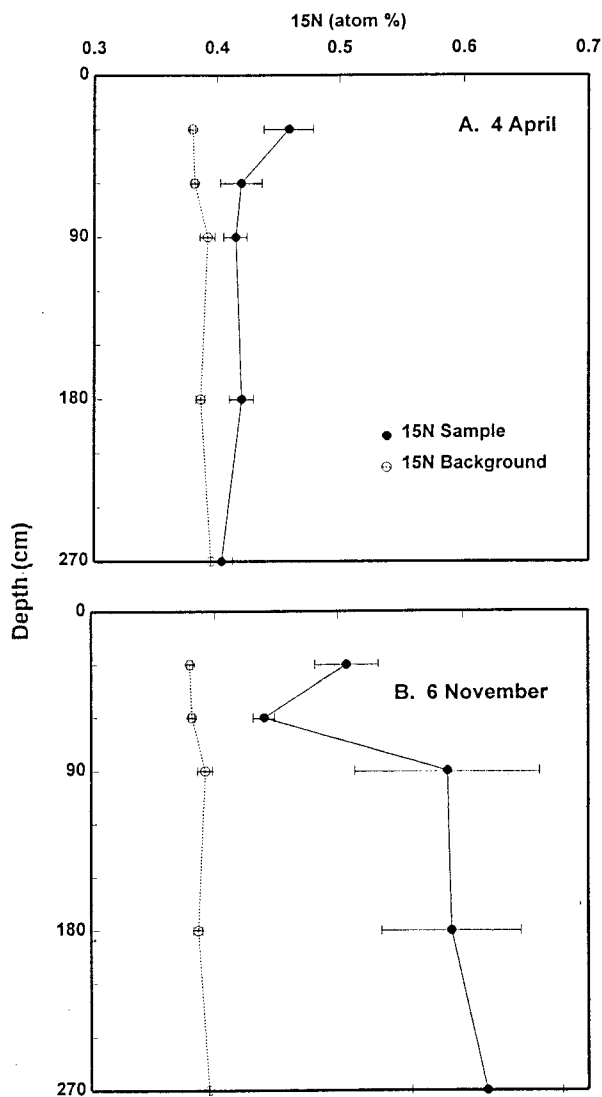


Fig. 1. ^{15}N distribution in the soil profile on (A) 4 Apr. 1996 after application of 117 kg labeled (10.4 atom percent) $(\text{NH}_4)_2\text{SO}_4$ and (B) 6 Nov. 1996 after application of 1059 kg labeled (10.4 atom percent) $(\text{NH}_4)_2\text{SO}_4$. Standard error of the mean ($n = 3$) is depicted by error bar.

Fig. 2. ^{15}N distribution in the soil profile on (A) 3 Mar. 1997 and (B) 21 Oct. 1997. Standard error of the mean ($n = 3$) is depicted by error bar.

consistent in corresponding tissues (shoot, leaf, catkin) from 1996 to 1997, but ^{15}N enrichment was higher in 1997 (Fig. 3). This was attributed to the remaining applications of labeled fertilizer ($598.8 \text{ kg}\cdot\text{ha}^{-1}$) yet to be applied in May and June of 1996, after the 2 May 1996 sample date. At the end of the 1996 growing season, recovery of ^{15}N from tissues was 19.5% (Table 2). ^{15}N enrichment was evident in both perennial and annual tissues, with the total N concentrations of leaf, catkin, and current-year shoot closely agreeing with values of corresponding tissues in apple trees (Grasmanis and Nicholas, 1971). While the 1996 data show that spring growth in pecan uses N applied during the current year, the 1997 data show that early growth is also dependent upon N applied the previous year.

In 1996, ^{15}N rapidly accumulated in leaf tissue (Fig. 4). Wallace et al. (1954) and Weinbaum et al. (1984b) found similar results when ^{15}N was applied to 'Washington Navel' and 'Valencia' orange [*Citrus sinensis* (L.)

Table 2. Mean ^{15}N recovery at the end of the 1996 and 1997 seasons following soil application of ^{15}N - $(\text{NH}_4)_2\text{SO}_4$ to pecan trees.

Component	^{15}N Recovery			
	1996		1997	
	($\text{kg}\cdot\text{ha}^{-1}$)	(atom %)	($\text{kg}\cdot\text{ha}^{-1}$)	(atom %)
Soil (0-270 cm)	8.1	35.4	2.9	12.5
	<i>Tree (by component)²</i>			
Leaf	1.3	5.6	0.6	2.7
Shuck	0.2	0.9	0.1	0.4
Kernel	0.8	3.4	0.2	0.8
Shell	0.1	0.6	0.0	0.1
Root	0.7	3.2	---	---
Wood ³	1.3	5.8	1.0	4.4
Tree (subtotal)	4.4	19.5	1.9	8.4
Total (soil + tree)	12.5	54.9	4.8	20.9

²Based on ^{15}N contents of plant parts, measurement of kernel and shell biomass, and estimation of leaf, shuck, wood, and root biomass by the King and Schnell (1972) model. The ^{15}N application rate used for recovery calculations was $23.03 \text{ kg}\cdot\text{ha}^{-1}$.

³Root data for 1997 were not recorded.

⁴Wood sample includes bark.

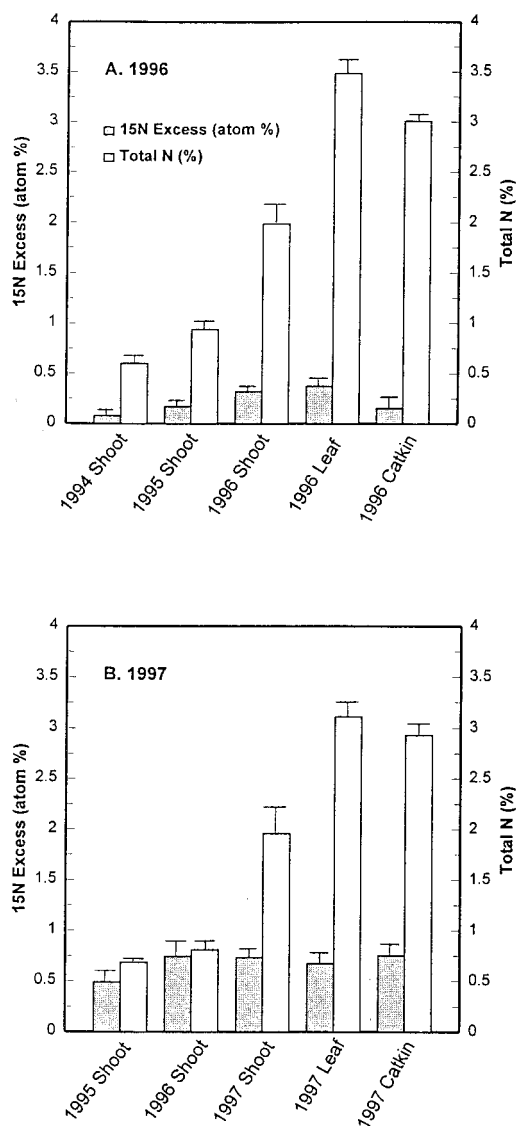


Fig. 3. ¹⁵N excess and total N concentrations in pecan tree tissues sampled on (A) 2 May 1996 and (B) 26 Apr. 1997. Standard error of the mean (n = 3) is depicted by error bar.

Osbeck] and 'Nonpareil' almond [*Prunus dulcis* (Mill.) D.A. Webb], respectively. The growing leaf tissue effectively acts as a reservoir of nitrogenous compounds imported from the roots (Titus and Kang, 1982). By 15 June 1996, all of the ¹⁵N ammonium sulfate (1059 kg-ha⁻¹) had been applied, which explains the high ¹⁵N concentrations throughout the remainder of the growing season. The amount of N recycled to the soil through leaf fall was represented by the senescent leaf sample of 15 Nov. 1996.

In 1997, the grower applied commercial ammonium sulfate (1800 kg-ha⁻¹) in several split applications to the orchard, including the research plots. The concentration of ¹⁵N in the leaf was nearly constant throughout the growing season (Fig. 5), while the total N showed a trend similar to that in 1996 (Fig. 4). The ¹⁵N present in the 1997 leaf tissue represents either the fertilizer-N from 1996 that was translocated from reserve tissue or N absorbed from the soil. The latter includes newly absorbed

fertilizer-N remaining in the rooting zone from the previous year and reabsorbed N from leaf and shuck decomposition.

At the end of the 1996 growing season, the total recovery of ¹⁵N was 54.9% (Table 2). Of this, 35.4% was in the soil and 19.5% in the tissue. Of the ¹⁵N recovered in the tissue, 4.0% (kernel + shell) was removed during harvest, 6.5% (leaf + shuck) was recycled, 3.2% remained in the root, and 5.8% remained in wood. The soil and tissue recovery values are lower than those reported by Allison (1966) and Bock (1984). However, Hunter (1956) also observed that little N is removed with the harvest of pecan. The unaccounted for N (45.1%) was presumably lost through leaching, denitrification, and ammonia volatilization. We suspect that leaching was the major cause of loss. Volatilization was kept to a minimum because the ¹⁵N fertilizer was incorporated by hand and the orchard irrigated within 24 h. Denitrification was probably low because of the low organic matter content of

the soil; a readily available energy source is required for denitrification (Myrold, 1998).

In 1997, although root data were not obtained, the total recovery of fertilizer-N applied in 1996 was 20.9%. At the end of the growing season, 12.5% remained in the soil profile and 8.4% in the tissue. Of the latter, 0.9% was harvested, 3.1% was recycled, and 4.4% remained in the wood.

Our experiment was conducted under fertilization, irrigation, and other management practices typical of commercial pecan production in the Southwest. It has provided the first quantitative data describing the fate of ¹⁵N fertilizer applied to pecan during the year of application and the subsequent year. The data indicate that management of both N fertilizer and water are closely linked. The soil at depths greater than ≈30 cm remained near field capacity the entire growing season, as determined by neutron probe and tensiometer measurements (data not shown). However, irrigation water was applied numerous times during the growing season and during the winter to keep the top 30 cm moist. Our data clearly show ¹⁵N accumulation below 90 cm and its eventual loss from the profile, particularly after a winter irrigation. Although some leaching is required to control salinity, applications of smaller amounts of water would conserve both water and N fertilizer, and would reduce nitrate contamination of groundwater.

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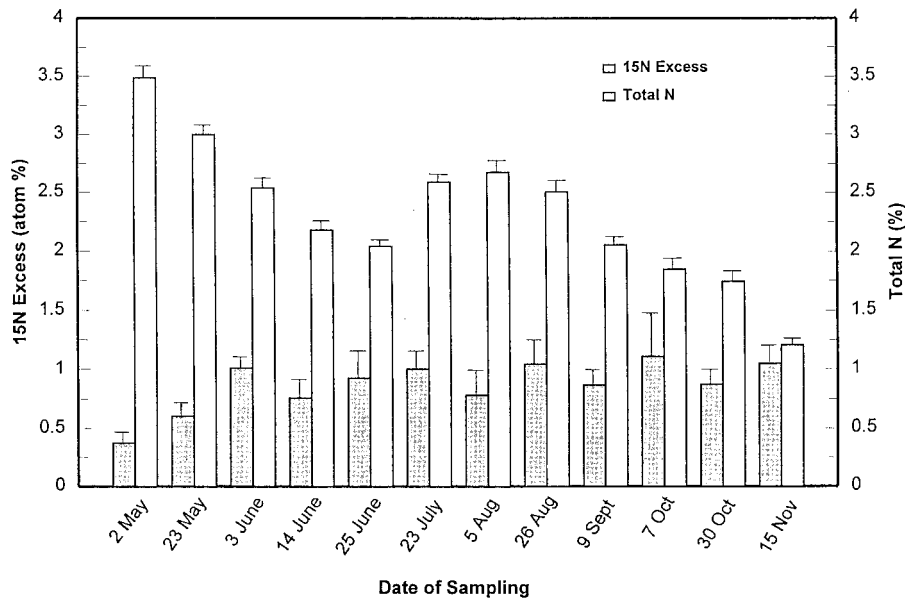


Fig. 4. ¹⁵N excess and total N concentrations in pecan leaf, 1996. Standard error of the mean (n=3) is depicted by error bar.

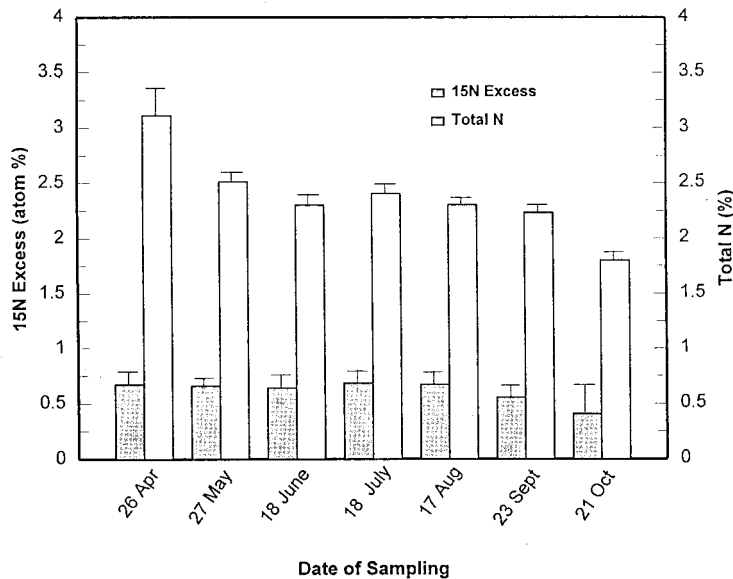


Fig. 5. ¹⁵N excess and total N concentrations in pecan leaf, 1997. Standard error of the mean (n=3) is depicted by error bar.

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