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Fate of ^{15}N-labelled fertilizer in a long-term field trial at Ropsley, UK

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SUMMARY

In 1992 and 1993, eight rates of ^{15}N-labelled fertilizer (0–245 kg ha^{-1}) were applied to winter wheat growing on the Ropsley long term field trial where eight different N amounts had been applied annually since 1978. The fate of the labelled N in the crop and topsoil (0–23 cm) was determined at harvest in the year of ^{15}N application and in the first and second residual years.

By harvest in the second residual year, 60–77% of the original labelled application had been recovered in the crop and topsoil with 23–40% lost. These losses virtually all occurred within the first two growing seasons; there was no significant loss during the second residual year. Significant changes in the ^{15}N balance were observed at N applications in excess of the range 140–175 kg ha^{-1} which suggested a marked decrease in the efficiency of N use and an increase in residual labile N in the soil at harvest. At low N applications (< 175 kg ha^{-1}), a positive added nitrogen interaction (ANI) was observed: 40–50% of this was a *residual* ANI due to the short or long term effect of applying N fertilizer, and the remainder was probably an *apparent* ANI due to pool substitution in the immobilization process. At large N applications (> 175 kg ha^{-1}) a negative ANI was observed: large N applications resulted in a net suppression of soil N uptake due to substitution by fertilizer for a limited plant N demand.

INTRODUCTION

Accurate predictions of the nitrogen requirements of cereals are needed to minimize the adverse environmental and economic effects of incorrect fertilizer use. This can only be achieved if the recovery of N from fertilizer and non-fertilizer sources by a crop is predictable. Recovery of N fertilizer is generally incomplete and highly variable. For example, Bloom *et al.* (1988) observed that the apparent recovery of N by winter wheat (measured as the difference between N offtake from fertilized and unfertilized crops) varied from 40 to 88% in 70 experiments between 1981 and 1986 at different sites in the UK. Powlson *et al.* (1992) observed similar crop recoveries using ^{15}N-labelled fertilizer in nine experiments carried out over a period of 4 years: by harvest, *c.* 46–87% (68% mean) of the labelled N applied was recovered in the crop, 18% was retained in the soil with 1–35% (13% mean) being lost.

The majority of fertilizer recovery studies have focused upon single year effects. However, as crop recovery of N fertilizer is usually incomplete, the nature and fate of any residual fertilizer N remaining in the soil at harvest and the long term consequences of applying fertilizer for a number of years, are of considerable environmental and economic importance. Investigations of long term field experiments have revealed that the application of N fertilizer for a number of years can increase both the total and mineralizable N content of a soil (Glendining & Powlson 1995). This has been seen to affect the N nutrition of subsequent crops significantly (Powlson *et al.* 1986) and may increase the risk of winter leaching losses (Glendining *et al.* 1989).

The aim of the experiment described in this paper was to determine the fate of ^{15}N-labelled fertilizer applied to winter wheat grown on a long term field trial at Ropsley in Lincolnshire where eight different rates of N fertilizer had been applied since 1978 (Bhogal *et al.* 1995, 1996). Thus, in 1992 and 1993, ^{15}N-labelled fertilizer was applied to three replicates of each of the different N applications and its recovery

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measured in crop and topsoil after harvest in the year of application (APP) and following the first and second residual years (R1 and R2). The effects of 15 years of different N fertilizer applications on the recovery of fertilizer N could thus be studied, with the aim of improving understanding of the fate of N fertilizer in arable cropping systems subject to sustained fertilizer regimes.

MATERIALS AND METHODS

Details of the site history and layout of the Ropsley long term field trial have been reported elsewhere (Bhogal *et al.* 1995, 1996). Briefly, from 1978 to 1990, winter wheat (WW) was grown in a four-course rotation of oilseed rape, WW, WW and winter barley; each of the four rotational strips contained ten blocks of the eight N treatments. In addition, from 1978 to 1982, two rates of P and K fertilizer were broadcast to the seedbed; five blocks in each rotational strip received 21·8 and 41·5 kg ha^{-1} P+K (PK1) and the remainder received 87·3 and 166 kg ha^{-1} P+K (PK2). As each rotational strip entered its second rotational cycle, an annual application equivalent to PK1 was broadcast to all the plots instead. Since 1990, continuous winter wheat (cv. Mercia) has been grown over the whole site at eight different N rates ranging from 0 to 245 kg ha^{-1} (N1–N8, 35 kg ha^{-1} increments); 35 kg ha^{-1} was applied as an early top-dressing (ETD) at tillering in March and the remainder applied at main top-dressing (MTD) at the beginning of stem extension in April or early May. The ^{15}N experiment was superimposed on the existing long term trial at Ropsley, following the basic protocol used by the AFRC Institute of Arable Crops Research, Rothamsted (Powlson *et al.* 1986, 1992; Macdonald *et al.* 1989; Hart *et al.* 1993). The soil is a Beccles series, sandy clay loam.

Application of ^{15}N-labelled fertilizer

In spring 1992 and 1993, microplots (2×2 m) were established within all eight N mainplots in three replicate blocks of one of the rotational strips of the Ropsley field trial. A second series of 24 microplots was set up in spring 1993 close to the opposite end of the same N mainplots. Details of the field operations during the period of study are given in Table 1. Double-labelled $^{15}NH_4{}^{15}NO_3$ was applied as a solution at both ETD and MTD using a spreader designed for accurate and even liquid application (Woodcock *et al.* 1982). Following application, each plot (4 m^2) was watered with 2 litres of deionized water in order to wash labelled fertilizer off the plants. The atom % ^{15}N enrichment values of the fertilizer solutions are given in Table 2. Prilled unlabelled NH_4NO_3 was applied to the surrounding mainplots, by hand, 4–10 days after the labelled fertilizer application to the microplots (Table 1).

All the microplots received a normal dressing of unlabelled, prilled NH_4NO_3 in the years following the single labelled fertilizer application, so that the fate of any residual labelled fertilizer left in the soil could be measured under continuing normal fertilizer practice. The locations of the microplots were adjusted each

Table 2. *Atom % excess ^{15}N enrichment of the fertilizer solutions applied in 1992 and 1993*

Nominal nitrogen application rate (kg ha^{-1})	Atom % excess ^{15}N enrichment	
	1992	1993
35, 70	7·89	8·08
105, 140	2·95	3·03
175, 210, 245	1·94	2·00

Table 1. *Field operations at Ropsley during the period 1992–94. ETD, early top-dressing of fertilizer; MTD, main top-dressing of fertilizer*

Operation	Year of fertilizer application		
	1992	1993	1994
Ploughed	23/9/91	20/9/92	21/9/93
Drilled	10/10/91	12/10/92	26/10/93
ETC: ^{15}N microplots	6/3/92	18/3/93	—
ETD: mainplots	16/3/92	22/3/93	15/3/94
MTD: ^{15}N microplots	21/4/92	28/4/93	—
MTD: mainplots	27/4/92	5/5/93	6/5/94
Harvest: ^{15}N microplots	3/8/92	17/8/93	9/8/93
Harvest: mainplots	21/8/92	26/8/93	13/8/94
Soil sampling			
^{15}N microplots – total N & ^{15}N	5/8/92	18/8/93	12/8/94
Mainplots – spring inorganic N	9/3/92	10/3/93	18/2/94
Mainplots – autumn inorganic N	6/11/92	—	—

year in order to allow for a shift of *c.* 35 cm in the topsoil due to ploughing, following the procedure adopted by the AFRC Institute of Arable Crops Research, Rothamsted (P. R. Poulton, personal communication). Ploughing was in opposite directions in alternate years to minimize lateral soil movement. The microplots are coded according to the year of harvest (1992–94) and length of time after their single ^{15}N application: results from the year of application, first residual year and second residual year are designated APP, R1 and R2 respectively.

Microplot harvest procedure

The central 1 m^2 of each microplot was harvested by hand each year just before the mainplots were harvested by combine (Table 1). This left a discard area (0.5 m) which has been shown to be sufficient to avoid edge effects (Powlson *et al.* 1986). The straw and ears were separated from a subsample of the whole crop (150 g , *c.* 16% of the total plot harvest) and dried at 80 °C for 48 h. The ears were threshed and the chaff retrieved and added to the straw subsample.

In order to study the residual effects of the ^{15}N fertilizer accurately, the stubble in the central 1 m^2 had to be incorporated in the normal autumn cultivation operations, so could not be harvested for ^{15}N assay. However, the two rows of wheat immediately adjacent to the central harvest area have been shown to have almost identical ^{15}N enrichments to those within the central 1 m^2 (Powlson *et al.* 1986). Therefore, stubble ^{15}N content was assayed using eight grab samples consisting of the whole plant and crown, taken from the discard region just outside the central 1 m^2 harvest area. The plants were cut at 10 cm above the crown, the roots removed and the remaining stubble and crown were washed to remove all adhering soil before being dried at 80 °C for 48 h. The oven-dry yields of grain (g), straw + chaff (Str) and stubble (Stub) were thus calculated for the whole microplot; grain yields were subsequently recalculated at 85% DM (by convention). All oven-dried plant samples were ground in a hammer mill and a subsample (*c.* 20 g) was finely ground in a tema mill prior to analysis of the total N content and ^{15}N enrichment by mass spectrometry.

Soil analysis

Total soil N and ^{15}N enrichment

The topsoil of each microplot was sampled to plough depth (0–23 cm) immediately after harvest (Table 1) in each of the three trial years using a 30 mm diameter gouge auger. Nine cores were taken from the central square metre of each microplot in a square grid pattern and bulked together. The samples were then air-dried, sieved to < 2 mm and ground using a mechanical pestle and mortar. Approximately 20 g of each sample was finely ground in a tema mill prior to analyses for total N and ^{15}N enrichment. Results are expressed on an oven-dry (105 °C) basis. The soil (< 2 mm) dry bulk density, determined at harvest 1994, was $1{\cdot}48 \pm 0{\cdot}01$ g cm^{-3}.

Soil inorganic N in the autumn and spring

The inorganic N content of the soil profile (0–90 cm) in the mainplots was measured in the autumn and spring (Table 1), by ADAS, from 1990 to 1994. Six replicate samples were taken from the 0–30 cm, 30–60 cm and 60–90 cm horizons of two blocks of the eight N treatments in each rotational strip. Replicate samples were bulked and stored frozen prior to determination of the ammonium and nitrate contents by standard analytical techniques (MAFF 1986).

Crop N uptake prior to fertilizer application and background ^{15}N enrichment

Plant and topsoil samples (0–15 cm) were taken in March 1992 for determination of the background ^{15}N enrichment. Two soil cores (0–15 cm) were taken from the topsoil of every plot at Ropsley (320 samples) and bulked according to (past) rotational strip, N and P + K treatment (64 samples; see Bhogal *et al.* (1995, 1996) for full details of the trial design and history). These were then air-dried and sieved (< 2 mm) prior to determination of the total N and organic matter content using standard analytical techniques (MAFF 1986). The background ^{15}N enrichment was only determined in samples taken from the rotational strip where the ^{15}N microplots were to be established (16 samples). Wheat plants were also harvested in March 1992 from an area (0·16 m^2) within two N1 (0 kg ha^{-1}) and two N8 (245 kg ha^{-1}) plots of this rotational strip. The two replicates were bulked together, dried (80 °C for 48 h) and ground. The background enrichments were 0·3659 and 0·3699 atom% ^{15}N for the plant and soils, respectively.

In spring of the second year of ^{15}N application (1993), the standing crop was too poorly established to warrant measurement of plant biomass and N uptake prior to fertilizer application. However, in the following year, the uptake of *residual* ^{15}N over winter was measured by sampling plants on 15 March 1994 from a 1 m row just outside the central harvest area of every microplot fertilized with ^{15}N in 1993, and assaying for ^{15}N and total N.

Determination of the relative availabilities of residual labelled N and native soil N

Uniquely, in 1993, the simultaneous harvest of labelled application and labelled residual plots (1993–APP, 1993–R1) permitted separation of the (unlabelled) soil N uptake (Np_s, calculated from the APP results) into contributions from residual fertilizer N ($Np_{f(res)}$) and native soil organic matter (Np_s^*). The relative availability (availability ratio) of these two N pools was determined by comparing the uptake of (native) soil N and residual fertilizer N with the total amount of N in their respective pools (Eqns (1–3)):

$$\text{Availability ratio} = \frac{\%Np_{s(tot)}}{\%Np_{f(res)}} \tag{1}$$

Where:

$$\%\ Np_{s(tot)} = \left(\frac{Np_s^*\,APP^{1993}}{Ns}\right)100 \tag{2}$$

and

$$\%\ Np_{f(res)} = \left(\frac{Np_f\,R1^{1993}}{Ns_f + Nstub_f}\right)100 \tag{3}$$

$\%\ Np_{s(tot)}$ = Proportion of the total (native) soil N pool (Ns) taken up by the plant.

$\%\ Np_{f(res)}$ = Proportion of the residual labelled N pool taken up by the plant.

$Np_s^*APP^{1993}$ = Unlabelled N in the plant originating from a native soil N source as distinct from fertilizer residues in 1993–APP (kg ha^{-1}).

Np_fR1^{1993} = Labelled N in the plant at harvest 1993–R1 which originated from the previous (1992) fertilizer application (kg ha^{-1}).

Ns_f = Labelled N in the soil which originated from fertilizer N (kg ha^{-1}).

$Nstub_f$ = Labelled N in the stubble which originated from fertilizer N (kg ha^{-1}).

Determination of the residual effect of past fertilizer application on the N uptake of subsequent crops

In 1991, ADAS started a series of experiments at Ropsley in order to determine the effect of past N applications on the yield and N uptake of the subsequent crop. Two blocks of the eight N application mainplots in each rotational strip (a total of 64 plots) received no application of N fertilizer for that season; these were termed *holiday plots*. This was repeated in 1992, 1993 and 1994 to two new blocks in each rotational strip. The holiday plots were harvested, by combine, with the rest of the trial and grain yields (Yg^H) and grain N offtake (Ng^H) were calculated. Each year, the previous year's holiday plots received a normal application of N fertilizer.

The holiday plots (N1–N8) in 1992 and 1993 therefore acted as valid controls to their respective (N1–N8) ^{15}N-fertilized plots because they shared the same application history. The holiday plots were therefore used to determine any additional soil N uptake from the ^{15}N-labelled microplots (in excess of control plots) due to the application of fertilizer (this is termed an *Added Nitrogen Interaction* or ANI; Jenkinson *et al.* (1985)). By contrast, use of the N1 microplot as a universal control is complicated by the variation in residual effect of past N applications over the range N1–N8. However, as the holiday plots were harvested by combine with the rest of the mainplots at Ropsley, they could not be directly compared with the results from the ^{15}N-labelled microplots, which were harvested by hand. Microplots have been seen to produce different yields compared to mainplots, due to differences in harvesting and sampling procedures; there is usually a greater retrieval of grain from hand-harvested microplots (Bloom *et al* 1988; Saffigna 1988). At Ropsley, in all 3 years, microplot grain yields were, on average, $1{\cdot}16 \pm 0{\cdot}11$ t ha^{-1} higher than the yields from their associated mainplots ($P < 0{\cdot}001$). Therefore in order to provide an estimate of the N uptake from (hypothetical) 'holiday' microplots (Np_s^{0H}), the ratio of grain N offtake from the holiday and fertilized mainplots (Ng^H/Ng^{main}) was multiplied by the total N uptake from the corresponding ^{15}N microplots (Np; Eqn (4)).

$$Np_s^{0H} = \left(\frac{Ng^H}{Ng^{main}}\right)Np \tag{4}$$

where Ng^H = nitrogen in the grain from the holiday plots (kg ha^{-1}), Ng^{main} = nitrogen in the grain from the fertilized mainplots (kg ha^{-1}) and Np = total N in the plants grown in the ^{15}N-labelled microplots (kg ha^{-1}).

The value of Np_s^{0H} could then be compared directly with unlabelled N uptake from the ^{15}N fertilized plots to give a measure of the ANI, independent of any historical effect of N application ($\text{ANI} = Np_s - Np_s^{0H}$).

RESULTS AND DISCUSSION

The N balance for the 1992 application of labelled fertilizer from 1992 to 1994 is shown in Table 3. By harvest 1994–R2, after three growing seasons, 40–63 % of the original ^{15}N application had been recovered in the harvested crop, 8–25 % in the stubble and topsoil (0–23 cm), with 23–40 % lost. Losses of labelled N virtually all occurred in the first two growing seasons and were fairly evenly split between the period from ^{15}N application and harvest–APP and from harvest–APP to harvest–R1 (on average, slightly more was lost over the latter period; Table 3). There was no significant loss during the second residual year. The total balance for the 1993 ^{15}N application appeared to follow the same pattern as that for 1992 (Table 4): by the end of the first residual year (1994–R1), 47–65 % had been recovered in the harvested crop, 10–21 % recovered in the stubble and topsoil with 21–32 % lost. Again approximately half

Table 3. *Total labelled N balance from 1992 to 1994 of the 1992 application of ^{15}N-labelled fertilizer* (*kg ha^{-1}*)

N applied (kg ha^{-1})	Recovery of labelled N at harvest 1992–APP (kg ha^{-1})			Recovery of labelled fertilizer at harvest 1993–R1 (kg ha^{-1})			Recovery of labelled fertilizer at harvest 1994–R2 (kg ha^{-1})		
	$Ng_f + Nstr_f$	$Nstub_f + Ns_f$	NL_f	$Ng_f + Nstr_f$	$Nstub_f + Ns_f$	NL_f	$Ng_f + Nstr_f$	$Nstub_f + Ns_f$	NL_f
34·0	12·4	16·9	4·73	0·57	10·3	10·8	0·43	7·24	13·4
66·7	32·1	22·3	12·3	0·47	9·17	25·0	0·49	16·7	16·9
100	52·7	32·9	14·8	1·05	18·5	27·8	0·90	19·2	26·7
133	81·8	43·9	7·61	1·24	22·9	27·4	1·12	19·0	30·2
166	101	55·7	8·93	2·64	15·8	46·2	1·22	12·8	48·0
200	108	68·9	22·6	4·06	27·9	59·6	1·54	19·5	66·4
232	121	85·7	25·1	7·70	32·0	71·1	1·97	40·4	60·8

APP, year of ^{15}N-labelled fertilizer application; R1 and R2, first and second residual years after ^{15}N-labelled fertilizer application; Ng_f, labelled nitrogen in the grain; $Nstr_f$, labelled nitrogen in the straw; $Nstub_f$, labelled nitrogen in the stubble; Ns_f, labelled nitrogen in the topsoil (0–23 cm); NL_f, cumulative loss of labelled nitrogen measured at harvest.

Table 4. *Total labelled N balance from 1993 to 1994 of the 1993 application of ^{15}N-labelled fertilizer* (*kg ha^{-1}*)

N applied (kg ha^{-1})	Recovery of labelled N at harvest 1993–APP (kg ha^{-1})			Recovery of labelled fertilizer at harvest 1994–R1 (kg ha^{-1})		
	$Ng_f + Nstr_f$	$Nstub_f + Ns_f$	NL_f	$Ng_f + Nstr_f$	$Nstub_f + Ns_f$	NL_f
34·4	15·7	12·6	6·10	0·57	7·23	10·9
69·3	37·4	23·0	8·94	0·95	12·2	18·8
102	57·0	24·3	21·1	1·39	15·0	29·0
136	85·8	34·9	15·7	2·11	19·3	29·3
170	107	44·9	18·1	2·79	16·2	43·9
204	129	50·8	24·1	3·81	19·2	51·9
238	147	74·5	16·7	6·37	34·1	50·8

APP, year of ^{15}N-labelled fertilizer application; R1 and R2, first and second residual years after ^{15}N-labelled fertilizer application; Ng_f, labelled nitrogen in the grain; $Nstr_f$, labelled nitrogen in the straw; $Nstub_f$, labelled nitrogen in the stubble; Ns_f, labelled nitrogen in the topsoil (0–23 cm); NL_f, cumulative loss of labelled nitrogen measured at harvest.

of this loss occurred in the period March–August of the first growing season and the remainder in the second growing season.

Recovery of labelled N in the crop (Np_f)

In both 1992–APP and 1993–APP, the proportional recovery of labelled N in the crop increased significantly with increasing N level up to 140 kg ha^{-1}, above which it remained constant or declined slightly ($P < 0·001$; Fig. 1*a*, *b*). Maximum recovery was virtually identical in both years (≈ 66%) and was attained at approximately the same N rate (140 kg ha^{-1}). The proportion of labelled N recovered in the crop at harvest in the first residual years (1993/94–R1) was, on average, only 2·45 ± 0·6% and 1·77 ± 0·19% of the original 1992 and 1993 applications of labelled N (Fig. 1*a*, *b*). This was less than 10% of the labelled N remaining in the soil at autumn of the application year. Crop recovery of labelled N in the second residual year (1992–R2) was even lower: only 0·91 ± 0·013% of the original application or 5·7 ± 0·005% of that remaining in the soil the previous autumn (Fig. 1*a*). These results are comparable to the proportional labelled N uptake observed in several other ^{15}N cereal experiments (Table 5).

Retention of labelled N in the topsoil (Ns_f)

Labelled N was assayed for the top 23 cm of soil at Ropsley following the results of experiments carried out at Rothamsted by Powlson *et al.* (1986, 1992), Macdonald *et al.* (1989) and Hart *et al.* (1993). In these trials, labelled N below 23 cm was < 5% of the original application. Negligible recoveries of labelled N in the subsoil (> 30 cm) have also been observed by Khanif *et al.* (1984), Recous *et al.* (1988*a*, *b*, 1992) and Esala (1994). The soil ^{15}N assay at Ropsley included labelled N retained in root residues, humus and inorganic N. Macdonald *et al.* (1989), however,

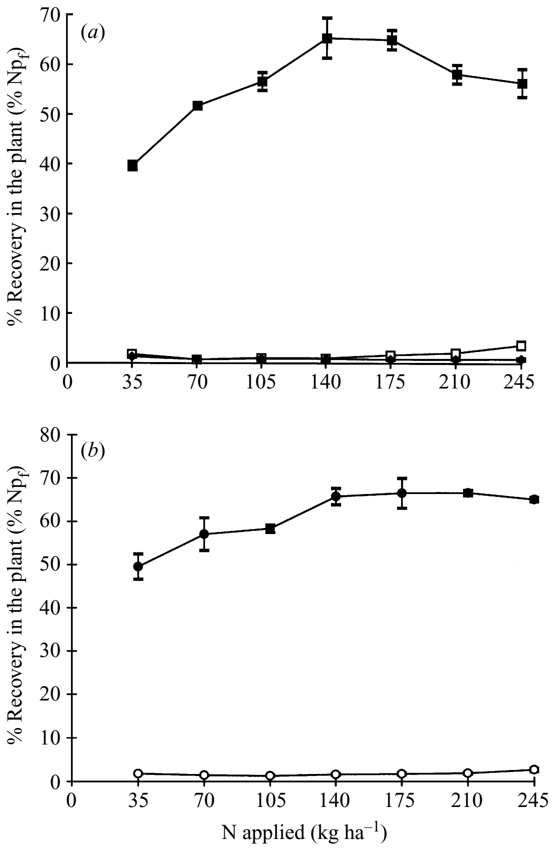

Fig. 1. Proportion of labelled N recovered in the plant (% Np_f) (*a*) at harvest in the year of application, 1992–APP (■), in the first residual year, 1993–R1 (□) and in the second residual year, 1994–R2 (◆) and (*b*) at harvest in the year of application, 1993–APP (●) and in the first residual year, 1994–R1 (○); data are the means of three blocks.

observed that labelled inorganic N in the topsoil at harvest was only 6% of the total labelled N in the topsoil.

The trend in *proportional* retention of labelled N in the topsoil (% Ns_f) with N rate was similar for both the 1992 and 1993 applications in the year of application and in the first and second residual years (Figs 2*a*, 3*a*). On all occasions, a significant decrease in the percentage retention of labelled N (% Ns_f) was observed with increasing N application up to 105 kg ha^{-1}, but a further increase in N application rate did not cause a significant change in %Ns_f. There was therefore a consistent and significant increase in the absolute amount of labelled N recovered with N application rate (Ns_f; Figs 2*b*, 3*b*).

Carbon-limited immobilization might be expected to produce an asymptote in labelled soil N with increasing N application rate but, as seen in Figs 2*b* and 3*b*, this was not evident. These results are in contrast to those observed on the Broadbalk field trial at Rothamsted where a significant *decrease* in the proportion of labelled N immobilized in the topsoil was observed with increasing N rate (up to 196 kg ha^{-1} y^{-1} N applied for *c*. 140 years; Powlson *et al*. (1986)); the value of Ns_f tended towards an asymptote with N application rate which suggested carbon-limited immobilization. At Ropsley, the total capacity for immobilization may therefore have been greater in plots which had received high rates of N application for a number of years. Alternatively, a greater amount of labelled N may have been in the inorganic form or retained in root residues or other highly labile organic forms following large N applications (Figs 2*b*, 3*b*).

Between harvest APP and R1, there was a fairly constant reduction in % Ns_f of, on average, 16 ± 1·4% (1993–R1; Fig. 2*a*) and 11 ± 0·% (1994–R1; Fig. 3*a*). The trend in the amount of labelled N recovered in the topsoil at harvest R1, with increasing N applications, was similar to that at harvest APP but a divergence was observed at N applications > 140 kg ha^{-1} between the two years (Figs 2*b*, 3*b*). The difference between Ns_f–APP and Ns_f–R1 (ΔNs_f) therefore showed a substantial increase at N applications > 140 kg ha^{-1} (Figs 2*c*, 3*c*), which suggests greater N lability of post-harvest (APP) residues at these greater applications. There was no significant difference between Ns_f at harvest 1993–R1 and 1994–R2 (Fig. 2*a*), indicating virtually no loss of labelled N from the soil after the first residual year.

Loss of labelled N (NL_f)

Losses in the year of application (NL_f–APP)

On average, 11·5 ± 1·8% and 13·3 ± 1·7% of the labelled N applied was lost over the 1992–APP and 1993–APP growing seasons respectively (Tables 3 and 4). These losses occurred either by leaching below 23 cm, denitrification or volatilization. The soil pH was 6·8 and the labelled $^{15}NH_4{}^{15}NO_3$ was applied in solution, so volatilization from the soil surface was probably minimal. Loss of labelled N by leaching was probably also minimal as there was a positive soil moisture deficit (SMD) following MTD in both 1992 and 1993; the mean SMDs for April and May were 11·3 and 61·6 mm in 1992 and 7·9 and 44·2 mm in 1993. Furthermore, the proportion of labelled N lost decreased significantly with increasing N rate (Table 6), which suggests denitrification rather than leaching. Denitrification is largely controlled by the microbial demand for oxygen and therefore by carbon supply so that the quantity of N lost is independent of the amount of labelled inorganic N added, provided the latter is not limiting (Jenkinson *et al* 1985). The low air-filled porosity at field capacity (8%) and low available water capacity (163 mm m^{-1}) of the Beccles series soil at Ropsley would also promote a rapid return to field capacity in the topsoil following rainfall

Table 5. *Examples of the recovery of* ^{15}*N-labelled fertilizer over several years in trials with wheat or barley; results are averages of the different treatments studied and are expressed as a percentage of the N applied*

		Np_f(%)				Ns_f(%)			
Reference	Treatments studied	APP	R1	R2	R3	APP	R1	R2	R3
Dowdell & Crees (1980)	Winter wheat, 3 different cultivation techniques, 80 kg ha^{-1} N	61·0	0·63	—	—	8·63	7·80	—	—
Riga *et al.* (1980)	Winter wheat, 100 kg ha^{-1} of $NaNO_3$ or $(NH_4)SO_4$ split applied in 2 ways (4 treatments)	53·0	1·44	1·42	0·78	14·0	12·8	11·9	10·9
Khanif *et al.* (1984)	Winter barley, 50 kg ha^{-1} N	57·0	—	—	—	32·0	—	—	—
Dowdell & Webster (1984)	Spring barley, 2 N rates (112 & 168 kg ha^{-1})	47·0	1·95	0·75	0·8	35·2	29·8	27·8	26·3
Powlson *et al.* (1986)	Winter wheat, 4 N rates (48, 96, 144 & 192 kg ha^{-1}) applied in 2 consecutive years	59·1	—	—	—	19·2	—	—	—
Recous *et al.* (1988*b*)	Winter wheat, 50 kg ha^{-1} $^{15}NH_4NO_3$ or $NH_4{}^{15}NO_3$ applied in March and 110 kg ha^{-1} $^{15}NH_4NO_3$ or $NH_4{}^{15}NO_3$ applied in April	46·6	—	—	—	27·8	—	—	—
Destain *et al.* (1989)	Winter wheat, 135 or 180 kg ha^{-1} N split applied in 3 ways	76·0	—	—	—	19·0	—	—	—
Recous *et al.* (1992)	Winter wheat, 80 kg ha^{-1} $^{15}NH_4NO_3$ or $NH_4{}^{15}NO_3$ applied to 3 different sites	48·5	—	—	—	22·4	—	—	—
Powlson *et al.* (1992)	Winter wheat, 2 sites: Saxmundham & Woburn, 142 or 150 kg ha^{-1} N, applied in 2 consecutive years	59·5	—	—	—	22·5	—	—	—
Powlson *et al.* (1992)	Winter wheat, Rothamsted multifactorial, an average of 216 kg ha^{-1} N applied in 4 consecutive years	71·2	—	—	—	13·5	—	—	—
Hart *et al.* (1993)	Winter wheat, 144 & 192 kg ha^{-1} applied to Broadbalk and 144 kg ha^{-1} applied to Saxmundham in 2 consecutive years	60·3	1·52	0·82	0·50	19·4	15·3	14·8	11·2
Hart *et al.* (1993)	Winter wheat, 150 kg ha^{-1} applied to Woburn in 2 consecutive years	60·9	1·55	0·90	0·50	23·0	10·5	11·0	12·4
Esala (1994)	Spring wheat, 4 N rates (45, 90, 135 & 180 kg ha^{-1})	55·5	6·50	< 1·00	—	35·5	19·3	19·7	—
Bhogal *et al.* (1995)	Winter wheat, 7 N rates (35, 70, 105, 140, 175, 210 & 245 kg ha^{-1}) applied in 2 consecutive years	58·7	1·61	0·91	—	30·0	15·4	16·3	—

APP, year of application of ^{15}N fertilizer; R1–R3, residual years 1–3; Np_f, labelled N in the plant at harvest; Ns_f, labelled N in the soil at harvest.

and active denitrification at field capacity. A number of other ^{15}N field experiments have attributed the majority of the labelled N losses observed at harvest–APP to denitrification in the period shortly after application (Dowdell & Crees 1980; Colbourn *et al.* 1984; Dowdell & Webster 1984; Khanif *et al.* 1984; Destain *et al.* 1989; Addiscott & Powlson 1992).

Losses in the residual years (NL_f–*R1/R2*)

Plant uptake of residual labelled N at Ropsley did not explain the considerable reduction in labelled soil N observed between harvests APP and R1 (Figs 1, 2 and 3). On average, 44·6 ± 8·8% and 44·3 ± 3·5% of the labelled N remaining in the soil at harvest 1992–APP and 1993–APP (respectively) was lost by harvest–R1. These losses were associated with exceptionally large amounts of rainfall and drainage over the 1992/93 and 1993/94 winters (Table 7) which may have caused substantial denitrification and leaching. In addition, the analysis of plant material in spring 1994–R1 (prior to unlabelled fertilizer application) revealed that the majority of residual labelled N uptake (96%) occurred later, from mineralization during late spring and summer following fertilizer application (Table 8).

By contrast, Dowdell & Crees (1980), Riga *et al.* (1980), Dowdell *et al.* (1984) and Hart *et al.* (1993) all observed smaller losses of residual labelled N: only 0–13% of the labelled N in the soil at harvest–APP was lost by harvest–R1 in these studies (Table 5). However, large losses of residual labelled N have also been observed in several studies. For example, Macdonald *et al.* (1992) observed that considerable loss of labelled N occurred during the first residual year when wheat followed wheat: 12, 42, 46 and 52% of the labelled N remaining in the soil at harvest–APP (1988) was lost from a clay loam, a chalky loam, a heavy clay and a sandy loam (respectively) during the

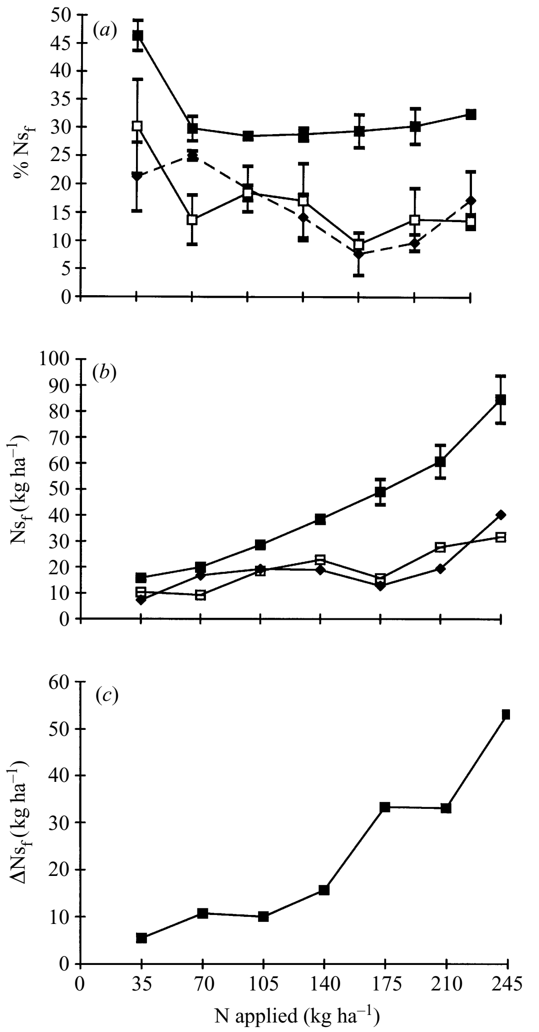

Fig. 2. (*a*) Proportion of labelled N recovered in the topsoil (% Ns_f, 0–23 cm) and (*b*) total labelled N recovered in the topsoil (kg ha^{-1} Ns_f, 0–23 cm) at harvest in the year of application, 1992–APP (■), the first residual year, 1993–R1 (□) and the second residual year, 1994–R2 (◆). (*c*) Loss of labelled N from the topsoil (ΔNs_f, 0–23 cm) between harvest–APP and R1 ($\Delta Ns_f = [Ns_f\text{–APP}] - [Ns_f\text{–R1}]$); data are the means of three blocks.

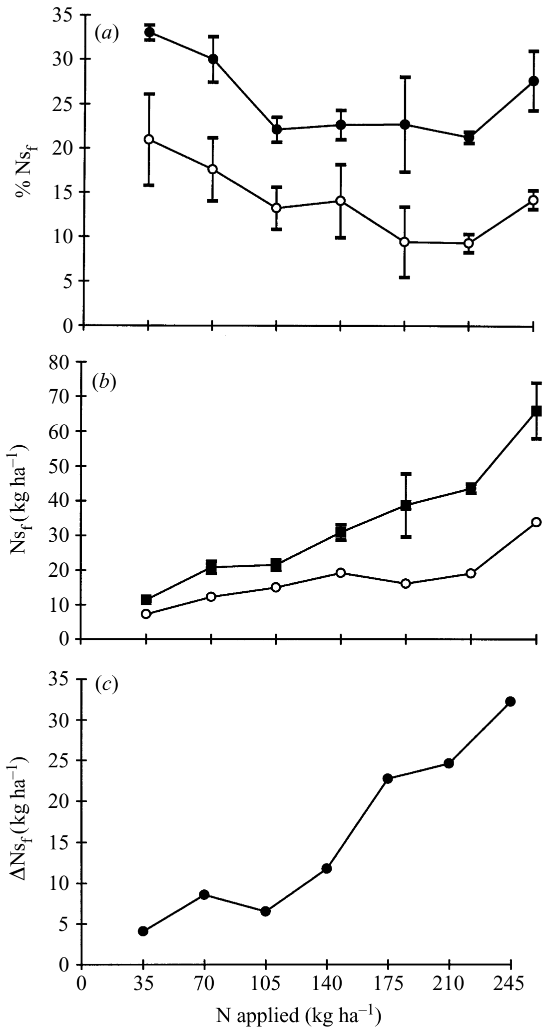

Fig. 3. (*a*) Proportion of labelled N recovered in the topsoil (% Ns_f, 0–23 cm), and (*b*) total labelled N recovered in the topsoil (kg ha^{-1} Ns_f, 0–23 cm) at harvest in the year of application, 1993–APP (●) and the first residual year, 1994–R1 (○). (*c*) Loss of labelled N from the topsoil (ΔNs_f, 0–23 cm) between harvest–APP and R1 ($\Delta Ns_f = [Ns_f\text{–APP}] - [Ns_f\text{–R1}]$); data are the means of three blocks.

first residual year (1989). Similarly, Hart *et al.* (1993) observed that 48% of the labelled N in the soil at harvest–APP was lost by harvest–R1 from a site at Woburn (Table 5). In the latter study, there was a high proportion of labelled N in the soil at harvest–APP (due to poor crop growth), the incorporated stubble had a low C:N ratio and decomposition was rapid due to a small soil clay content. Similar results were also observed by Esala (1994), where 45% of the residual labelled N was lost over the first residual year (Table 5). These losses were associated with a high proportion of labelled inorganic N in the soil at harvest–APP (due to poor crop growth). In the latter two experiments, as at Ropsley, Ns_f remained virtually unchanged after the first residual year and losses were negligible. This suggests that the labelled N remaining in the soil at harvest–R1 is present in more stable organic forms than at harvest–APP and is less readily mineralized and lost over winter or taken up by subsequent crops.

Relative availabilities of residual labelled and native soil N

Residual fertilizer N contributed little to plant uptake of soil N ($Np_{f(res)}$; Fig. 4). However, when the uptake of *native* soil N (Np_s^*) and residual fertilizer N ($Np_{f(res)}$) were compared to the total amounts in their respective pools (Eqns (1–3)), the residual fertilizer was on average 3 to 4 times more 'available' for plant uptake in comparison to the native soil N during the first residual year (availability ratio = 3·41 ± 0·48 %; Table 9).

Leitch & Vaidyanathan (1983) calculated the ratio of the percentage residual organic ^{15}N mineralized to the percentage native soil organic N mineralized in a 14-day anaerobic incubation (32 °C) of soil sampled from the 0–5 cm layer of an ^{15}N field experiment, at harvest–APP. In accordance with the crop recovery results from Ropsley, residual organic ^{15}N was seen to be three times more mineralizable than native soil organic N. Both van Praag *et al.* (1980) and Shen *et al.* (1989) observed availability ratios of 7·8 (0–10 cm soil) and 7·0 (0–23 cm) in similar incubation studies using soils collected at harvest–APP of ^{15}N experiments carried out in Belgium and on Broadbalk (UK) respectively.

The measurement of the average availability of residual fertilizer N at Ropsley, was based on the *uptake* of residual fertilizer N (Np_f–R1^{1993}; Eqn (3)). However, as already observed, the majority (≈ 96 %) of this uptake occurred from mineralization during the summer following (unlabelled) fertilizer application (Table 8). If the loss of residual labelled N seen at harvest–R1 occurred in the (previous) winter, after harvest–APP and before fertilizer application in spring, then the relative availability of the residual N would be underestimated by the calculation in Eqn (3). Furthermore, uptake of residual N (after spring) does not give a true measure of the total mineralization of residual N; losses of labelled N over the winter period would also have been due, mainly, to mineralization of labelled organic N. In order to correct this underestimation, the availability ratio was multiplied by the ratio (R, Table 9) of labelled N retained in the soil at harvest–APP (Ns_f–APP + $Nstub_f$–APP) to the labelled N recovered in the crop and soil at harvest–R1 (Ns_f–R1 + $Np_{f(res)}$; Eqn (5), Table 9). The resulting 'corrected' availability ratio

Table 6. *Proportion of labelled N unaccounted for (%NL_f) at harvest in 1992–APP and 1993–APP*

Nominal N rate (kg ha^{-1})	% NL_f 1992–APP	% NL_f 1993–APP
35	13·9	17·7
70	18·4	12·9
105	14·8	20·6
140	5·71	11·5
175	5·37	10·6
210	11·3	11·8
245	10·8	7·01

Table 7. *Total rainfall and drainage (hydrologically effective rainfall) over the 1992/93 and 1993/94 winters at Ropsley. Data obtained from the Meteorological Station at RAF Cranwell*

Autumn/winter	Rainfall (mm)	Drainage (mm)
1/9/1992 – 1/3/1993	427	193
1/9/1993 – 1/3/1994	462	240
1978–1993 mean	325	130

Table 8. *Total N uptake (Np) and uptake of residual labelled N ($Np_{f(res)}$) by winter wheat in spring (S), prior to fertilizer application (15/3/94) and at harvest–R1 (H) (9/8/94)*

N applied (kg ha^{-1})	N uptake (kg ha^{-1}) Np (S)	$Np_{f(res)}$ (S)	Np (H)	$Np_{f(res)}$ (H)
35	2·98	0·03	70·6	0·60
140	3·42	0·08	154	2·18
245	4·58	0·19	235	6·57

Np, nitrogen in the plant (kg ha^{-1}); $Np_{f(res)}$, labelled nitrogen in the plant which originated from the fertilizer applied the previous year (kg ha^{-1}); S, spring, prior to fertilizer application (15/3/94); H, harvest–R1 (9/8/94).

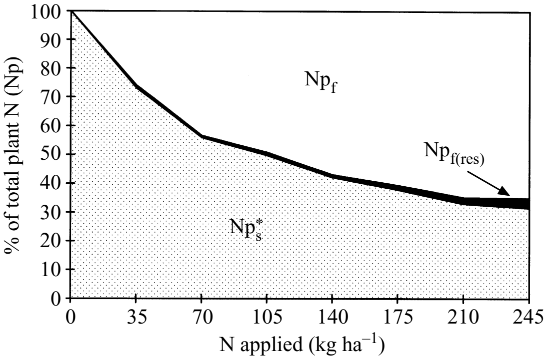

Fig. 4. Uptake of nitrogen from the native soil N pool (Np_s^*, ▨), residual fertilizer N pool ($Np_{f(res)}$, ■) and current labelled fertilizer N (Np_f, □) by the crop at harvest 1993, expressed as a percentage of the total plant N (Np); data are the means of three blocks.

Table 9. *The relative availabilities of residual and native soil nitrogen for crop uptake*

Nominal N applied (kg ha^{-1})	Availability ratio	R	Corrected availability ratio
35	3·96	2·16	8·55
70	2·03	1·76	3·57
105	2·74	2·03	5·56
140	2·22	2·14	4·75
175	3·63	2·95	10·7
210	4·40	2·96	13·0
245	6·21	2·04	12·7

See Eqns (1–3) for the calculation of the availability ratio. R, ratio of labelled N retained in the soil at harvest–APP to the recovery of residual labelled N in the crop and soil at harvest–R1.

$$R = \frac{(Ns_f + Nstub_f) - APP}{(Ns_f + Np_{f(res)}) - R1} \qquad (5)$$

Corrected availability ratio = Availability ratio × R.

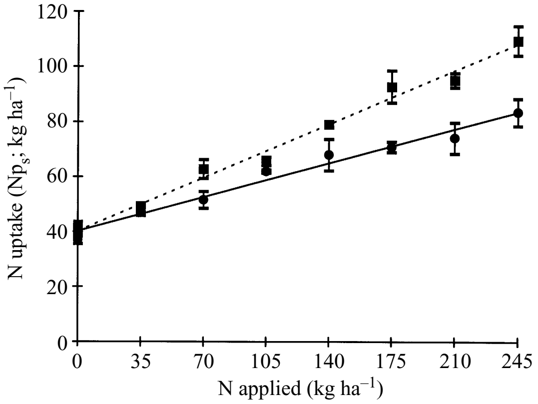

Fig. 5. Uptake of unlabelled N (Np$_s$) at harvest 1992–APP (■) and 1993–APP (●) as a function of N application; data are the means of three blocks; $Y = 0{\cdot}279X + 40{\cdot}212$ in 1992–APP (- - - -) and $Y = 0{\cdot}178X + 40{\cdot}167$ in 1993–APP (—).

therefore describes the bio-availability of ^{15}N residues present in the soil in spring relative to the availability of the soil humus N. It is based on the assumption that all loss of residual labelled N measured at harvest–R1 occurred prior to any uptake by the crop during the first residual year. The availability ratio was thus increased to an average of 8·40 ± 1·46 (Table 9), which is similar to the findings of van Praag *et al.* (1980) and Shen *et al.* (1989). Either measure of relative availability is merely an operational definition. The ^{15}N residues are clearly highly heterogeneous and must possess a range of labile characteristics. The most

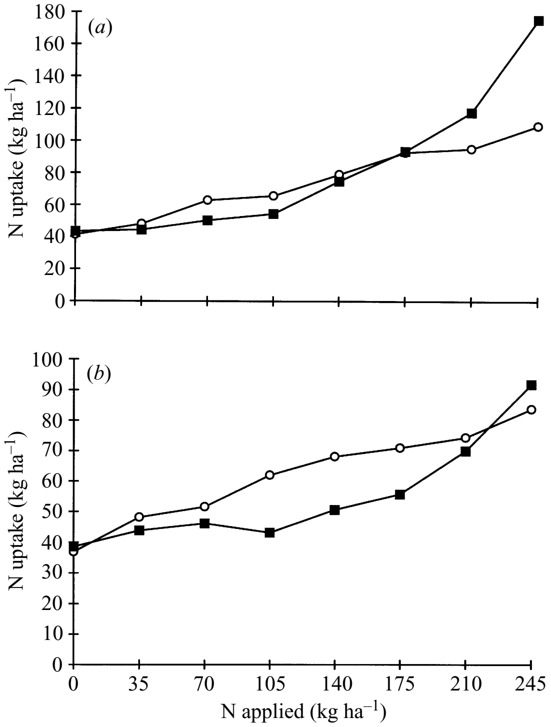

Fig. 6. Uptake of unlabelled N from the ^{15}N-labelled microplots (Np$_s$, ○) and the total N uptake from hypothetical holiday plot controls (Np$_s^{0H}$, ■) in (*a*) 1992–APP and (*b*) 1993–APP.

available fraction(s) of the N residues is lost over winter during a period of minimal plant demand and is therefore not included in the estimate of residual N availability.

Uptake of unlabelled N by the crop (Np$_s$) in the year of ^{15}N application (1992–APP and 1993–APP)

A significant increase in unlabelled N uptake (Np$_s$) with N application rate was observed in both 1992–APP and 1993–APP ($P < 0{\cdot}001$; Fig. 5). An increase in unlabelled N uptake due to the application of N fertilizer is frequently observed in ^{15}N experiments (Dowdell & Crees 1980; Riga *et al.* 1980; Smith *et al.* 1984; Powlson *et al.* 1986, 1992; Rao *et al.* 1991) and is commonly termed an *Added Nitrogen Interaction* or ANI (Jenkinson *et al.* 1985). An ANI can be *real* or *apparent*, positive or negative and can occur in the presence or absence of plants. A *real* ANI occurs when fertilizer N causes a net change in a process which affects soil N availability. An *apparent* ANI arises as a result of a change in the *proportions* of labelled and unlabelled N utilized in any transfer mechanism with no net change in the magnitude of the process. For example labelled N may be

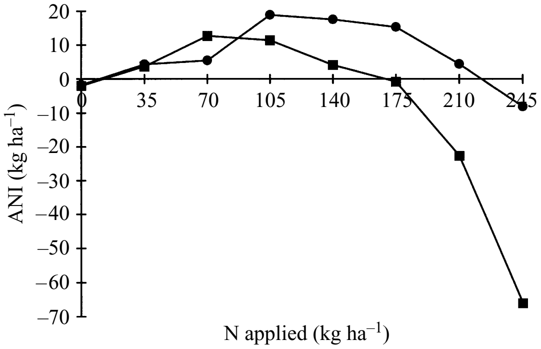

Fig. 7. The added nitrogen interaction (ANI) at Ropsley in 1992–APP (■) and 1993–APP (●), calculated using the N uptake from the hypothetical control microplots (ANI = $Np_s - Np_s^{0H}$).

immobilized or denitrified in place of soil N (pool substitution), so that a greater proportion of the (unlabelled) soil N is free to be taken up by the plant (Powlson & Barraclough 1993), resulting in a positive apparent ANI.

It is well recognised that different N application rates will cause a variation in the residual N available to a following crop. This may apply following a single year, or possibly a number of years, of different fertilizer N treatments. For example, Powlson *et al.* (1986), working on soils from the Broadbalk experiment that had received large applications of N for over 140 years, associated an increase in Np_s with increasing N application. At Ropsley, a significant effect of previous N fertilizer history on both grain yield and N offtake was observed in the 'holiday' plots when fertilizer was withdrawn for 1 year. In both 1992 and 1993 there was a significant increase in N uptake from the holiday plots (Np_s^{0H}) with increasing *historical* N application level (Fig. 6*a*, *b*). The difference between soil N uptake from the ^{15}N fertilized plots (Np_s) and the holiday plots (Np_s^{0H}) (Fig. 6*a*, *b*) gives a measure of the ANI ($Np_s - Np_s^{0H}$) which is independent of any historical effects of N application (Fig. 7). However, it is not possible to ascribe this effect to either a *real* or *apparent* ANI unequivocally.

At N applications < 175 kg ha^{-1} in 1992 and 210 kg ha^{-1} in 1993, the addition of fertilizer caused an increase in uptake of unlabelled N (Fig. 6*a*, *b*) and therefore a *positive* ANI (Fig. 7). The magnitude of this ANI was only 4–13 kg ha^{-1} in 1992 and 4–19 kg ha^{-1} in 1993. This was considerably less than the increased soil N uptake calculated using the *long-term* zero N plot as a control, instead of the appropriate holiday plot (7–38 kg ha^{-1} in 1992–APP and 11–31 kg ha^{-1} in 1993–APP; Fig. 5). On average, therefore, 40–50% of the increase in soil N uptake (Np_s) over that of the long term control plot (N1), was due to the residual effect of N fertilizer, probably from the previous year. This may be called a *residual* ANI (ANI_{res}); the remainder of the increase in Np_s (over control, N1) was probably the result of pool substitution.

The proportion of *fertilizer* N recovered by the crop reached a maximum at *c.* 140 kg ha^{-1} N and, from Fig. 7, it is clear that the mid-range N applications also corresponded with a reversal in the trend of ANI with N application. The change from a positive to negative ANI with increasing N rate was gradual, there was no clear critical N level at which the ANI reached a maximum: 70–105 kg ha^{-1} in 1992 and 105–140 kg ha^{-1} in 1993. As N applications increase, the carbon-limited processes of immobilization and denitrification, which lead to pool substitution and the positive ANI observed, eventually become satisfied almost wholly by labelled N. Up to this point, unlabelled soil N would be made progressively more available for plant uptake, to give a positive apparent ANI. This should reach an asymptote as the maximum immobilization rate of unlabelled N was reached and there would be no further increase in soil N availability with N application. Still greater N applications therefore should not cause any further increase in fertilizer immobilization and the increased soil N availability would become constant. However, as plant N demand becomes satisfied with increased N application, the positive ANI is progressively reduced as labelled N increasingly excludes soil N from uptake by the crop. The ANI is zero when the extra soil N made available by pool substitution exactly balances the suppression of demand for soil N caused by the availability of labelled N to plants with a limited demand. Further suppression of soil N uptake then causes a negative ANI. Thus at N applications greater than 175 kg ha^{-1} (1992) or 210 kg ha^{-1} (1993) the addition of fertilizer caused a net *suppression* of soil N uptake and thus a *negative* ANI (Fig. 7).

However, the retention of labelled fertilizer in the soil at Ropsley did not reach an asymptote (Figs 2 and 3). This might suggest that immobilization had not reached a maximum, perhaps due to greater intrinsic immobilization capacity in the historically higher yielding (high N) plots or simply greater current carbon supply from enhanced root growth. Nevertheless, at high N rates, the effect of suppression of unlabelled N uptake due to limited plant demand exceeded any increased availability of unlabelled N due to continuing pool substitution (a negative ANI). Alternatively, a greater proportion of labelled N retention at first harvest (Ns_r–APP) may simply have been inorganic N. There was no significant effect of N application rate on the inorganic N content of the topsoil (0–23 cm) in autumn 1992 which was, on average, 14·2 ± 0·45 kg ha^{-1} across all the N rates.

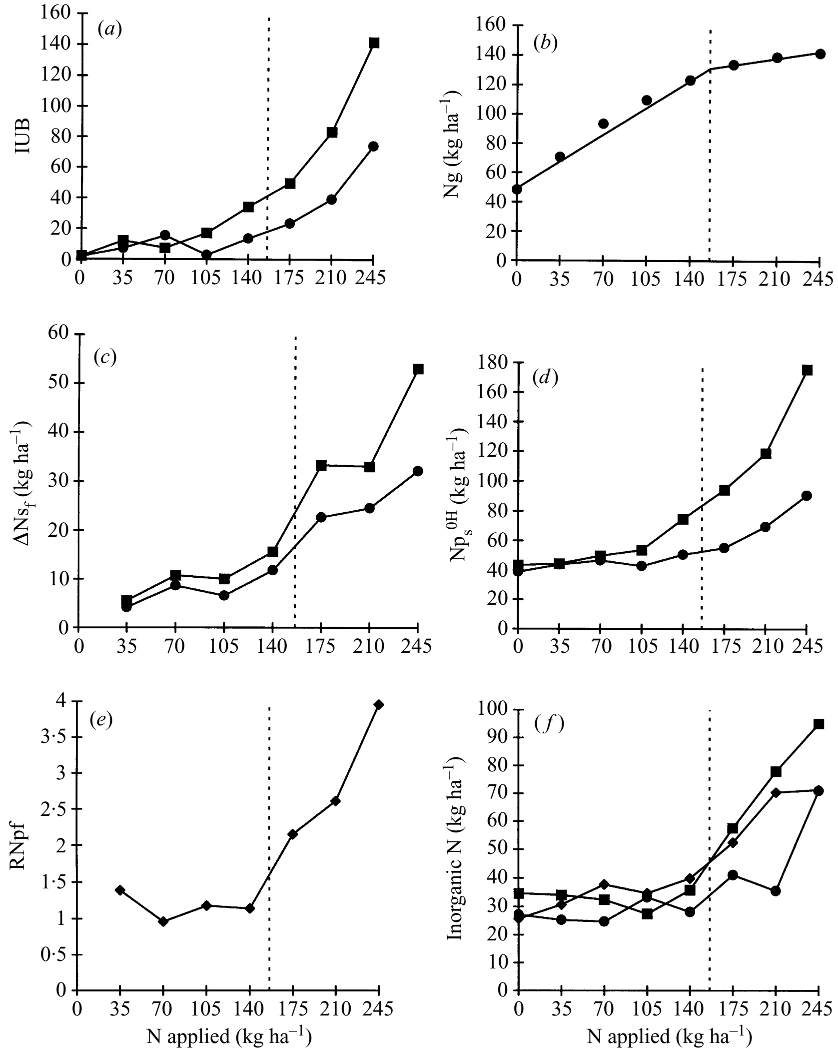

Fig. 8. Several analogues of nitrogen-use efficiency, as a function of N application rate including: (*a*) immobilization⇔uptake balance (IUB) at Ropsley in 1992–APP (■) and 1993–APP (●); IUB = $Ns_f - ANI$. (*b*) Average grain N offtake (Ng) by second wheat (●) at Ropsley from 1978 to 1990; solid line = the two-straight-line fitted as described in Bhogal *et al.* (1995). (*c*) Change (loss) in labelled N in the topsoil (0–23 cm) between the first and second residual years. ■ = 1992; ● = 1993. (*d*) Total N uptake from the hypothetical holiday microplots (Np_s^{0H}) in 1992–APP (■) and 1993–APP (●). (*e*) Ratio of labelled N uptake between the first and second residual years ($RNp_f = Np_{f(res)}R1 : Np_{f(res)}R2$). (*f*) Inorganic N in the soil profile in March 1992 (■), November 1992 (◆) and March 1993 (●). Broken vertical line = the average Nf^{break} from 1978 to 1990 (156±7·11 kg ha^{-1}).

However this measurement was taken in November, over 2 months after the measurement of Ns_f at harvest, so some inorganic N may already have been lost from the topsoil.

Esala (1994) also observed decreases in soil N uptake (a *negative* ANI) by spring wheat with increase in N application rate (0–180 kg ha^{-1}) in three separate years of experiments with ^{15}N-labelled fertilizer in Finland (1990–92). These decreases occurred when the grain yield response to N fertilizer reached an asymptote or declined. In both 1990 and 1992, grain yields were severely restricted by drought, so that the plant N demand was low and uptake of fertilizer (and soil) N was incomplete. Thus large amounts of labelled fertilizer-N were observed in the soil at harvest. In 1991, crop yields were not limited by drought and a negative ANI did not occur until N was applied in excess of the estimated optimum requirement of 90 kg ha^{-1}. At N applications less than 90 kg ha^{-1}, a positive ANI was observed – i.e. the application of fertilizer increased soil N uptake (Np_s). The results of Esala (1994) are thus similar to those observed at Ropsley in 1992 and 1993, where a positive ANI was observed at low N rates and a negative ANI at N application rates in excess of the optimum.

Immobilization ⇔ Uptake balance (*IUB*)

The apparent balance between labelled N immobilization and *additional* uptake of soil N as a result of pool substitution (IUB = Ns_f–ANI; Fig. 8*a*) was calculated in order to examine the possible effect of pool substitution on soil N availability. 'Immobilization' here is equated with fertilizer N retention in autumn–APP (0–23 cm layer) and includes any inorganic N present at the time of measurement. The trend shown in Fig. 8*a* results from fertilizer N progressively dominating both the immobilization and plant N uptake sinks as N applications are increased. At N applications < 140 kg ha^{-1}, the increase in soil N uptake by the crop, over that of the control holiday plots, was comparable to the amount of fertilizer N immobilized in the soil. This is the result of a constant efficiency of N uptake over this N application range (Bhogal *et al.* 1995) coupled with immobilization of labelled fertilizer N in place of unlabelled soil N. The similarity of ANI and Ns_f supports the interpretation of ANI as an *apparent* ANI. The efficiency of N uptake declined at N applications > 160 kg ha^{-1} with a corresponding increase in IUB. The latter trend could have been the result of one or more of the following processes.

1. Decline in ANI (Np_s–Np_s^{0H}) with large applications of fertilizer N.

   At high N rates, plant uptake is not proportional to the total inorganic N pool. Therefore, a decline in the uptake of soil N (Np_s) relative to the control N (Np_s^{0H}) could result from a decreasing proportion of unlabelled N being taken up by the plants.
2. Increase in labelled N retention in the soil (Ns_f).
   - (i) At large N application rates, greater residual labelled *inorganic* N may be present in the soil at the time of measurement.
   - (ii) Greater labelled root N in the soil at large N rates would be included in the soil sampling procedure and assayed as Ns_f.
   - (iii) An increase in immobilization of labelled N at the larger N rates would constitute a *real* ANI and could arise due to a greater return of crop residues or a higher humus content in the microplots receiving large N applications or greater carbon supply from root exudates etc in the year of study.

Residual effects of high N fertilizer rates over 16 *years* (*1978–94*) *at Ropsley*

Analysis of historical data from the 1978–90 Ropsley field trial by Bhogal *et al.* (1995) revealed that the intersection of a two-straight-line function fitted to the variation in grain N offtake with N applied (termed the breakpoint or Nf^{break}), occurred at an average N application of *c.* 160 (156 ± 7·11) kg ha^{-1} for the second wheat crop in rotation (Fig. 8*b*). This level of N application marks a change in the efficiency of N use and coincides with the N rate where changes in parameters of the ^{15}N balance were also observed at Ropsley from 1992 to 1994 (Fig. 8*a*–*f*). Thus, the increase in IUB (Fig. 8*a*), the decline in labelled N in the topsoil between harvest–APP and R1 (ΔNs_f; Fig. 8*c*), the increase in N uptake by crops grown on holiday plots receiving no N (Np_s^{0H}; Fig. 8*d*) and the marked increase in the ratio of labelled N uptake between the first and second residual years (RNp_f; Fig. 8*e*) all provide evidence of greater amounts and increased lability of residual fertilizer N when N applied > Nf^{break}. There was also a conspicuous increase in the inorganic N content of the soil profile (0–90 cm) at N applications > 160 kg ha^{-1} measured by ADAS in November and March, prior to fertilizer application (Fig. 8*f*). The latter results are very similar to the pattern demonstrated by other recent studies (e.g. Chaney 1990).

Bhogal *et al.* (1995) also observed a significant increase in the *total* organic N content of the topsoil at N applications > 160 kg ha^{-1} at Ropsley, relative to control plots. There was apparently an annual rate of N accumulation (over a period of 14 years, 1978–1992) equivalent to *c.* 8% of the N applied annually for the two largest N applications (15 and 21 kg ha^{-1} y^{-1}). Thus inefficient N fertilizer use at N applications > 160 kg ha^{-1} not only caused a significant increase in residual labile N but also led to the gradual accumulation of total N in the soil.

The results from the ^{15}N experiment carried out at Ropsley support the earlier findings of Bhogal *et al.* (1995) that suggested a marked change in the efficiency of N use at N applications > 160 kg ha^{-1}. The increase in residual N availability at these applications will not only affect the nutrition of subsequent crops (and should therefore be taken into account when predicting fertilizer requirements) but can increase the risk of N losses by leaching or denitrification over winter. The results also highlight the problems associated with interpreting field experiments with ^{15}N where an increase in unlabelled N uptake with N applied (an ANI) is often observed. The use of plots where fertilizer was withdrawn for a year enabled the division of the ANI into that arising from the short or long term residual effect of previous applications (ANI_{res}) and that due to an apparent or real (single year) ANI.

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