Evaluating Nutrient Availability in Semi-arid Soils
With Resin Capsules and Conventional Soil Tests

Mary Pletsch Jones

A thesis submitted to the faculty of
Brigham Young University
in partial fulfillment of the requirements for the degree of
Master of Science

Von D. Jolley, Chair
Bruce L. Webb
Bryan G. Hopkins
Phil S. Allen

Department of Plant and Wildlife Sciences
Brigham Young University
August 2011

Copyright © 2011 Mary Pletsch Jones
All Rights Reserved
ABSTRACT

Evaluating Nutrient Availability in Semi-arid Soils With Resin Capsules and Conventional Soil Tests

Mary Pletsch Jones
Department of Plant and Wildlife Sciences, BYU
Master of Science

Commonly used soil analysis and resin capsule procedures are used to assess nutrient status in fertile soils, but their validity in semi-arid ecosystems is unknown. Three studies were performed to assess resin capsule effectiveness in semi-arid ecosystems. An incubation study was completed in which loamy sand and sandy clay loam soils were treated with rates of N, P, Fe and Zn. Each soil treatment was implanted with a resin capsule and incubated for 60 or 120 days. Resin capsules reflected NH4-N and P fertilizer at low rates in the loamy sand. NO3-N reflected rates in both soils, but did not reflect Fe or Zn application. Resin capsule NH4-N was a better indicator than KCl-extractable NH4-N, but resin capsule NO3-N was not as effective as water extraction, and resin capsule P was poor compared to NaHCO3-P. A second study was performed in glasshouse conditions using the incubation study soils. Soils were treated with rates of N, P and resin capsules were placed in pots. Pots were seeded with squirreltail grass (Elymus elymoides) and placed in a glasshouse. Resin capsules were removed at 120 days, soil samples taken, grass harvested and yield measured. Yield and total nutrient removal was correlated to resin NH4-N, marginally related to resin or soil NO3-N, and unrelated to resin P. Yield and total nutrient removal was correlated with application rates and resin NH4-N and NaHCO3-extracted P. The third field study, compared two sites with rates of N and P application were established on clay loam and sandy loam soils. Resin capsule and conventional soil tests for NO3-N, NH4-N and P were measured and plant nutrient status examined. Resin capsules were removed and replaced and soil samples taken every 90 days. Resins P was not related to P application or to plant tissue P but NaHCO3-extracted P was, while resin NO3-N, KCl-extracted NO3-N and NH4-N were correlated to N application and plant N. Soil test P was more effective in predicting P status and bioavailability than resin capsules. Resin NH4-N and NO3-N predicted N status and bioavailability, but soil tests were just as effective in semi-arid conditions.

Keywords: adsorption, desert soil, nitrogen, phosphorus, plant available nutrients and resin capsule.
ACKNOWLEDGEMENTS

This project has been an extensive process, including several years of research, in which numerous individuals have contributed to the success of this research. Above all I would like to thank Bruce L. Webb and Von D. Jolley for their extensive involvement and supervision of this study. I would like to thank the Brigham Young University Plant and Soil Analysis Laboratory for their dedicated assistance to the entire research project, in addition to the resin, soil and plant analysis performed. Multiple professors and undergraduate researchers put extensive time into the incubation, greenhouse and field studies. I would also like to thank my graduate committee for their advice and contributions to my research and thesis. Finally, I would like to thank my husband, family and friends for their support in my scholastic endeavors. I have learned so much from my associations with professors and academic course studies.

Research was funded by the Brigham Young University Plant and Soil Analysis Laboratory and the Brigham Young University Department of Plant and Wildlife Sciences.
Table of Contents

Title Page.........................................................................................................................i
Abstract..........................................................................................................................ii
Acknowledgements........................................................................................................iii
Table of Contents...........................................................................................................iv
List of Tables...................................................................................................................viii
List of Figures..................................................................................................................x
Manuscript #1 – Evaluating Nutrient Availability in Desert Soils with Resin Capsules and
Conventional Soil Tests. I. Incubation Studies

Abstract.........................................................................................................................2
Introduction....................................................................................................................3
Materials and Methods.................................................................................................5
Results and Discussion.................................................................................................6
Conclusion.....................................................................................................................9
References.....................................................................................................................10
Table 1.........................................................................................................................11
Table 2.........................................................................................................................12
Table 3.........................................................................................................................13
Table 4.........................................................................................................................14
Figure 1.......................................................................................................................15
Figure 2.......................................................................................................................16
Figure 3.......................................................................................................................17
Manuscript #2 - Evaluating Nutrient Availability in Desert Soils with Ion Exchange Resin Capsules and Conventional Soil Tests. II. Native Plant Bioavailability Under Greenhouse Conditions

Abstract .......................................................................................................................... 19
Introduction ...................................................................................................................... 19
Materials and Methods .................................................................................................. 21
Results and Discussion ................................................................................................. 23
Conclusion ...................................................................................................................... 27
References ..................................................................................................................... 29
Table 1 ............................................................................................................................. 31
Table 2 ............................................................................................................................. 32
Table 3 ............................................................................................................................. 33
Figure 1 ........................................................................................................................... 34
Figure 2 ........................................................................................................................... 35
Figure 3 ........................................................................................................................... 36
Figure 4 ........................................................................................................................... 37
Figure 5 ........................................................................................................................... 38
Figure 6 ........................................................................................................................... 39
Figure 7 ........................................................................................................................... 40
Figure 8 ........................................................................................................................... 41
Figure 9 ........................................................................................................................... 42
Figure 10 ......................................................................................................................... 43
Manuscript #3 - Evaluating Nutrient Availability in Desert Soils with Ion Exchange Resin Capsules and Conventional Soil Tests. III. Field Studies

Abstract.................................................................................................................45
Introduction...........................................................................................................46
Materials and Methods......................................................................................49
Results and Discussion......................................................................................51
Conclusion...........................................................................................................54
References.........................................................................................................56
Table 1 ...............................................................................................................59
Table 2 ...............................................................................................................60
Table 3 ...............................................................................................................61
Table 4 ...............................................................................................................62
Table 5 ...............................................................................................................63
Figure 1 .............................................................................................................64
Figure 2 .............................................................................................................65
Figure 3 .............................................................................................................66
Figure 4 .............................................................................................................67
Appendix A
Field Experiment Randomized Block Design .................................................69
Appendix B
Procedure for Resin Capsule Extraction .......................................................71
Procedure for Ammonium and Nitrate Extraction ...........................................72
Procedure Ammonium by Latchet.................................................................73
Procedure for Nitrate by Latchet...............................................................74
Procedure for NaHCO₃-extracted P............................................................75
Procedure for Total Nitrogen Analysis......................................................77
Procedure for Perchloric Acid Plant Digestion........................................78
LIST OF TABLES

MANUSCRIPT #1

Table 1. Soil characteristics of two native soils obtained from Moab and Rush Valley, Utah that were used in the incubation study.

Table 2. N, P, Fe and Zn treatments applied to two soils and incubated with resin capsules at 50% field capacity in two volumes of soil for 60 and 120 days.

Table 3. $R^2$ values for parameters measured by resin capsules and conventional soil tests as related to N or P fertilizer application

Table 4. Iron, S, and Zn measured in resin capsules relative to an untreated control soil.

MANUSCRIPT #2

Table 1. Soil characteristics of loamy sand (Moab, Utah) and sandy clay loam (Rush Valley, Utah) soils used in greenhouse study.

Table 2. Concentrations of N (%) of the above ground portion of squirreltail grass after growing for 120 days at 0, 5.5, 11, 22 and 44 kg ha$^{-1}$ N and concentrations of resin NH$_4$-N and NO$_3$-N and KCl-extracted NH$_4$-N and NO$_3$-N used to correlate with plant N contents. Regression coefficients and $P$ values are given; $P$ values < 0.05 indicate significant relationships.

Table 3. Concentrations of P (%) of the above ground portion of squirreltail grass after growing for 120 days at 0, 11, 22 and 44 kg ha$^{-1}$ P and of resin P and NaHCO$_3$-extracted P used to correlate with plant total P. Regression coefficients and $P$ values are given; $P$ values < 0.05 indicate significant relationships.
Table 1. Selected characteristics of clay loam (Rush Valley) and sandy loam (Skull Valley) soils at the two field sites. Samples were obtained from each of five blocks (replications) prior to fertilizer application.

Table 2. Precipitation distribution for Rush Valley\(^1\) and Skull Valley\(^2\) (Utah) research sites during the period of experiments.

Table 3. Resin and traditional soil testing procedure measurements for four times (August, November 2009 and March, May 2010). Values represent the average of six treatments (0, 5.5, 11, 22, 44 and 88 kg ha\(^{-1}\) N or P applied 13 May 2009), and two locations (Rush Valley and Skull Valley, UT). Numbers with the same letter are not significantly different at \(P<0.05\), Tukey Studentized Range Test.

Table 4. Average soil P, NO\(_3\)-N, NH\(_4\)-N and (NO\(_3\) + NH\(_4\)-N) concentrations as measured using resin or soil analyses on soils and concentration of N and P in whole crested wheat plants from Skull Valley and Rush Valley Utah. Values are the average of six N and six P treatments and four sampling dates.

Table 5. R\(^2\) values for correlation of soil and resin test to plant N and P values.
LIST OF FIGURES

MANUSCRIPT #1

Figure 1. Resin capsule and KCl-extractable NH$_4$-N measured in loamy sand and sandy clay loam soils treated with 0, 11.2, 22.4 and 44.8 kg N ha$^{-1}$. Within resin capsule or KCl extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, c or d used to compare loamy sand; y and z used to compare sandy clay loam).

Figure 2. Resin capsule and KCl-extractable NO$_3$-N measured in loamy sand and sandy clay loam soils treated with 0, 11.2, 22.4 and 44.8 kg N ha$^{-1}$. Within resin capsule or water extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, c or d used to compare loamy sand; w, x, y and z used to compare sandy clay loam).

Figure 3. Resin capsule and sodium bicarbonate extractable P measured in loamy sand and sandy clay loam soils treated with 0, 22.4 and 44.8 kg P$_2$O$_5$ ha$^{-1}$. Within resin capsule or sodium bicarbonate extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, or c used to compare loamy sand; x, y and z used to compare sandy clay loam).

MANUSCRIPT #2

Figure 1. Yield of squirreltail grass 120 days after the application of 0, 5.5, 11, 22, 44 kg ha$^{-1}$ N and of 0, 11, 22 and 44 kg ha$^{-1}$ P. Values are the average of two soils.
Figure 2. Yield of squirreltail grass 120 days after application of 0, 5.5, 11, 22, 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NH\(_4\)-N. Values represent the average of two soils.

Figure 3. Yield of squirreltail grass 120 days after application of 0, 5.5, 11, 22, 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NO\(_3\)-N. Values represent the average of two soils.

Figure 4. Total N and P removal by squirreltail grass 120 days after application of 0, 5.5, 11, 22, 44 kg ha\(^{-1}\) N and 0, 11, 22, 44 kg ha\(^{-1}\) P as related to N and P application. Values represent the average of two soils.

Figure 5. Total N removal by squirreltail grass 120 days after application of 0, 5.5, 11, 22, 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NH\(_4\)-N. Values represent the average of two soils.

Figure 6. Total N removal by squirreltail grass 120 days after application of 0, 5.5, 11, 22, 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NO\(_3\)-N. Values represent the average of two soils.

Figure 7. Yield of squirreltail grass 120 days after application of 0, 11, 22, 44 kg ha\(^{-1}\) P as related to resin P and NaHCO\(_3\)-P. Values represent the average of two soils.

Figure 8. Total P removal by squirreltail grass 120 days after application of 0, 11, 22, 44 kg ha\(^{-1}\) P as related to resin and NaHCO\(_3\)-extracted P. Values represent the average of two soils.

Figure 9. Resin and KCl-extracted NH\(_4\)-N and NO\(_3\)-N after 120 days growing squirreltail grass as related to application of 0, 5.5, 11, 22, and 44 kg ha\(^{-1}\) N and resin and NaHCO\(_3\)-
extracted P 120 days growing squirreltail grass as related to application of 0, 11, 22, and 44 kg ha\(^{-1}\) P. Values represent the average of two soils.

Figure 10. Resin and NaHCO\(_3\)-extracted P after 120 days growing squirreltail grass as related to application of 0, 11, 22, and 44 kg ha\(^{-1}\) P. Values represent the average of two soils.

MANUSCRIPT #3

Figure 1. Resin and KCl-extracted soil NH\(_4\)-N measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha\(^{-1}\) N; fertilizer applied 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and four periods of time (August, November 2009 and March, May 2010).

Figure 2. Resin and KCl-extracted soil NO\(_3\)-N measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha\(^{-1}\) N fertilizer was applied 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and four periods of time (August, November 2009 and March, May 2010).

Figure 3. Concentration of N and P in Crested Wheat grass grown after application of 0, 5.5, 11, 22, 44, 88 kg N and P ha\(^{-1}\) on 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and two periods of time (June 2009 and June 2010). Linear regression was completed using SAS.

Figure 4. Resin and soil P measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha\(^{-1}\) P; fertilizer was applied 13 May 2009. Values represent the average of two locations and four sampling periods (August, November 2009 and March, May 2010).
MANUSCRIPT #1- EVALUATING NUTRIENT AVAILABILITY IN SEMI-ARID SOILS WITH RESIN CAPSULES AND CONVENTIONAL SOIL TESTS. I. INCUBATION STUDIES

(Published in Communications in Soil Science and Plant Analysis, 2011)
EVALUATING NUTRIENT AVAILABILITY IN SEMI-ARID SOILS WITH RESIN CAPSULES AND CONVENTIONAL SOIL TESTS. I. INCUBATION STUDIES

Mary P. Jones¹, Bruce L. Webb¹, Daniel A. Cook¹ and Von D. Jolley¹

¹Department of Plant and Wildlife Sciences, Brigham Young University, Provo, UT 84602

ABSTRACT
Assessing the nutrient status of low-input, low-fertility, semi-arid soils poses some unique challenges. Commonly used soil analysis procedures and resin capsules generally assess nutrient status of fertile agricultural soils. Ion exchange resin capsules (Unibest Company, Bozeman, MT, USA) provide a viable alternative to traditional soil analysis procedures. A study was conducted to determine the effectiveness of resin capsules to extract low levels of nutrients applied to native soils. Loamy sand and sandy clay loam semi-arid soils from Utah were treated with combinations of four rates of N as ammonium nitrate (34-0-0), three rates of P as phosphoric acid (0-72-0) and two rates of FeSO₄·7H₂O and ZnSO₄·7H₂O (including an untreated control). Each soil treatment was implanted with a resin capsule placed into either 250 or 1000 cm³ of soil after addition of water equivalent to 50% field capacity and incubated for either 60 or 120 d at 25°C. After the appropriate incubation time, capsules were washed and extracted using 2M HCl, and Fe, NH₄-N, NO₃-N, S, and Zn analyzed in the extract. Conventional soil tests were completed on incubated soils (60 or 120 d). Resin capsules reflected NH₄-N and P fertilizer applied, at low rates, in the loamy sand but not in the sandy clay loam. Neither Fe nor Zn application was reflected in resin capsules, but the accompanying S was clearly quantified. Comparing the resin capsule to conventional soil test procedures, resin capsule NH₄-N was clearly a better indicator than KCl-extractable NH₄-N; resin capsule NO₃-N was effective, but
not as good an indicator as water extraction; resin capsule P was reflective of soil applied P in loamy sand but not in sandy clay loam; while sodium bicarbonate was effective in both soils. Resin capsules show promise for use in low input conditions, but additional understanding of interactions in variable soils is needed.

INTRODUCTION

Forty years ago the ion-exchange resin capsule for testing soil nutrient values was introduced as a simple and quick method to analyze soils. The use of resin capsules allows for a broad array of nutrients to be analyzed after various lengths of residency in soils and was proposed as a replacement for labor intensive, expensive and lengthy traditional soil analysis procedures. Resin capsules are effective in determining nutrient status of terrestrial and aquatic ecosystems and of soils (Yang et al. 1991). The ‘ion exchange resin capsules’ were developed to provide a simple, convenient, repeatable resin methodology (Skogley et al. 1996) to measure soil nutrient supplying capacity. Resin capsules are elliptical mesh balls approximately 2 cm in diameter which contain resin beads that act as sinks for ions in soil solution in a manner similar to plant absorption of nutrients. A major purpose of the resin capsule is to correlate the plant available nutrients in the soil to that adsorbed by the resin capsule. The chemistry of the resin capsule can be described as anions (- charge) and cations (+ charge) encapsulated in resin beads. These ions when placed in the soil will exchange with other cations and anions in the soil solution. The resins contain ions of hydrogen (H⁺), hydroxide (OH⁻), and bicarbonate (HCO₃⁻) which allow exchange of anions and cations present in soils and, consequently, estimates plant available nutrients in soils (Yang et al. 1991). Much of the focus of resin capsule research has been on the macronutrients N, P, K and the micronutrients Cu, Fe, Mn, and Zn. To equilibrate with these nutrients, capsules must be buried in the soil for a specified time after which they are extracted.
chemically and the extract is analyzed to quantify the nutrients adsorbed.

The majority of the research and commercial use of resin capsules has been carried out in highly fertile soils. Use of these capsules has expanded to low input, often low precipitation ecosystems such as range and forest lands. While general concerns have been raised about the difficulty of comparing resin-based techniques and traditional soil measurements (Johnson et al. 2005; Sherrod et al. 2002), there are even greater concerns regarding their use in low-nutrient, arid soils, such as those in Southern and Western Utah. These soils and the environment in which they are found are unique compared to soils generally tested using resin capsules. Studies in these conditions have shown that measurements from resin capsules and traditional soil testing methods are not always comparable and that they should not be considered interchangeable; furthermore, interpretive differences between resin and traditional soil testing methods are poorly understood (Sherrod et al. 2002; Sherrod et al. 2003). Our interest was stimulated by extensive utilization of ion-exchange resin capsules by researchers with the U.S. Geological Survey and Brigham Young University on native low-fertility ecosystems. Previous studies (Sardi et al. 1996) showed some correlation among resin capsule soil contact time, fertilizer rate and resin nutrient concentration. Before more extensive application in low-input arid soils, further research is needed on the comparative values of these two soil test approaches and on the value of ion exchange resin capsules.

Our objective is to examine the relative abilities of resin capsules and conventional soil tests to reflect additions of low application rates of nutrients in semi-arid soils. An incubation experiment was conducted to determine if four relatively low rates of N, three rates of P and two rates of Fe and Zn would be reflected in resin capsules placed in two soils in suboptimal moisture conditions (50% of field capacity) as well as by conventional soil test analyses (two
volumes of soil and two sampling times were included to assess impacts of these parameters. This report will emphasize results comparing the two different soil types.

MATERIALS AND METHODS

Incubation Experiment

Two soils varying in characteristics and generally low in plant nutrients (Table 1) were collected from Moab and Rush Valley, UT (mesic typic Haplocalcid and mesic xeric Haplocacid respectively), air dried, and sieved (2-mm sieve). Two sizes of plastic containers with lids (250 and 1000 cm$^3$, respectively) were partially filled with either 240 or 1200 g of soil. The amount of water needed to bring each soil to 50% of field capacity was determined and solutions were prepared to deliver this volume of water and the appropriate concentration of NH$_4$NO$_3$, H$_3$PO$_4$, and FeSO$_4$$\cdot$7H$_2$O and ZnSO$_4$$\cdot$7H$_2$O for the six treatments used (Table 2). The nutrient solution/water was sprayed uniformly to individual soil treatment replications (three replications per treatment) and mixed thoroughly. A single resin capsule was then placed carefully in the center of the soil in each treatment container, capped with a lid and incubated at 25ºC in a laboratory for either 60 or 120 d, similar to a study by Skogely and Schaff (1985). After the prescribed incubation time, resin capsules were removed and individually rinsed with pressurized distilled water until all the visible soil was removed from inside and outside the resin capsule in preparation for extraction and nutrient analysis. Each treatment was replicated three times with two soils, two soil volumes and two incubation times for a total of 144 individual treatments. At the end of 60 or 120 d of incubation, soils were air dried and appropriate samples were subjected to conventional soil tests; KCl-extractable NH$_4$-N, water extractable NO$_3$-N, and sodium bicarbonate extractable P.

Soil Nutrient and Statistical Analyses
Nutrients were extracted from each resin capsule by placement into a 50 mL centrifuge tube, addition of 20 mL of 2M HCl, shaken for 20 min, and filtered using a medium fast filter. The process was repeated three times for a total of 60 mL of extract. The extract was then analyzed using an automated analyzer (Lachat Instruments, Loveland, CO, USA) for NO$_3$-N and NH$_4$-N, and inductively coupled plasma (ICP, Thermo Electron Corporation, Franklin, MD, USA) spectroscopy for Fe, P, S, and Zn.

For comparison, conventional soil test methods were also used. Nitrate was extracted with distilled water and the concentration determined by the chromotropic acid method (Sims and Jackson 1971). Ammonium was extracted with 2M KCl and the concentration determined using an automated analyzer by the sodium salicylate-sodium nitroprusside method (Rowland 1983). Phosphorus was extracted with 0.5M sodium bicarbonate and the concentration determined using the ammonium molybdate, ascorbic acid method (Olsen et al. 1954).

Statistics were accomplished using STATISTIX 8 (Analytical Software, Tallahassee, FL, USA). Appropriate analyses of variance models were used and basic mean separation was accomplished using Least Significant Difference. Means were also subjected to regression analysis and R$^2$ values are reported.

RESULTS AND DISCUSSION

Because of space limitations, consistent soil by treatment interactions, and unique soil differences observed for the parameters measured, all data presented compare soils (averaging values over the two sampling times and two soil volumes). Results related to sampling times and soil volumes will not be presented because differences were not significant. Also, values for N or P were averaged when treatments were repetitive for N and P (Treatments 2 and 3 = 11.2 kg N ha$^{-1}$, Treatments 4 and 5 = 22.4 kg N ha$^{-1}$, Treatments 2, 4 and 6 = 22.4 kg P ha$^{-1}$ and Treatments
3 and 5 = 44.8 kg P ha\(^{-1}\); Treatment 6 = 44.8 kg N ha\(^{-1}\); Table 2). Statistics were generated and those values are given in graphs and tables.

Resin capsule NH\(_4\)-N was significantly related to N rate in the loamy sand (Figure 1; Table 3; \(R^2 = 0.79\), significant) but was not related in the sandy clay loam (Figure 1; Table 3; \(R^2 = 0.12\), not significant). Potassium chloride-extractable NH\(_4\)-N was unrelated to the rate of N applied to either soil (Figure 1). For example, NH\(_4\)-N was either higher or the same at low N rates as for the high application rates (non significant \(R^2\) values of 0.10 and 0.0002 for loamy sand and sandy clay loam, respectively). This was to be expected as the resin capsule was in contact with the NH\(_4\)-N from the initial treatment application while KCl-extractable NH\(_4\)-N was measured on soil sampled after 60 and 120 d of incubation. Regardless, N treatment applications were reflected in NH\(_4\)-N in resin capsules for loamy sand but not for the sandy clay loam. Especially significant is the capability of resin capsule NH\(_4\)-N to distinguish between 0 and 11.2 kg N ha\(^{-1}\) application rate in the loamy sand (Figure 1).

All relationships between rate of N and NO\(_3\)-N measured in resin capsules and by water extraction were statistically significant in both soils (Figure 2). However, the predictability of the relationship was better for water extractable than for resin capsule NO\(_3\)-N (Table 3). The \(R^2\) values for resin capsule NO\(_3\)-N and N rate are 0.33 and 0.15 (both statistically significant) and for water extraction NO\(_3\)-N and N rate are 0.71 and 0.85 for loamy sand and sandy clay loam, respectively. Thus, conventional soil tests were more reflective of the NO\(_3\)-N than the resin capsule. This might be expected as resin capsules are passive sinks for nutrients and diffusion may limit the total amount of NO\(_3\)-N moving to and captured by the resin capsule. It might also be related to the fact that more NH\(_4\)-N was attracted to the capsule and consequently less NO\(_3\)-N was available near the capsule to be captured by the resin sink. This explanation is supported
when NO₃-N and NH₄-N [(NO₃+NH₄)-N] are summed and regressed against N rates as the R² values nearly double (Table 3, (NO₃+NH₄)-N). Yet there is still a relatively poor, albeit significant, relationship for the sandy clay loam soil (R² = 0.27). Summing NO₃-N and NH₄-N decreases the R² values from conventional soil tests, an observation not surprising considering the poor NH₄-N relationships observed. Although resin capsule NO₃-N was proportional to the fertilizer applied, it was unable to distinguish between 0 and 11.2 kg N ha⁻¹ in either soil, while water extractable NO₃-N distinguished among all levels of applied N in both soils (Figure 2).

The results for P were similar to those for NO₃-N, with resin capsule P clearly delineating among the three P rates with the loamy sand but with no ability to distinguish among treatments with the sandy clay loam soil (Figure 3). Sodium bicarbonate extractable P did distinguish among the three relatively low P application rates in both soils (Figure 3). The R² values support these observations (Table 4)--resin capsule P for the sandy clay loam soil was the only non-significant relationship. The highest predictability was associated with NaHCO₃-extractable P (R² of 0.46 and 0.74 for loamy sand and sandy clay loam, respectively). In all cases where close relationships between applied P and resin or bicarbonate P were found, the 0 and 22.4 kg ha⁻¹ treatments were clearly distinguishable.

The other nutrients applied were Fe and Zn (applied as part of treatment 2 as FeSO₄·7H₂O and ZnSO₄·7H₂O). There were no significant differences between treated and untreated soils for Fe or Zn measured in resin capsules (Table 3). But significant quantities in SO₄-S were measured in both soils for treatment 2, where SO₄-S forms of micronutrients were applied (Table 4). Considering that SO₄-S was applied at 9.2 kg ha⁻¹ and that conventional soil tests for SO₄-S are considered moderately effective at best (SO₄-S was not determined with a “conventional method” since none are considered adequate), this finding is impressive and points out a
potential strong benefit in resin capsule use.

CONCLUSION

Resin capsules reflected NH$_4$-N and P fertilizer applied at low rates in the loamy sand, but not in the sandy clay loam soil. Fertilizer N application was reflected in resin capsule NO$_3$-N in both loamy sand and sandy clay loam soils, but resin capsule NO$_3$-N could not distinguish between 0 and 11.2 kg ha$^{-1}$ fertilizer N application. Neither Fe nor Zn application was reflected in resin capsules, but the small amount of S applied, with these fertilizers, was clearly identified.

Comparing resin capsule to conventional soil test procedures, resin capsule NH$_4$-N was clearly a better indicator than KCl-extractable NH$_4$-N; resin capsule NO$_3$-N was effective, but not as clear an indicator as water extraction; resin capsule P was reflective of soil applied P in loamy sand but not in sandy clay loam soil, while sodium bicarbonate was effective in both soils. In each case where P application rate was reflected in resin or soil P, 0 and 22.4 kg P ha$^{-1}$ were distinguishable. Resin capsule SO$_4$-S was effective in indentifying the low level SO$_4$-S application.

Resin capsules show promise for use in low-input semi-arid soils, but more research beyond this study is needed to understand the soil by treatment interactions observed with NH$_4$-N and P in this research. Specifically, resin capsules need to be correlated to plant available nutrients, other research of greenhouse and field studies should be performed. The potential for using resins for delineating SO$_4$-S levels needs further investigation.
REFERENCES


Table 1. Soil characteristics of two native soils obtained from Moab and Rush Valley, UT used in the incubation study

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Soil Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Location</td>
<td>Moab, UT</td>
</tr>
<tr>
<td>Texture</td>
<td>Loamy sand</td>
</tr>
<tr>
<td>Sand, %</td>
<td>82</td>
</tr>
<tr>
<td>Silt, %</td>
<td>10</td>
</tr>
<tr>
<td>Clay, %</td>
<td>8</td>
</tr>
<tr>
<td>Cation exchange capacity, meq 100 g⁻¹</td>
<td>2.0</td>
</tr>
<tr>
<td>CaCO₃, %</td>
<td>5.7</td>
</tr>
<tr>
<td>Organic Matter, %</td>
<td>0.1</td>
</tr>
<tr>
<td>pH</td>
<td>8.2</td>
</tr>
<tr>
<td>Sodium bicarbonate phosphorus, mg kg⁻¹</td>
<td>5.6</td>
</tr>
<tr>
<td>Sodium bicarbonate potassium, mg kg⁻¹</td>
<td>35</td>
</tr>
<tr>
<td>NO₃-N, mg kg⁻¹</td>
<td>1.0</td>
</tr>
<tr>
<td>Electrical conductivity, dS m⁻¹</td>
<td>0.4</td>
</tr>
<tr>
<td>Sodium adsorption ratio</td>
<td>2.4</td>
</tr>
<tr>
<td>Rush Valley, UT</td>
<td>Sandy clay loam</td>
</tr>
<tr>
<td>Sand, %</td>
<td>51</td>
</tr>
<tr>
<td>Silt, %</td>
<td>24</td>
</tr>
<tr>
<td>Clay, %</td>
<td>25</td>
</tr>
<tr>
<td>Cation exchange capacity, meq 100 g⁻¹</td>
<td>11.0</td>
</tr>
<tr>
<td>CaCO₃, %</td>
<td>38</td>
</tr>
<tr>
<td>Organic Matter, %</td>
<td>1.0</td>
</tr>
<tr>
<td>pH</td>
<td>7.5</td>
</tr>
<tr>
<td>Sodium bicarbonate phosphorus, mg kg⁻¹</td>
<td>6.4</td>
</tr>
<tr>
<td>Sodium bicarbonate potassium, mg kg⁻¹</td>
<td>210</td>
</tr>
<tr>
<td>NO₃-N, mg kg⁻¹</td>
<td>1.4</td>
</tr>
<tr>
<td>Electrical conductivity, dS m⁻¹</td>
<td>0.4</td>
</tr>
<tr>
<td>Sodium adsorption ratio</td>
<td>0.4</td>
</tr>
</tbody>
</table>
Table 2. Nitrogen, P, Fe and Zn treatments applied to two soils and incubated with resin capsules at 50% field capacity in two volumes of soil for 60 and 120 d.

<table>
<thead>
<tr>
<th>Treatment Number</th>
<th>Nitrogen (N) (^1)</th>
<th>Phosphorus (P) (^1)</th>
<th>Micronutrients (^1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>11.2</td>
<td>22.4</td>
<td>Zn=5.6, Fe=11.2, S=9.2</td>
</tr>
<tr>
<td>3</td>
<td>11.2</td>
<td>44.8</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>22.4</td>
<td>22.4</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>22.4</td>
<td>44.8</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>44.8</td>
<td>22.4</td>
<td>0</td>
</tr>
</tbody>
</table>

\(^1\)N was applied as NH\(_4\)NO\(_3\), P as H\(_3\)PO\(_4\), Fe as FeSO\(_4\)\(_7\)H\(_2\)O and Zn as ZnSO\(_4\)\(_7\)H\(_2\)O
Table 3. R² values for parameters measured by resin capsules and conventional soil tests as related to N or P fertilizer application

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Related to</th>
<th>Resin Capsule</th>
<th>Conventional Soil Tests</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Loamy sand</td>
<td>Sandy clay loam</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Loamy sand</td>
<td>Sandy clay loam</td>
</tr>
<tr>
<td>Ammonium N applied</td>
<td></td>
<td>0.79*</td>
<td>0.12&lt;sup&gt;NS&lt;/sup&gt;</td>
</tr>
<tr>
<td>Nitrate N applied</td>
<td></td>
<td>0.33*</td>
<td>0.15*</td>
</tr>
<tr>
<td>Nitrate + Ammonium</td>
<td>N applied</td>
<td>0.71*</td>
<td>0.27*</td>
</tr>
<tr>
<td>Phosphorus P applied</td>
<td></td>
<td>0.41*</td>
<td>0.02&lt;sup&gt;NS&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>1</sup>* is significant at the 0.05 level of probability and NS is not significant at the 0.05 level of probability.

<sup>2</sup>Conventional soil test extractants were 2M KCl, distilled water and sodium bicarbonate for NH₄-N, NO₃-N and P, respectively. Nitrate + Ammonium represent the sum of NO₃- and NH₄-N in resin capsule or conventional soil tests.
Table 4. Iron, S, and Zn measured in resin capsules relative to an untreated control soil.

<table>
<thead>
<tr>
<th>Fertilizer Treatment, kg ha$^{-1}$</th>
<th>Fe</th>
<th>S</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Fertilizer</td>
<td>26.5</td>
<td>395</td>
<td>1.8</td>
</tr>
<tr>
<td>Plus Fertilizer$^1$</td>
<td>26.3</td>
<td>1890</td>
<td>1.7</td>
</tr>
<tr>
<td>Statistical Significance, $p &lt; 0.05^2$</td>
<td>NS</td>
<td>**</td>
<td>NS</td>
</tr>
</tbody>
</table>

$^1$Nutrients were applied as follows (kg ha$^{-1}$): 11.2 Fe, 9.2 S, and 5.6 Zn.  
$^2$** is significant at the 0.01 level of probability and NS is not significant at the 0.05 level of probability.
Figure 1. Resin capsule and KCl-extractable NH₄-N measured in loamy sand and sandy clay loam soils treated with 0, 11.2, 22.4 and 44.8 kg N ha⁻¹. Within resin capsule or KCl extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, c or d used to compare loamy sand; y and z used to compare sandy clay loam).
Figure 2. Resin capsule and KCl-extractable NO$_3$-N measured in loamy sand and sandy clay loam soils treated with 0, 11.2, 22.4 and 44.8 kg N ha$^{-1}$. Within resin capsule or water extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, c or d used to compare loamy sand; w, x, y and z used to compare sandy clay loam).
Figure 3. Resin capsule and sodium bicarbonate extractable P measured in loamy sand and sandy clay loam soils treated with 0, 22.4 and 44.8 kg P$_2$O$_5$ ha$^{-1}$. Within resin capsule or sodium bicarbonate extraction for a given soil, columns with the same letter are not significantly different at the 0.05 level of probability (a, b, or c used to compare loamy sand; x, y and z used to compare sandy clay loam).
MANUSCRIPT #2- EVALUATING NUTRIENT AVAILABILITY IN SEMI-ARID SOILS WITH RESIN CAPSULES AND CONVENTIONAL SOIL TESTS. II. NATIVE PLANT BIOAVAILABILITY UNDER GLASSHOUSE CONDITIONS

(Prepared for submission to Communications in Soil Science and Plant Analysis)
EVALUATING NUTRIENT AVAILABILITY IN SEMI-ARID SOILS WITH RESIN CAPSULES AND CONVENTIONAL SOIL TESTS. I. NATIVE PLANT BIOAVAILABILITY UNDER GLASSHOUSE CONDITIONS

Mary P. Jones¹, Bruce L. Webb¹, Von D. Jolley¹, Bryan G. Hopkins¹, and Daniel A. Cook¹

¹Department of Plant and Wildlife Sciences, Brigham Young University, Provo, UT 84602

ABSTRACT

Resin capsule technology developed for nutrient analysis in agriculture soils recently expanded to semi-arid soils without knowledge of effectiveness. This study determined if resin nutrient adsorption is correlated to plant uptake and yield in semi-arid soils. Two semi-arid soils were treated with five rates of nitrogen (N) and four rates of phosphorous (P), placed in pots with resin capsules, seeded with squirreltail grass (Elymus elymoides) and grown in a glasshouse for 120 d followed by biomass determination and nutrient analysis of capsules, soils, and tissues. Yield and total nutrient removal were highly correlated to fertilizer application rates, resin NH₄-N, and NaHCO₃-extracted P, marginally correlated to resin or soil NO₃-N, and unrelated to resin P. Use of resin capsules to estimate N bioavailability is promising, but P bioavailability is not effectively estimated with resin capsules; instead NaHCO₃ extraction is recommended.

INTRODUCTION

Four decades ago ion-exchange resin capsules were introduced for estimating plant available nutrients in soils with some limited use in agricultural soils. Resin capsules provide a simple, convenient, repeatable methodology (Skogley et al. 1996) to assess soil nutrient supplying capacity. Resin capsules are small elliptical balls that are slightly larger than a marble and contain resin beads which equilibrate with ions in soil solution and in some ways mimic plant
nutrient absorption (Yang et al. 1991a). The capsules are buried in soils at a prescribed depth for a specified time and are subsequently removed, and adsorbed nutrients extracted and quantified. Theoretically, the quantity of nutrients adsorbed by a resin capsule is related to the quantity available to plant roots, and therefore, resins give an indication of diffusion controlled plant processes (Yang et al. 1991a, 1991b).

The majority of the research and commercial use of resin capsules has been carried out in relatively fertile agricultural soils. Recently, use of resin capsules has extended to semi-arid ecosystems where the soil attributes and climatic conditions do not match the situations for which they were most studied; thus creating a question regarding the validity of their use in these conditions. While general concerns have been raised about the difficulty of comparing resin-based techniques and traditional soil measurements (Johnson et al. 2005), there are even greater concerns regarding their use in low-nutrient semi-arid soils, such as those in Southern and Western Utah. Studies under these conditions have shown that measurements from resin capsules and traditional soil testing methods are not fully comparable and that they should not be considered interchangeable (Sherrod et al. 2003; Jones et al. 2011). Furthermore, interpretive differences between resin and traditional methods are poorly understood (Sherrod et al. 2002; Sherrod et al. 2003). Before expansion of resin capsule use with low-nutrient semi-arid soils, further research is needed to determine the comparative value of these two approaches to soil testing.

In a previous study, Jones et al. (2011) incubated sandy clay loam and loamy sand soils (Rush Valley and Moab, Utah, respectively) for 120 d at low moisture content (50% field capacity) after treatment with six levels of N and P. There was a better correlation between nutrient application rate and nutrient concentration in the resin capsule extraction for NH₄-N
compared to conventional soil test procedures (KCl-extraction). However, NO$_3$-N and P extracted from the resin capsule were not as well correlated to rates of N and P as water extractable NO$_3$-N and NaHCO$_3$-extractable P. Traditional NaHCO$_3$-P extraction was effective in both soil types but resin capsule P was poorly correlated to P rates in the loamy sand but not correlated at all in the sandy clay loam soil. The variable findings in this carefully controlled incubation study suggested that resin capsules may have a place for use in low input conditions, but additional understanding of the interactions among soils is needed. Plant growth, nutrient concentration and uptake need to be related to resin capsule nutrient adsorption and compared to traditional soil tests to assess the relative value of each methodology.

This study was conducted to determine if NO$_3$-N, NH$_4$-N and P adsorbed by resin capsules or measured by conventional soil tests are equally related to yield, nutrient concentration, and uptake of squirreltail grass (*Elymus elymoides*) at five rates of N and four rates of P in a relatively controlled glasshouse conditions. Soils were maintained at or below 50% field capacity during the growth period.

**MATERIALS AND METHODS**

Two semi-arid soils collected from Moab and Rush Valley, Utah [loamy sand (mesic typic haplocalcid) and sandy clay loam (mesic xeric haplocalcid), respectively; Table 1] were dried, sieved to pass a 2 mm screen and mixed thoroughly. Standard 20.3-cm diameter pots with four bottom holes (covered with water permeable paper) were filled with 2.5 and 2.0 kg of loamy sand and sandy clay loam soil, respectively. Treatments included four rates of P at each of five rates of N (kg ha$^{-1}$ on a weight basis) which included 0, 5.5, 11, 22 and 44 kg N ha$^{-1}$ applied as ammonium nitrate (NH$_4$NO$_3$; 34-0-0) and 0, 11, 22 and 44 kg P ha$^{-1}$ applied as phosphoric acid (H$_3$PO$_4$; 0-72-0). Treatments were replicated three times in a completely randomized block
design under glasshouse conditions without supplemental light and temperatures of 20-25ºC. A solution with the appropriate quantities of dissolved \( \text{NH}_4\text{NO}_3 \) and/or \( \text{H}_3\text{PO}_4 \) in water was sprayed on each soil and thoroughly hand mixed before placement into pots to deliver nutrients and to raise the soil water content to 50% field capacity. Resin capsules were placed approximately 7 cm below the soil surface. Four seeds of squirreltail grass were planted in each pot and consistently watered over the duration of the experiment to maintain approximately 50% of field capacity or less. At 120 d, biomass was harvested (entire plant above soil), yield gravimetrically determined and dried in a forced air oven at 65ºC for at least 48 hr. Each sample was ground (0.85-mm mesh screen) and analyzed for total N using the Dumas method with a LECO Truespec N analyzer (LECO, St Joseph, MI, USA; McGeehan and Naylor 1988) and for P by digesting with nitric and perchloric acid and analyzed (Johnson and Ulrich 1959) with inductively coupled plasma spectroscopy (ICP, Thermo Electron Corporation, Franklin, Maryland, USA). Total removal of N and P by squirreltail grass was calculated (yield*%N or yield*%P) to compare to rates of N and P applied and to resin and conventional soil extraction.

At the same time as plants were harvested, resin capsules were removed, individually washed with distilled water, air dried and stored in plastic bags at approximately 5ºC until extraction and analysis were completed. Phosphorus, \( \text{NO}_3\)-N and \( \text{NH}_4\)-N were extracted from resin capsules by placing each capsule into a 50 ml centrifuge tube containing 20 ml of 2M HCl, shaking for 20 min, and filtering with 12.5 cm medium-fast filter paper. The process was repeated three times for a total of 60 ml of extract. The extract was then analyzed for \( \text{NO}_3\)-N and \( \text{NH}_4\)-N colorimetrically using a rapid flow analyzer (LACHAT Instruments, Loveland, CO, USA) and for P using ICP. Approximately 8-cm depths of soil (measured from the surface) from each pot was collected, air dried, and ground through a 2-mm sieve. Soil from each pot was
extracted for NO$_3$-N and NH$_4$-N using 2M KCl (KCl-extracted NO$_3$-N, KCl-extracted NH$_4$-N), and the concentrations determined using the rapid flow analyzer by the sodium salicylate-sodium nitroprusside method (Rowland 1983). Soil P was extracted with 0.5M sodium bicarbonate (NaHCO$_3$-extracted P) and the concentration determined using the ammonium molybdate, ascorbic acid method with a spectrophotometer (Olsen et al. 1954).

Statistics were performed by STATISTIX®8 (Analytical Software, Tallahassee, FL, USA). Appropriate analysis of variance models were used and mean separation was accomplished using Tukey Multiple Comparisons test. Means were subjected to regression analysis.

RESULTS AND DISCUSSION

Squirreltail Grass N Relationships

Since responses to N fertilizer were similar in the two soils and there were no significant soil by N rate interactions, data presented herein are averages of two soils. Squirreltail grass yields were closely related to amounts of N fertilizer applied ($r^2=0.992$, $P=0.0003$; Figure 1). Such yield increases are expected with conventional crops but have not been reported for squirreltail grass (Havlin et al. 2005). In addition to yield relating to rate of N application, yield was highly correlated with resin capsule NH$_4$-N ($r^2=0.922$, $P=0.0095$; Figure 2). Whereas KCl-extracted NH$_4$-N measured in soils obtained at the end of the experimental period was not predictive of plant yield ($r^2=0.129$, $P=0.553$; Figure 2). This observation with KCl-extracted NH$_4$-N is likely due to the time KCl-extracted NH$_4$-N was sampled (after 120 d growth period). These results suggest that resin capsules quickly adsorbed NH$_4$-N and the equilibrium established when NH$_4$-N was high (when first applied) remained adsorbed in the resin even after soil NH$_4$-N levels declined due to plant uptake, nitrification, or volatilization (Havlin et al. 2005). When yield was
compared to resin or KCl-extracted NO$_3$-N, there was a positive, but not statistically significant relationship for both methods of measuring NO$_3$-N ($r^2=0.617$, $P=0.115$, and $r^2=0.545$, $P=0.154$ respectively; Figure 3). This is not surprising with KCl-extracted NO$_3$-N, as samples were obtained at the end of the growth period. However, the relationship with resin NO$_3$-N would be expected to be more highly correlated, similar to the NH$_4$-N, since resin capsules were in place to effect an equilibrium during the entire 120-d growth period. This casts doubt on the ability of resin capsules to effectively monitor soil NO$_3$-N (Austin et al. 1977).

Squirreltail grass N concentration was significantly correlated to rate of N application ($r^2=0.328$, $P=0.031$, Table 2) but was not significantly related to resin or KCl-extracted NH$_4$-N and NO$_3$-N ($P$ values of 0.323 or higher; Table 2). Binkley (1984) determined in a glasshouse study that resins were poor competitors of N when placed in pots growing ryegrass (*Lolium perenne* L.) compared to resins placed in pots without plants, and thus correlation between N concentration and resin N was much better in pots without ryegrass.

Total removal of plant N was highly correlated to application rates for N ($r^2=0.985$, $P=0.0008$; Figure 4). Resin NH$_4$-N was likewise strongly correlated with the total removal of N ($r^2=0.985$, $P=0.0008$; Figure 5), whereas KCl-extracted NH$_4$-N had a negative, nonsignificant relationship ($r^2=0.100$, $P=0.604$; Figure 5). This further confirms that NH$_4$-N was likely adsorbed to resin capsules early in the growth process before uptake, nitrification, or volatilization occurred, whereas KCl-extracted NH$_4$-N was measured at the end of the growing period when NH$_4$-N was already volatilized, converted to NO$_3$-N, or absorbed by plants. Resin NO$_3$-N and KCl-extracted NO$_3$-N produced positive, and in the case of resin NO$_3$-N, a nearly significant relationship to total plant removal of N ($r^2=0.7572$, $P=0.055$ and $r^2=0.6116$, $P=0.118$ respectively; Figure 6). This is the only hint of resin NO$_3$-N being effective in defining N
relationships in squirreltail plant growth in our study.

Squirreltail Grass P Relationships

Squirreltail grass responses to P fertilizer were similar in the two soils and there were no significant soil by P rate interactions; thus, data presented herein are averages of the two soils. Squirreltail grass yield related to amounts of P fertilizer applied ($r^2=0.967$, $P=0.016$, Figure 1) and also significantly correlated with NaHCO$_3$-extracted P ($r^2=0.918$, $P=0.042$; Figure 7), indicating that this test was effective at extracting bioavailable P from the soil. However, resin P and yield were not significantly related ($r^2=0.330$, $P=0.426$; Figure 7). It is probable that P mobility was very limited (likely less than a few mm) in these high pH calcareous soils with low organic matter. Although plant roots were able to grow to and scavenge the P from the fertilizer, it is likely that very few, if any, P fertilizer granules resided in close enough proximity to a resin capsule for any interaction to occur. The traditional NaHCO$_3$ method involves sampling from a larger volume of soil than the resin capsule and, therefore, was more likely to include microsites of soil impacted by P fertilizer granules.

Total P removal by squirreltail grass was highly correlated to P application rates ($r^2=0.955$, $P=0.023$; Figure 4), to NaHCO$_3$-extracted P ($r^2=0.994$, $P=0.003$; Figure 8), but not to resin P ($r^2=0.583$, $P=0.237$; Figure 8). Phosphorus concentration of squirreltail grass was not significantly related to P rate, resin capsule P or NaHCO$_3$-extracted P (Table 3). Sardi (1996) studied relationships between plant uptake, yield and resin P adsorption in acid soils of Georgia and found no significant relationships among yield or plant uptake and resin P in the four acid soils tested.

In summary, total removal of N was highly correlated with amounts of N applied and resin NH$_4$-N, but not to resin or KCl-extracted NO$_3$-N. On the other hand, total removal of P in
squirreltail grass was not related to resin P but was highly correlated to rate of P application and to NaHCO₃-extracted P.

Conventional Soil Test and Resin Capsule Relationships

A previous incubation study utilizing these two soils emphasized relationships among P and N application rates, resin NH₄-N, NO₃-N and P compared to KCl-extracted NH₄-N, NO₃-N and NaHCO₃-extracted P (Pletsch et al. 2011). In the glasshouse study reported herein, we were most interested in the plant-soil-resin interactions just discussed above. However, rates of N and P applied will be compared herein to resin NH₄-N, NO₃-N and P and to KCl-extracted NH₄-N, NO₃-N and NaHCO₃-extracted P. As with observations relating yield and total N uptake to N application rates, resin NH₄-N was highly correlated to N application rates ($r^2=0.955, P=0.004$; Figure 9), while KCl-extracted NH₄-N was not ($r^2=0.089, P=0.625$). Relationships between rates of N applied and resin or KCl-extracted NO₃-N were positive, nearly significant with resin NO₃-N but not significant with KCl-extracted NO₃-N ($r^2=0.694, P=0.080$ and $r^2=0.6209, P=0.113$ respectively; Figure 9). Since relationships with resin NH₄-N are so strong in this study, it suggests that the initial NH₄-N levels were quickly adsorbed by the resin in the soils. Resins act as a nutrient sink (Amer et al. 1955; Skogley et al. 1990) and nutrients adsorbed to the resin would be expected to remain for a long-time period after initial adsorption. The reason for weaker relationships with NO₃-N may be due to plant uptake or denitrification after nitrification converted NH₄-N to NO₃-N (Havlin et al. 2005). This finding is opposite that of Binkley (1984) who determined that NO₃-N was more mobile, and hence more likely to be adsorbed by the resin than NH₄-N. Binkley and Matson (1983) determined that under controlled conditions, ion exchange resin capsules were as successful as traditional soil testing procedures for predicting N availability (sum of NH₄-N and NO₃-N). Summing NH₄-N+NO₃-N did not improve
the relationships reported in our study (data not presented). Johnson et al. (2005) found that temperature and moisture had a significant effect on resin NO$_3$-N uptake with dry conditions not favoring resin adsorption of NO$_3$-N. This could help explain the poor relationships between N rate and NO$_3$-N since our soils were maintained at 50% field capacity or less and this may have affected movement to the resin capsule. Poorer relationships with KCl-extracted NH$_4$-N or NO$_3$-N soil analyses are likely related to time of sampling as soils were sampled at the end of the growth period after plant uptake, denitrification, immobilization or other soil processes had already impacted the fate of NO$_3$-N. Binkley (1984) observed in a glasshouse study that resin N adsorbed from soil in pots without ryegrass growing exhibited better correlations with N concentration than in pots in which ryegrass was grown. Thus, active plant growth in our study also could have reduced the effectiveness of resin capsule adsorption of NO$_3$-N.

Phosphorus relationships among resin and soil extracted P and application rates of P were nearly opposite that of N. Sodium bicarbonate-extracted P was highly correlated to rates of P application, while resin P was not ($r^2=0.980, P=0.010$ and $r^2=0.503, P=0.291$, respectively; Figure 10). Previous studies reported mixed results on the ability of the resin to adsorb P. In an incubation study, Pletsch et al. (2011) observed significant adsorption of P in a loamy sand but not in a sandy clay loam soil. Vaidyanathan and Nye (1970) reported an inability to calculate resin diffusion coefficients of phosphate in low moisture soils. Barrow and Shaw (1977) reported difficulty in resin adsorption of P and attributed it to the mesh barrier between the resin and soil. However, Bache and Rogers (1970) found that resin-extractable P was a superior method to predict P uptake and relative yield in acid soils. Lajatha (1988) determined that in desert soils, hydroxyl resins were inferior at adsorbing P to bicarbonate forms of resins. Our study used the hydroxyl resin as those are better for defining N relationships and we were interested in a
universal adsorption approach.

CONCLUSIONS

Resin capsule NH$_4$-N related closely to yield and total N removal of squirreletal grass and to rate of N applied, but KCl-extractable NH$_4$-N from soils sampled 120 d after N application did not. Resin capsule and KCl-extracted NO$_3$-N were not significantly correlated to yield, total N removal or to rate of N application. Resin capsule P was not related to yield, total P uptake by squirreletal grass or rate of P application, while NaHCO$_3$-extracted soil P was highly correlated to them. Concentrations of N and P in squirreletal grass could not be related to any resin or extractable N or P measurement—only N rate related significantly to whole plant N concentration. Our results with incubation and glasshouse studies suggest possible value in using resin capsules to assess NH$_4$-N but not to assess P status; resin NO$_3$-N was not better than soil tests in these two studies. Thus there is a need for further investigation in the field on semi-arid soils to further delineate the relationships.
REFERENCES


Table 1. Soil characteristics of loamy sand (Moab, Utah) and sandy clay loam (Rush Valley, Utah) soils used in glasshouse study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Moab, Utah</td>
</tr>
<tr>
<td></td>
<td>Rush Valley, Utah</td>
</tr>
<tr>
<td>Location</td>
<td>Loamy sand</td>
</tr>
<tr>
<td></td>
<td>Sandy clay loam</td>
</tr>
<tr>
<td>Texture</td>
<td>82</td>
</tr>
<tr>
<td></td>
<td>51</td>
</tr>
<tr>
<td>Sand, %</td>
<td>82</td>
</tr>
<tr>
<td>Silt, %</td>
<td>82</td>
</tr>
<tr>
<td>Clay, %</td>
<td>82</td>
</tr>
<tr>
<td>Cation exchange capacity, meq 100 g⁻¹</td>
<td>2.0</td>
</tr>
<tr>
<td></td>
<td>11.0</td>
</tr>
<tr>
<td>CaCO₃, %</td>
<td>5.7</td>
</tr>
<tr>
<td></td>
<td>38</td>
</tr>
<tr>
<td>Organic matter, %</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
</tr>
<tr>
<td>pH</td>
<td>8.2</td>
</tr>
<tr>
<td></td>
<td>7.5</td>
</tr>
<tr>
<td>Sodium bicarbonate phosphorus, mg kg⁻¹</td>
<td>5.6</td>
</tr>
<tr>
<td></td>
<td>6.4</td>
</tr>
<tr>
<td>Sodium bicarbonate potassium, mg kg⁻¹</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>210</td>
</tr>
<tr>
<td>NO₃⁻-N, mg kg⁻¹</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>1.4</td>
</tr>
<tr>
<td>Electrical conductivity, dS m⁻¹</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td>Sodium adsorption ratio</td>
<td>2.4</td>
</tr>
<tr>
<td></td>
<td>0.4</td>
</tr>
</tbody>
</table>
Table 2. Concentrations of N (%) of the above ground portion of squirreltail grass after growing for 120 d at 0, 5.5, 11, 22 and 44 kg ha\(^{-1}\) N and concentrations of resin NH\(_4\)-N and NO\(_3\)-N and KCl-extracted NH\(_4\)-N and NO\(_3\)-N used to correlate with plant N concentrations. Regression coefficients and \(P\) values are given; \(P\) values < 0.05 indicate significant relationships.

<table>
<thead>
<tr>
<th>N rate kg ha(^{-1}) N</th>
<th>Plant total N(^1)</th>
<th>Resin NH(_4)-N(^2)</th>
<th>KCl-extracted NH(_4)-N(^2)</th>
<th>Resin NO(_3)-N(^2)</th>
<th>KCl-extracted NO(_3)-N(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%</td>
<td>mg capsule(^{-1})</td>
<td>mg kg(^{-1})</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td>0.8434</td>
<td>111.6</td>
<td>13.9</td>
<td>256.3</td>
<td>7.1</td>
</tr>
<tr>
<td>5.5</td>
<td>1.0220</td>
<td>110.7</td>
<td>14.9</td>
<td>241.3</td>
<td>8.6</td>
</tr>
<tr>
<td>11</td>
<td>0.7527</td>
<td>121.4</td>
<td>14.3</td>
<td>192.7</td>
<td>8.2</td>
</tr>
<tr>
<td>22</td>
<td>1.0931</td>
<td>176.5</td>
<td>11.0</td>
<td>230.4</td>
<td>6.8</td>
</tr>
<tr>
<td>44</td>
<td>1.0623</td>
<td>305.8</td>
<td>13.8</td>
<td>525.9</td>
<td>12.5</td>
</tr>
<tr>
<td>(r^2)</td>
<td>0.328</td>
<td>0.316</td>
<td>0.224</td>
<td>0.217</td>
<td>0.102</td>
</tr>
<tr>
<td>(P)-value</td>
<td>0.031</td>
<td>0.323</td>
<td>0.421</td>
<td>0.429</td>
<td>0.601</td>
</tr>
</tbody>
</table>

\(^1\) \(r^2\) and \(P\) value define the relationship between N rate and plant total N and its significance. See figures 9 and 10 for relationship of resin NH\(_4\)-N and NO\(_3\)-N and KCl-extracted NH\(_4\)-N and NO\(_3\)-N to rate of N application.

\(^2\) \(r^2\) and \(P\) value define the relationship between plant total N and resin NH\(_4\)-N, NO\(_3\)-N and KCl-extracted NH\(_4\)-N and NO\(_3\)-N and its significance.
Table 3. Concentrations of P (%) of the above ground portion of squirreltail grass after growing for 120 d at 0, 11, 22 and 44 kg ha$^{-1}$ P and of resin P and NaHCO$_3$-extracted P used to correlate with plant total P. Regression coefficients and $P$ values are given; $P$ values < 0.05 indicate significant relationships.

<table>
<thead>
<tr>
<th>P rate kg ha$^{-1}$ P</th>
<th>Plant total P$^1$ %</th>
<th>Resin P$^2$ mg capsule$^{-1}$</th>
<th>NaHCO$_3$-extracted P$^2$ mg kg$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>1.019</td>
<td>25.14</td>
<td>4.48</td>
</tr>
<tr>
<td>11</td>
<td>1.195</td>
<td>21.23</td>
<td>4.81</td>
</tr>
<tr>
<td>22</td>
<td>1.499</td>
<td>33.51</td>
<td>5.68</td>
</tr>
<tr>
<td>44</td>
<td>1.187</td>
<td>32.40</td>
<td>7.30</td>
</tr>
<tr>
<td>$r^2$</td>
<td>0.127</td>
<td>0.367</td>
<td>0.077</td>
</tr>
<tr>
<td>$P$-value</td>
<td>0.644</td>
<td>0.395</td>
<td>0.723</td>
</tr>
</tbody>
</table>

$^1$r$^2$ and $P$-value define the relationship between P rate and plant total P and its significance. See Figure 11 for the relationship between resin P and NaHCO$_3$-extracted P to rate of P application. $^2$r$^2$ and $P$-value define the relationship between plant total P and resin P and NaHCO$_3$-extracted P and its significance.
Figure 1. Yield of squirreltail grass 120 d after the application of 0, 5.5, 11, 22, and 44 kg ha\(^{-1}\) N and of 0, 11, 22 and 44 kg ha\(^{-1}\) P. Values are the average of two soils.
Figure 2. Yield of squirreltail grass 120 d after application of 0, 5.5, 11, 22, and 44 kg ha$^{-1}$ N as related to resin and KCl-extracted NH$_4$-N. Values represent the average of two soils.
Figure 3. Yield of squirreltail grass 120 d after application of 0, 5.5, 11, 22, and 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NO\(_3\)-N. Values represent the average of two soils.
Figure 4. Total N and P removal by squirreltail grass 120 d after application of 0, 5.5, 11, 22, and 44 kg ha\(^{-1}\) N and 0, 11, 22, and 44 kg ha\(^{-1}\) P as related to N and P application. Values represent the average of two soils.
Figure 5. Total N removal by squirreltail grass 120 d after application of 0, 5.5, 11, 22, and 44 kg ha\(^{-1}\) N as related to resin and KCl-extracted NH\(_4\)-N. Values represent the average of two soils.
Figure 6. Total N removal by squirreltail grass 120 d after application of 0, 5.5, 11, 22, and 44 kg ha$^{-1}$ N as related to resin and KCl-extracted NO$_3$-N. Values represent the average of two soils.
Figure 7. Yield of squirreltail grass 120 d after application of 0, 11, 22, and 44 kg ha\(^{-1}\) P as related to resin P and NaHCO\(_3\)-P. Values represent the average of two soils.
Figure 8. Total P removal by squirreltail grass 120 d after application of 0, 11, 22, and 44 kg ha\(^{-1}\) P as related to resin and NaHCO\(_3\)-extracted P. Values represent the average of two soils.
Figure 9. Resin and KCl-extracted NH$_4$-N and NO$_3$-N after 120 d growing squirreltail grass as related to application of 0, 5.5, 11, 22, and 44 kg ha$^{-1}$ N and resin and NaHCO$_3$-extracted P 120 d growing squirreltail grass as related to application of 0, 11, 22, and 44 kg ha$^{-1}$ P. Values represent the average of two soils.
Figure 10. Resin and NaHCO$_3$-extracted P after 120 d growing squirrel tail grass as related to application of 0, 11, 22, and 44 kg ha$^{-1}$ P. Values represent the average of two soils.
MANUSCRIPT #3- EVALUATING NUTRIENT AVAILABILITY IN
SEMI-ARID SOILS WITH RESIN CAPSULES AND CONVENTIONAL
SOIL TESTS. III. FIELD STUDIES

(Prepared for submission to Communications in Soil Science and Plant Analysis)
ABSTRACT

Commonly used soil analysis and resin capsule procedures are employed to assess nutrient status in fertile agriculture soils, but their validity in semi-arid ecosystems is unknown. Previous studies suggest effectiveness of resin capsules and conventional soil tests varies depending on specific nutrient and soil type. Field studies with six rates of N and P application were established on crested wheat stands in Rush and Skull Valley, UT on clay loam and sandy loam soils, respectively. Resin capsule and conventional soil tests for NO$_3$-N, NH$_4$-N and P were implemented and crested wheatgrass plant nutrient status examined. Resin capsules were removed and replaced and soil samples taken every 90 d for one year. Resin P was not related to P application, but NaHCO$_3$-extracted P was, while resin NH$_4$-N, resin NO$_3$-N, KCl-extracted NO$_3$-N and KCl-extracted NH$_4$-N were related N application. Only KCl-extracted NO$_3$-N and NH$_4$-N levels were related to plant tissue N. Overall traditional soil tests are more effective than resin capsules in semi-arid field conditions, but resin capsules have potential for use in N assessment.
INTRODUCTION

Resin capsules are approximately two-cm diameter elliptical mesh balls containing resin beads that, when placed in the soil, allow equilibrium exchange of anions and cations in soil solution and, consequently, estimation of plant available nutrients (Yang et al. 1991). Thus, a resin acts as a nutrient sink. After the resin capsule reaches equilibrium with soil solution, nutrients in capsules can be extracted chemically and quantified. Resin capsules are typically buried in the soil under actual field conditions for specified periods of time.

Four decades ago ‘ion exchange resin capsules’ were developed to provide a simple, convenient, repeatable methodology to measure soil nutrient supplying capacity and were promoted to replace conventional soil test procedures (Skogley et al. 1996). Resin capsule technology has focused on measuring nitrogen (N), phosphorus (P), potassium (K), copper (Cu), iron (Fe), manganese (Mn), zinc (Zn) and sulfur (S) on fertile soils (Yadvinder-Singh et al. 2000; Schoenau et al. 1993). Nevertheless, conventional soil tests continue to be the dominate assessment tool. The desire for streamlined laboratory analyses and for ‘universal’ extractants (Skogley 1992) has rekindled interest in resin procedures as an alternative to traditional soil tests, including testing in semi-arid zone ecosystems.

There are several potential advantages of resin capsules over traditional soil test procedures. In contrast to a sampling at a single point in time, its high capacity as a nutrient sink allows estimation of nutrient supplying capacity of the soil over both somewhat short to very long periods of time. Plant roots take up nutrients via three mechanisms, namely: diffusion, root interception, and mass flow with water uptake. The resin capsule soil test method somewhat mimics soil-plant interactions as nutrients diffuse to and into the capsule, depleting nutrients in the surrounding soil which then stimulates equilibrium driven solubilization of nutrients from
soil minerals and organic matter (Skogley et al. 1990). Another potential advantage is the ability to simultaneously extract multiple nutrients from resin capsules, which is an attractive alternative to most traditional soil tests (Skogley et al. 1990). Abrams and Jarrell (1992) observed the adsorption of P and Fe in various types of saturated soils by resin capsules and results appeared promising. However, their findings suggested that tests need to be done in saturated and unsaturated conditions with multiple nutrients before a standardization of the procedure is implemented.

Despite promotion and many factors favoring acceptance, resin capsules have not replaced conventional soil test methods. There have been general concerns about resin-based techniques replacing traditional soil tests even on highly fertile soils (Johnson et al. 2005, Sherrod et al. 2002). The primary disadvantage of resin technology is a lack of calibration work that arguably needs to be performed for each unique environment and species. Correlation and critical soil test levels are variable by species (and often by hybrid/cultivar) with traditional soil testing and it is likely that this is the case for resin technology as well. And, because nutrient adsorption by the capsule is dependent on solute delivery, changing environmental conditions may produce variable results. For example, dry or cold environmental conditions may slow or prevent diffusion of nutrients to the resin, as well as reduce their effectiveness and predictability (Skogley 1992; Sherrod et al. 2003). This weakness may be particularly challenging as resin technology is expanded into low-nutrient, semi-arid zone soils.

Extension of resin capsule use beyond its agricultural soil niche is creating additional questions concerning the validity of its use in poorly fertile, semi-arid soils, such as those in much of the Western U.S., including much of Utah. Studies in these conditions have shown that measurements from resin capsules and traditional soil testing methods are seldom analogous and
they should not be considered interchangeable (Sherrod et al. 2002, 2003). Sherrod et al. (2002) ranked resin capsules, bags, and membranes on three arid, calcareous soils based on both net nutrient and proportional extraction and concluded that none produced comparable results. They also unsuccessfully attempted to compare the three resin procedures to a conventional soil test. They could not recommend any of the three resin methodologies as “most effective.” Sherrod et al. (2003) later attempted to establish a standard method to characterize nutrient bioavailability in terrestrial ecosystems by charging resin capsules with H⁺ and OH⁻, HCl, or NaHCO₃ and measured the proportion of attraction of 12 nutrients in arid calcareous soils. They were unable to make recommendations regarding a standard resin method because the chemistry involved in the adsorption reactions between soil and resin in these sandy, calcareous soils were too variable and poorly understood. Sardi et al. (1996) found that the longer resin capsules were in contact with the soil, the greater was nutrient adsorption. However, highly variable results of resin adsorption from soil equilibrium suggested a need for further study to understand how to have more precision and accuracy with resin capsule-based soil testing in poorly fertile soils. Only limited research establishing relationships between resin nutrient adsorption at low level nutrient concentrations or application rates exists (Sardi et al. 1996; Pletsch et al. 2009; Jones et al. 2011). Jones et al. (2011) treated clay loam and silt loam soils with six levels of N and P fertilizer mixed with the soils and incubated them for 120 d under low soil moisture. Resin capsules reflected NH₄-N and P applications at low rates in loamy sand, but not in sandy clay loam soil, while NO₃-N was reflected in resin capsules incubated in both soils. Under these controlled conditions, traditional soil tests were more effective in reflecting application rates of both P and NO₃-N, but resin capsules were a better reflection of NH₄-N than a soil test (Jones et al. 2011). Thus even in closely controlled incubation experiments resin capsules were less
effective than soil tests for reflecting low levels of P and NO₃-N and results varied between the two soil types. In a glasshouse study, Pletsch et al. (2009) mixed five rates of N and four rates of P fertilizer into these same soils. Squirreltail grass (*Elymus elymoides*) was planted in these soils and grown with soil moisture at or near 50% field capacity for 120 d. Yield and total removal of N were closely related to resin NH₄-N and to application rates of N fertilizer. However, resin P adsorption was not correlated to squirreltail grass yield or to total P removal, while NaHCO₃-extracted P was highly correlated. Resin and KCl-extracted NO₃-N were not significantly correlated to yield and total N removal. These results under the controlled environments of incubation and glasshouse are promising and suggest a need for further investigation of the relationships among conventional and resin capsule tests and applied nutrients under low fertility and less controlled conditions of the field. The research reported herein focuses on surface application of five rates of N and P fertilizer to two semi-arid soils with the objective of relating resin capsule, soil test and nutrient application rates at four sampling times over a 12-month period.

**MATERIALS AND METHODS**

Field studies were conducted in Rush Valley and Skull Valley, Utah, USA on low fertility clay loam and sandy loam soils, respectively (Table 1). Annual precipitation would classify these areas as semi-arid (Table 2). Previously fenced field sites prevented free range livestock from disturbing the study area. At each site, 1.0 m² plots were established. A resin capsule with an attached labeled plastic string (to aid in locating the capsule) was placed in the soil to a depth of 5 cm in each plot on 13 May 2009 prior to fertilizer application. Immediately after resin capsule placement, rates of 0, 5.5, 11, 22, 44, and 88 kg ha⁻¹ of N and P were surface applied and lightly raked into soils to as deep as two cm to incorporate the fertilizer, with five replications placed in a completely randomized block design. Granular triple superphosphate (0-45-0) and ammonium
sulfate (20-0-0) were fertilizer sources. After resin placement and fertilization, resins were removed and replaced approximately every 90 d (18 August and 17 November 2009, 23 March and 18 May, 2010-- frozen soil in February 2010 prevented removal and replacement until 23 March 2010).

After resin capsules were removed from the soil, excess soil was gently removed and the capsules were placed in labeled, sealed plastic bags and stored at 5°C until analysis was performed. Resins were rinsed with distilled water to wash fine soil particles from the surface before nutrients were extracted by washing each capsule three times using 20 ml of 2M HCl. The extract (total of 60 ml of 2M HCl) was then analyzed for NO$_3$-N and NH$_4$-N using a rapid flow analyzer (Lachat Instruments, Loveland, CO, USA) and for P using inductively coupled plasma spectroscopy (ICP, Thermo Electron Corporation, Franklin, MD, USA).

Conventional soil samples were collected to a depth of 5 cm each time resin capsules were removed. After transport to the laboratory, soils were immediately air dried and ground through a 2 mm sieve. The soils were extracted for NO$_3$-N and NH$_4$-N using 2M KCl (referred to as KCl-extracted NO$_3$-N and KCl-extracted NH$_4$-N) and the concentration determined using a rapid flow analyzer by the sodium salicylate-sodium nitroprusside method (Rowland 1983). Phosphorus was extracted with 0.5M sodium bicarbonate and the concentration determined using the ammonium molybdate, ascorbic acid method (Olsen et al. 1954).

Whole plant samples of the dominant grass species, crested wheatgrass (*Agropyron desertorum*) were taken randomly from each treatment 15 June 2009 and 2010 by cutting three bunches of crested wheatgrass approximately two inches above the soil surface. Samples were oven-dried, ground (0.85 mm sieve) and analyzed for total N using a LECO Truspec N analyzer (LECO Instruments, St. Joseph, MI, USA; McGeehan and Naylor 1988) and P using ICP
Results of resin, soil and plant analyses were statistically analyzed using SAS (Version 9.1, SAS Institute, 2003, Cary, NC, USA). Analysis of variance (ANOVA) using Tukey's Studentized Range test for mean separation, correlation and regression were used to analyze data. Levels of significance are either specifically reported or are at $P = 0.05$.

RESULTS AND DISCUSSION

Soil by treatment and time by treatment interactions were not significant which allows data to be presented as the average of two soils and four sampling times. There was a predictable relationship between rate of N applied and resin NH$_4$-N ($r^2 = 0.806$, $P = 0.015$; Figure 1), and an even stronger relationship between N rate and KCl-extracted NH$_4$-N ($r^2 = 0.987$, $P \leq 0.0001$; Figure 1). Analysis of variance interpretation supports that differences in resin NH$_4$-N among N rates are weaker ($P = 0.075$) than differences with KCl-extracted NH$_4$-N ($P < 0.0001$). In this study, N was applied as ammonium sulfate and raked into a depth of two cm so a less robust relationship with resin NH$_4$-N and N rate than KCl-extracted NH$_4$-N could be due to nitrification converting NH$_4$-N to NO$_3$-N in surface layers prior to coming in contact with resin capsules. However, any predictable relationship in the field coupled with positive observations in incubation and glasshouse studies support resin capsule use in semi-arid soils as a promising technology for assessing N (Pletsch et al. 2009, Jones et al. 2012). In previous studies, N was evenly distributed throughout the root zone, thus, guaranteeing contact with the resin capsule while in the NH$_4$-N form (Jones et al. 2012).

Resin NH$_4$-N was at a maximum in November but lower and at similar levels on other dates (Table 3). Potassium chloride-extracted NH$_4$-N declined from August to November, was similar from November to March and declined again from March to May. We have no
explanation for these patterns. Likewise we have no explanation for why resin NH₄-N was higher in sandy loam (Skull Valley) than the clay loam soil (Rush Valley, Table 4).

Both resin and KCl-extracted NO₃-N correlated to application rates of N, although resin NO₃-N was slightly less predictable ($r^2=0.895$, $P=0.0043$; Figure 2) than KCl-extracted NO₃-N ($r^2=0.996$, $P<0.0001$; Figure 2). Interpretation of analysis of variance supported highly significant observed differences among N rates with both resin and KCl-extracted NO₃-N ($P<0.0001$) Thus, KCl-extracted NO₃-N established a finer fitting relationship among rates of N than resin NO₃-N. That resin NO₃-N was strongly correlated to N application rates suggests that resin capsules may be effective in assessing mobile nutrient status in these semi-arid land soils even if placement of that nutrient is not mixed directly into the zone of capsule placement.

The patterns for NO₃-N measured over time with resin capsules or KCl-extraction differed dramatically (Table 3). The pattern for KCl-extracted NO₃-N changes could be associated with plant uptake through August followed by mineralization between the August and November samplings. The sharp decreases of NO₃-N between November and May could result from leaching and/or denitrification (Havlin et al. 2005). The pattern observed for resin NO₃-N could be explained by plant uptake maintaining low NO₃-N in August, little or no NO₃-N reaching resin capsules from August to November (resin capsules were placed at 5 cm). The peak in resin NO₃-N in March could be related to NO₃-N made available in the surface soil by mineralization/nitrification from the August to November period being leached into the zone of resin capsule placement during winter precipitation (Table 2) and the sharp decline by May could be due to further leaching, denitrification or plant uptake by native grasses (Table 2 and 3; Havlin et al. 2005). Average resin NO₃-N and KCl-extracted NO₃-N was higher in the clay loam (Rush Valley) than the sandy loam soil (Skull Valley, Table 4). This may be related to greater
leaching from the surface soils in the sandy loam soil (20.3 cm and 15.5 cm, precipitation from June 2009 to June 2010 for Skull Valley and Rush Valley, respectively; Table 2).

Rates of N were reflected in plant N concentrations ($R^2=0.947$, $P=0.012$; Figure 3). There was nearly significant difference in plant N concentration between plants grown in Skull Valley and Rush Valley ($P=0.0615$; Table 4). There was significantly higher N concentration measured in plants in June 2009 (1.17%) than June 2010 (0.67% N; $P<0.0001$), which suggests that the N application in March 2009 was effectively absorbed by plants and that the effects were greatly reduced by the second season (June 2010). Crested wheat total N concentrations were correlated to KCl-extracted NO$_3$-N and NH$_4$-N, but not to resin NH$_4$-N or NO$_3$-N (Table 5).

The P extracted from resin capsules did not significantly reflect P application levels ($r^2=0.060$, $P=0.64$; Figure 4), but NaHCO$_3$-extracted P did ($r^2=0.992$, $P<0.0001$; Figure 4). Interpretation of analysis of variance values directly supported these findings ($P=0.933$ and $P<0.0001$ for resin and NaHCO$_3$-extracted P, respectively). Previous studies reported mixed results on the ability of resins to adsorb P (Vaidyanathan and Nye 1970, Bache and Rogers 1970). Phosphorus fixation and resulting lack of mobility may have been a contributing factor in preventing adsorption by resin capsules as the calcareous soils may have fixed fertilizer P immediately upon dissolution (Vaidyanathan and Talibudeen, 1970). However, if fixation were the main factor, NaHCO$_3$-extractable P should have been equally ineffective in measuring added P. Barrow and Shaw (1977) reported difficulty in resin adsorption of P and attributed it to the mesh barrier between the resin and soil. Low P adsorption may also be attributed to low soil moisture in semi-arid soils, i.e. the lower levels of water present in a soil the less P diffusion to deliver P to the resin for adsorption (Amer et al. 1955). In this case it also might be related to the lack of movement of P into the soil layer containing the resin capsule (5 cm) due to low moisture
levels. Most likely, the explanation is related to the fact that P is not very mobile in soil, especially in calcareous, arid soil, and the resin capsules were likely not in close enough proximity with the applied fertilizer.

More resin P was found in November than in the other three sampling periods in which resin P values were similar (Table 3). This may have been the result of increased dissolution of the granular P into the soil during fall precipitation. Bicarbonate-extractable P did not change significantly over time despite an apparent drop from August to November (Table 3). Original soil test P levels were higher in Rush Valley soil (clay loam) than Skull Valley soils (sandy loam, Table 1). This was also reflected in the NaHCO₃-extracted P values averaged across six treatment levels (Table 4). However, resin P measured higher in Skull Valley than Rush Valley and that is opposite to native P levels reported (Table 4). We have no firm explanation for this observation other than the rate of P solubilization may have been relatively higher for Skull Valley because of higher precipitation (Table 2). Schaff and Skogley (1982) reported that P diffusion increased with an increase in water, especially in sandy soils.

Plant P tissue concentrations were not affected by P treatments ($r^2=0.034$, $P=0.835$; Figure 3) nor were there significant differences between 2009 and 2010 plant samples (0.14 and 0.13% P, respectively, $P=0.167$). However, average crested wheatgrass P concentration was significantly lower grown on the clay loam soil of Rush Valley than in the sandy loam soil of Skull Valley (Table 4). Neither resin P nor NaHCO₃-extracted P related to total plant P concentrations ($r^2=0.019$ and 0.034, respectively; Table 5) due primarily to no change in P concentration of crested wheatgrass with increasing P fertilizer application (Figure 3).

CONCLUSION

Conventional soil tests are confirmed as very effective and recommended for use in predicting
nutrient status in these semi-arid zone soils. In these two field studies, KCl-extracted NO$_3$-N and NH$_4$-N produced predictable and significant relationships with rates of applied N and N concentrations of crested wheatgrass. Sodium bicarbonate-extracted P also reflected P application rates, but did not relate to P content of crested wheatgrass, primarily because P content of wheatgrass did not change with P application. Resin NO$_3$-N and NH$_4$-N both gave predictable relationships with N rates, but were not as good of predictors as KCl-extracted NO$_3$-N or NH$_4$-N. Because resin capsule NH$_4$-N did strongly reflect N application rates in previous incubation and glasshouse studies where fertilizer was thoroughly mixed with soils, this positive result with resin NO$_3$-N and NH$_4$-N in the field may justify resin use for N assessment. However, it is clear from incubation, glasshouse and field studies that resin capsule P cannot be recommended to assess P fertility levels, even in situations where P fertilizer is thoroughly mixed with soils. Overall, conventional soil tests were superior to resin capsules in these two low nutrient, semi-arid ecosystems reflecting low application rates of N and P, but use of resins for N assessment could be an acceptable alternative.
REFERENCES


Rowland, A. P. 1983. An automated method for the determination of ammonium-N in ecological


Table 1. Selected characteristics of clay loam (Rush Valley) and sandy loam (Skull Valley) soils at the two field sites (Utah, USA).

<table>
<thead>
<tr>
<th>Soil characteristic</th>
<th>Rush Valley, UT</th>
<th>Skull Valley, UT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Textural classification</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sand, %</td>
<td>35</td>
<td>63</td>
</tr>
<tr>
<td>Silt, %</td>
<td>35</td>
<td>21</td>
</tr>
<tr>
<td>Clay, %</td>
<td>30</td>
<td>16</td>
</tr>
<tr>
<td>Organic matter, %</td>
<td>1.7</td>
<td>0.8</td>
</tr>
<tr>
<td>pH</td>
<td>7.2</td>
<td>7.3</td>
</tr>
<tr>
<td>Sodium bicarbonate P, mg kg⁻¹</td>
<td>8.2</td>
<td>5.8</td>
</tr>
<tr>
<td>Sodium bicarbonate K, mg kg⁻¹</td>
<td>336</td>
<td>285</td>
</tr>
<tr>
<td>NO₃-N, mg kg⁻¹</td>
<td>1.7</td>
<td>0.8</td>
</tr>
<tr>
<td>Electrical conductivity, dS m</td>
<td>0.49</td>
<td>0.43</td>
</tr>
</tbody>
</table>
Table 2. Quarterly precipitation distribution during the period of experiments at Rush Valley and Skull Valley (Utah, USA).

<table>
<thead>
<tr>
<th>Period of precipitation</th>
<th>Rush Valley(^1)</th>
<th>Skull Valley(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>cm</td>
<td>cm</td>
</tr>
<tr>
<td>June to August 2009</td>
<td>5.0</td>
<td>4.3</td>
</tr>
<tr>
<td>August to November 2009</td>
<td>3.2</td>
<td>2.0</td>
</tr>
<tr>
<td>November 2009 to March 2010</td>
<td>3.6</td>
<td>4.7</td>
</tr>
<tr>
<td>March to June 2010</td>
<td>3.7</td>
<td>9.1</td>
</tr>
<tr>
<td>Total</td>
<td>15.5</td>
<td>20.1</td>
</tr>
</tbody>
</table>

\(^1\) Utah Climate Center, 2011, Vernon, UT
\(^2\) Utah Climate Center, 2011, Dugway, UT
Table 3. Average values of resin and conventional soil test measurements for four times (August, November 2009 and March, May 2010). Values represent the average of six treatments (0, 5.5, 11, 22, 44 and 88 kg ha\(^{-1}\) N or P applied 13 May 2009) and two locations (Rush Valley and Skull Valley, UT). For comparing across times, values with the same letter are not significantly different at \(P<0.05\), Tukey Studentized Range Test.

<table>
<thead>
<tr>
<th></th>
<th>Month sampled</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2009</td>
<td>2010</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>August</td>
<td>November</td>
<td>March</td>
<td>May</td>
<td></td>
</tr>
<tr>
<td>Resin NH(_4)-N, µg resin capsule(^{-1})</td>
<td>87 b</td>
<td>196 a</td>
<td>63 bc</td>
<td>128 b</td>
<td></td>
</tr>
<tr>
<td>KCl-extracted NH(_4)-N, µg g(^{-1})</td>
<td>20 a</td>
<td>12 bc</td>
<td>15 b</td>
<td>7 c</td>
<td></td>
</tr>
<tr>
<td>Resin NO(_3)-N, µg resin capsule(^{-1})</td>
<td>69 b</td>
<td>78 b</td>
<td>153 ab</td>
<td>60 b</td>
<td></td>
</tr>
<tr>
<td>KCl-extracted NO(_3)-N, µg g(^{-1})</td>
<td>12 a</td>
<td>15 b</td>
<td>6 c</td>
<td>5 c</td>
<td></td>
</tr>
<tr>
<td>Resin P, µg resin capsule(^{-1})</td>
<td>21 b</td>
<td>28 a</td>
<td>20 b</td>
<td>18 b</td>
<td></td>
</tr>
<tr>
<td>NaHCO(_3)-extracted P, µg g(^{-1})</td>
<td>41 a</td>
<td>35 a</td>
<td>36 a</td>
<td>34 a</td>
<td></td>
</tr>
</tbody>
</table>
Table 4. Soil and crested wheatgrass tissue nutrient concentrations averaged across six N and six P fertilizer treatments (0, 5.5, 11, 22, 44 and 88 kg ha\(^{-1}\) N or P applied 13 May 2009) and four sampling dates (August, November 2009 and March, May 2010) for field studies at Skull Valley and Rush Valley, UT, USA. Soil nutrients extracted from resin capsules and using traditional soil test methods (KCl and NaHCO\(_3\) extractions for NO\(_3\)-N, NH\(_4\)-N and P, respectively).

<table>
<thead>
<tr>
<th>Nutrient</th>
<th>Rush Valley</th>
<th>Skull Valley</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Clay loam</td>
<td>Sandy loam</td>
<td></td>
</tr>
<tr>
<td>Ammonium-N</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin(^1)</td>
<td>101</td>
<td>136</td>
<td>0.0202</td>
</tr>
<tr>
<td>Soil(^1)</td>
<td>13</td>
<td>14</td>
<td>0.4904</td>
</tr>
<tr>
<td>Nitrate-N</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin</td>
<td>121</td>
<td>59</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Soil</td>
<td>11.6</td>
<td>7.4</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Phosphorus</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin</td>
<td>18</td>
<td>25</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Soil</td>
<td>42</td>
<td>32</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Whole plant (crested wheatgrass)</td>
<td>Total N(^2)</td>
<td>0.96</td>
<td>0.88</td>
</tr>
<tr>
<td></td>
<td>Total P(^2)</td>
<td>0.09</td>
<td>0.19</td>
</tr>
</tbody>
</table>

\(^1\) Resin value is µg resin capsule\(^{-1}\) and soil value is µg g\(^{-1}\).
\(^2\) Whole crested wheatgrass plant values for N and P is percent (%).
Table 5. $r^2$ values for correlations among soil or resin NH$_4$-N, NO$_3$-N and P and crested wheat N and P concentrations.

<table>
<thead>
<tr>
<th>Resin or Soil Parameter</th>
<th>Related to:</th>
<th>Resin $r^2$</th>
<th>Soil $r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH$_4$-N</td>
<td>Plant N</td>
<td>0.480</td>
<td>0.631*</td>
</tr>
<tr>
<td>NO$_3$-N</td>
<td>Plant N</td>
<td>0.556</td>
<td>0.726*</td>
</tr>
<tr>
<td>P</td>
<td>Plant P</td>
<td>0.019</td>
<td>0.034</td>
</tr>
</tbody>
</table>

* is significant at $P<0.05$ and NS is not significant at $P<0.05$. 

\(^{1}\)
Figure 1. Resin and KCl-extracted soil NH$_4$-N measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha$^{-1}$ N; fertilizer applied 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and four periods of time (August, November 2009 and March, May 2010).
Figure 2. Resin and KCl-extracted soil NO$_3$-N measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha$^{-1}$ N fertilizer was applied 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and four periods of time (August, November 2009 and March, May 2010).

\[ y_{\text{resin}} = 31.363 + 1.286x \quad r^2 = 0.895 \quad P = 0.0043 \]

\[ y_{\text{soil}} = 6.119 + 0.1176x \quad r^2 = 0.996 \quad P < 0.0001 \]
Figure 3. Concentration of N and P in crested wheatgrass grown after application of 0, 5.5, 11, 22, 44, 88 kg N and P ha\(^{-1}\) on 13 May 2009. Values represent the average of two locations (Rush Valley and Skull Valley) and two periods of time (June 2009 and June 2010). Linear regression was completed using SAS.
Figure 4. Resin and soil P measurements after application of 0, 5.5, 11, 22, 44, 88 kg ha\(^{-1}\) P; fertilizer was applied 13 May 2009. Values represent the average of two locations and four sampling periods (August, November 2009 and March, May 2010.)

Resin: 
\[ y = 22.03 - 0.00848x \]  
\[ r^2 = 0.060 \]  
\[ P = 0.64 \]

Soil: 
\[ y = 16.85 + 0.6846x \]  
\[ r^2 = 0.992 \]  
\[ P < 0.0001 \]
Rush Valley, Replication and Treatments

(Plot # - Treatment #)

<table>
<thead>
<tr>
<th>Rep 1</th>
<th>101-4</th>
<th>102-5</th>
<th>103-1</th>
<th>104-2</th>
<th>105-6</th>
<th>106-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rep 2</td>
<td>201-1</td>
<td>202-5</td>
<td>203-2</td>
<td>204-6</td>
<td>205-3</td>
<td>206-4</td>
</tr>
<tr>
<td>Rep 3</td>
<td>301-6</td>
<td>302-4</td>
<td>303-5</td>
<td>304-3</td>
<td>305-2</td>
<td>306-1</td>
</tr>
<tr>
<td>Rep 4</td>
<td>401-2</td>
<td>402-1</td>
<td>403-5</td>
<td>404-6</td>
<td>405-3</td>
<td>406-4</td>
</tr>
<tr>
<td>Rep 5</td>
<td>501-4</td>
<td>502-2</td>
<td>503-3</td>
<td>504-6</td>
<td>505-1</td>
<td>506-5</td>
</tr>
</tbody>
</table>

Skull Valley, Replication and Treatments

(Plot # - Treatment #)

<table>
<thead>
<tr>
<th>Rep 1</th>
<th>101-3</th>
<th>102-5</th>
<th>103-1</th>
<th>104-4</th>
<th>105-2</th>
<th>106-6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rep 2</td>
<td>201-6</td>
<td>202-1</td>
<td>203-3</td>
<td>204-2</td>
<td>205-4</td>
<td>206-5</td>
</tr>
<tr>
<td>Rep 3</td>
<td>301-2</td>
<td>302-1</td>
<td>303-6</td>
<td>304-4</td>
<td>305-3</td>
<td>306-5</td>
</tr>
<tr>
<td>Rep 4</td>
<td>401-1</td>
<td>402-5</td>
<td>403-6</td>
<td>404-2</td>
<td>405-4</td>
<td>406-3</td>
</tr>
<tr>
<td>Rep 5</td>
<td>501-3</td>
<td>502-6</td>
<td>503-4</td>
<td>504-5</td>
<td>505-2</td>
<td>506-1</td>
</tr>
</tbody>
</table>

Replication of Field Study Randomized Plot Design for Rush Valley, UT and Skull Valley UT. Treatments (N-kg ha⁻¹, P-kg ha⁻¹ P₂O₅): 1-(0, 0), 2-(5.6, 5.6), 3-(11.2, 11.2), 4-(22.4, 22.4), 5-(44.8, 44.8), 6-(89.6, 89.6).
RESIN CAPSULE EXTRACTION

Procedure:

1. Remove capsule from enclosed bag with tweezers and rinse thoroughly with distilled water using the pressurized nozzle from the tap removing all trapped soil particles.
2. Place capsule in 50 ml centrifuge tube.
3. Add 20 ml 2N HCL.
4. Shake for 20 minutes.
5. Filter through 15 cm filter paper into 125 ml plastic bottles.
6. Repeat steps 6-8 two (2) more times, for a total of 3 times ending with a total of 60 ml.
7. Run on the Inductively Coupled Plasma water as the blank (ICP, Thermo Electron Corporation, Franklin, Maryland).
8. Analyze for NH$_4^+$ and NO$_3^-$ on Lachat (LACHAT Instruments, Loveland, CO))

Reagents:

2 N HCL: 166.8 ml concentrated HCL and to a volume of 1 liter with distilled water.
AMMONIUM AND NITRATE EXTRACTION

Procedure:

1. Weigh 2.5 grams of soil into a centrifuge tube and add 25 ml of 2M KCl. Cap the tubes and shake for 1 hour.
2. Centrifuge the samples for approximately 5 minutes and filter into a 125 ml plastic bottle. This extract is used to determine both ammonium and nitrate.
3. Ammonium must be run the same day that it is extracted.

Reagent:

2M KCl: In 1 L volumetric, add 4.717 g of (NH₄)₂SO₄. Add 1 ml of concentrated HCl and fill to volume with distilled water.

References:


AMMONIUM BY LACHAT

1. Prepare reagents for NH₄⁻N.
2. Hypochlorite needs to be made new each day.
3. Prepare standards of 20, 8, 2, 0.5, 0.1, 0 ppm NH₄⁻N. Use 2M KCl for soil or 2 M HCl for resins to dilute the standards (2 M KCl in soil extraction of Ammonium and Nitrate Latchet).
4. Neutralize samples of 10 ml HCl with 1 ml 50% NaOH. Cover with parafilm, place stoppers on mixture and invert upside-down to mix.

Reagents:

2 M Potassium Chloride Carrier and Standards Diluent. Dissolve 150 g of KCl and bring to a volume of 1000 ml.

EDTA Solution (6%). In a 1 liter beaker, dissolve 66 g of EDTA disodium salt dehydrate in about 900 ml of distilled water with a magnetic stirrer. The pH at this point is approximately 5.2 and the solution is cloudy. Adjust the pH to 7.0 with sodium hydroxide pellets (approx. 25 pellets). Stir with a magnetic stirrer. Add sodium hydroxide pellets in five pellet increments until pH 7.0 is reached. The solution becomes clearer as the pH of the solution nears pH 7.0. Transfer the solution to a 1 liter volumetric flask and dilute to the mark with distilled water. Invert to mix.

Buffer. Dissolve 28 g of sodium hydroxide (NaOH) and 50 g sodium phosphate dibasic heptahydrate (Na₂HPO₄.7H₂O) in about 900 ml of distilled water. Dilute to the mark with water and mix.

Salicylate – Nitroprusside Color reagent. Dissolve 150 g sodium salicylate (C₆H₄(OH)(COO)Na), and 1.0 g sodium nitroprusside (Na₂Fe(CN)₅NO.2H₂O), in about 800 ml of distilled water. Dilute to the mark with water and invert to mix.

Hypochlorite reagent. In a 500 ml volumetric flask, dilute 250 ml 5.25 % sodium hypochlorite, (NaOCl) to the mark with distilled water. Invert to mix. Household bleach can be used.
NITRATE BY LATCHET

1. Reagents should be made to run NO$_3$-N.
2. Prepare standards of 40, 20, 12, 4, 0.4, 0 ppm NO$_3$-N. Use 2M KCl to dilute the standards for soil and 2M HCl for resin.
3. Neutralize resin HCl samples (10 ml HCl) with 1 ml 50% NaOH. Cover with parafilm, place stoppers on mixture and invert upside-down to mix.

Reagents:

2 M Potassium Chloride Carrier and Standards Diluent. Dissolve 150 g of KCl and bring to a volume of 1000 ml.

15 M Sodium hydroxide. In a 500 ml beaker, add 250 ml water. Slowly add 150 g sodium hydroxide. CAUTION: The solution will get very hot! Swirl until dissolved. Cool and store in a plastic bottle.

Ammonium chloride buffer, pH =8.5. In a 1 L volumetric flask, dissolve 85 g ammonium chloride and 1.00 g disodium ethylenediamine tetraacetic acid dihydrate in about 800 ml water. Adjust the pH up to 8.5 with 15 M sodium hydroxide. Dilute to the mark and invert to mix.

Sulfanilamide color reagent. To a 1 L volumetric flask add about 600 ml water. Then add 100.00 ml 85% phosphoric acid, 40.0 g sulfanilamide, and 1.0 g N-1-naphthylenediamine dihydrochloride (NED). Shake to wet and stir to dissolve 20 minutes. Dilute to the mark, and invert to mix. Store in a dark bottle and discard when pink.

References:

SODIUM BICARBONATE EXTRACTED PHOSPHORUS

Procedure:

1. Place 2.5 g of soil in a 250 ml Erlenmeyer flask. Adjust the pH of the NaHCO$_3$ in the 2 liter bottle using 6 N NaOH or 6 N HCl. to read 8.3 pH. Add 50 ml of 0.5 molar NaHCO$_3$ to the flasks. Place the rack on the reciprocating shaker and shake at low speed for 30 minutes.

2. After shaking, immediately filter the suspension through 15 cm medium-fast filter paper. To accomplish this, fold the filter paper so it will sit on top of the 100 ml plastic cups. Pour the suspension through the filter paper into the cups.

3. Take a 5 ml aliquot of the filtrate and add 10 ml of distilled water. Make this dilution in the large plastic tubes. Add 5 ml of reagent B with a pipette and then swirl vigorously.

4. Let the solution stand for 15 minutes and then read the color intensity on the Spectronic 7 Genesys Spectrophotometer at a wavelength of 880 nm. Use your blank to set the transmittance at 100 %, and then read the transmittance on the samples.

5. The ppm phosphorus is determined by comparing the samples with a standard curve. This curve is obtained by making working standards of 0.25, 0.5, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, and 7.0 ppm from the stock solution. Develop the color in these standards just as was done with the soil extract. The curve is made by plotting the transmittance of the standard against the concentration.

6. The curve follows Beer's law, so the log of transmittance falls in a straight line. This means a regression equation can be calculated and the results computed on a calculator.

Reagents:

Reagent A: Dissolve 12 g of ammonium molybdate in 250 ml of distilled water. Dissolve 0.2908 g of antimony potassium tartrate in 100 ml of distilled water. Make 100 ml of 5 N H$_2$SO$_4$ by adding 148 ml of concentrated H$_2$SO$_4$ to water and bringing to a volume of 1 liter. Add these three solutions together and bring to a volume of 2000 ml with distilled water. Store in a Pyrex bottle in a dark, cool place.

Reagent B: Dissolve 0.528g of ascorbic acid in 100 ml of reagent A (for 50ml use 0.264g of ascorbic acid and for 200 ml use 1.056 g ascorbic acid). This solution needs to be made every day.

0.5 Molar Sodium Bicarbonate: Add 1 liter of distilled water to 42 g of NaHCO$_3$. Let the solution equilibrate overnight. The pH needs to be adjusted with each use.
Reference:

TOTAL NITROGEN

Was analyzed for total N using the LECO Truespec nitrogen analyzer, Dumas method, (LECO, St Joseph, MI).

Reference:

PERCHLORIC ACID PLANT DIGESTION

ICP Element Determination

Procedure:

1. Weigh 0.5 g of plant material into 50 ml marked glass test tubes in racks of 40.

2. Add 5 ml of concentrated HNO₃ and let sit for at least 2 hours under the hood (preferably overnight).

3. On the small block, set the temperature at 220 degrees. The block should be on at least 30 minutes before running the samples.

4. Turn on the overhead hood. Individually place the test tubes on the block and leave on until approximately 50% of the nitric acid has evaporated. This is approximately 5-10 minutes. Do not leave the room. Check the samples periodically during this time so that they do not dry out. If dry, the samples need to be rerun.

5. After 10 minutes, remove the samples and add 1 ml of perchloric acid to each sample.

6. Place the samples back on the block and leave for 45 minutes. If after 45 minutes the samples are clear, then remove them. If they still have a yellow tint, leave them on for a few more minutes. Keep checking them to make sure they don’t burn dry.

7. After the samples are removed, put approximately 25 ml of distilled water into each sample.

8. Wash down the hood for 10 minutes following digestion.

9. Fill the test tubes to volume (50 ml). Cap them using the yellow, silicon stoppers or the clear plastic stopper cups and shake.

10. Determine P by using the ICP, inductively coupled plasma spectroscopy (ICP, Thermo Electron Corporation, Franklin, Maryland (no dilutions are necessary).

Reference: