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Methods for Measuring Nitrogen Release from Controlled-Release Fertilizer Used for Vegetable Production[1]

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Introduction

Enhanced-efficiency fertilizers (EEFs) are a group of fertilizers that reduce the risk of nutrient loss to the environment and subsequently increase fertilizer use efficiency (Slater, 2010). This increase may be accomplished through maintaining nutrients in the root zone by physical barriers (coating), reduced solubility, or retaining nutrients in a less leachable form (Trenkel, 2010). There are three subgroups of EEFs with different characteristics for horticultural production systems:

1. Slow-release fertilizers (SRFs): contain nitrogen (N) in a less-soluble, plant-unavailable form that usually needs microbial degradation to provide plant-available N.
2. Stabilized fertilizers: have a chemical inhibitor to either stop the oxidation of ammonium (NH_4^+) to nitrate (NO_3^-) by bacteria or to slow the enzymatic transformation of urea to NH_4^+ (Trenkel, 1997).
3. Controlled-release fertilizers (CRFs): urea, ammonium nitrate, potassium nitrate, or other soluble fertilizer materials coated with a polymer (polyethylene and ethylene-vinyl-acetate or thermoplastics), resin, sulfur, or a hybrid of sulfur coated urea (SCU) coated with a polymer or resin (Figure 1).

Figure 1. Application of CRFs in raised beds, prior to plastic mulch installation.
Credits: Monica Ozores-Hampton

This fact sheet examines laboratory, growth chamber, greenhouse, and field methods for measuring nitrogen release in controlled-release fertilizers (CRFs).

CFRs release nutrients in water at a predictable rate when used at the manufacturer-specified temperature (Trenkel, 2010). The European Committee for Standardization's method determines nutrient release time based on 75% nutrient release from CRFs. The European Union has developed both standard and accelerated laboratory procedures for measuring N release from CRFs; however, researchers in the United States are still developing a universal test for CRFs for commerce purposes. Growth chamber and

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greenhouse methods are used to evaluate or compare how CRFs will act in a particular controlled environment, while field methods are used to measure N release in commercial vegetable field conditions (Broschat and Moore, 2007; Huett and Gogel, 2000; Simonne and Hutchinson, 2005). Each research method has its own advantages and disadvantages (Engelsjord et al., 1996; Sartain et al., 2004; Simonne and Hutchinson, 2005).

Laboratory Methods

Laboratory methods allow for CRF incubation in controlled environmental conditions, compared to field conditions. They may be used to compare and quickly screen CRFs but can only be used to predict laboratory release, not field release when used alone. There are two types of laboratory methods used that are based on release time: the standard method, which incubates CRF for specified nutrient release time or until a threshold amount of nutrients (e.g., 75%) are released (Dai et al., 2008; Du et al., 2006; European Committee for Standardization, 2002); and the accelerated method, which incubates CRF for a shorter time at a higher temperature (Dai et al., 2008; Du et al., 2006; European Committee for Standardization, 2002).

1. **Temperature-controlled incubation method (TCIM)-standard:** The standard TCIM incubates a beaker containing CRF and water at a constant temperature of 77 °F (Dai et al., 2008; Ko et al., 1996; Shaviv, 2001) (Figure 2). Incubation times are based on manufacturer stated release length (4-month release), or based on research objectives such as measuring release until 100% of the urea is released (Dai et al., 2008; Ko et al., 1996).

Figure 2. Standard temperature-controlled incubation of 0.44 oz of CRF in 8.4 fl oz water incubated at 77 °F
Credits: Monica Ozores-Hampton

2. Temperature-controlled incubation method (TCIM)–accelerated: This method uses jacketed chromatography columns in hollow glass tubes surrounded by an integrated water jacket where the sample can be placed in the inside while the water controls the temperature (Sartain et al., 2004) (Figure 3). Four separate extractions per sample are used, with temperatures increasing from 77 to 140°F over time to obtain a release curve. Water or dilute citric acid (0.2 N) can be used as the extracting solution. Another method uses five separate funnels to incubate trincote, resin-coated CRF at 122 to 194 °F and continuously leaches CRF samples for 6 hours (Dai et al., 2008). The results of the high temperature incubations were compared to a standard TCIM at 77 °F and revealed that 176 °F was the optimal temperature partially due to reduced coating integrity at 194 °F (Dai et al., 2008). Accelerated TCIMs have the advantage of reducing the time and labor cost compared to the standard TCIM, but neither predict field release.

Figure 3. An accelerated temperature controlled incubation unit as described by Sartain et al. (2004).
Credits: Luther Carson

Growth Chamber and Greenhouse Methods

Growth chamber and greenhouse methods may be used to test CRF products in conditions more similar to a particular production system, compared to laboratory methods (Abraham and Rajasekharan Pillai, 1996; Broschat, 1996; Broschat and Moore, 2007; Sato and Morgan, 2008).

1. Column extraction method: Columns that measure 11.8 inches long and 2 to 3 inches in diameter are used (Broschat, 1996; Broschat and Moore, 2007; Huett and Gogel, 2000; Sartain et al., 2004). The bottom end of the

column is fitted with mesh or gauze, then placed in a funnel or reservoir, or capped (the cap contains a luer fitting for drainage). The columns are positioned vertically and filled with media and the top is capped (Figure 4).

A 0.176 oz sample of CRF can be placed 0.4 to 2 inches below the media surface. The standard column method uses sand washed with hydrochloric acid to reduce the likelihood of nutrient retention by the media. Columns are leached at different frequencies and volumes of water depending on the goal of the research and the size of the column. Huett and Gogel (2000) and Broschat and Moore (2007) leached columns three times per week using 1.7 and 2.7 fl oz of water, respectively. The leachate was collected once per week for 53 weeks (Huett and Gogel, 2000). Conversely, Broschat and Moore (2007) collected the leachate weekly until its nutrient concentrations were less than 3 ppm; a resin coated fertilizer had 100% nitrate release and 10% iron release in 64 and 40 weeks, respectively, before the concentrations fell below 3 ppm. Medina et al. (2008) and Medina et al. (2009) leached columns with 16.9 fl oz citric acid (0.1 N) at increasing intervals from 7 to 270 days and 7 to 180 days, respectively. Incubation temperatures in growth chambers or greenhouses should match field soil temperatures or the manufacturers' specified temperature if testing manufacturers' claims.

Figure 4. Columns made of PVC used to test CRF nutrient release.
Credits: Medina et al., 2011

2. Plastic bag method: Plastic zipper bags are filled with 3.5 to 8.8 oz of soil and a sample of CRF incubated at room temperature (Cahill et al., 2010; Sartain et al., 2004) (Figure 5). Cahill et al. (2010) found a high amount of variation between the results for polymer-coated urea, phosphate coated urea, granular urea, and urea ammonium nitrate incubated using this method. Sartain et al. (2004) reported a strong smell of NH3 when the bags were opened and a maximum N recovery rate of 60% from biosolids, SCU, urea-formaldehyde, isobutylidene diurea, and urea. For these reasons, the plastic bag method will be a poor research tool to use in CRF-N research, making the column extraction method the preferred greenhouse and growth chamber method.

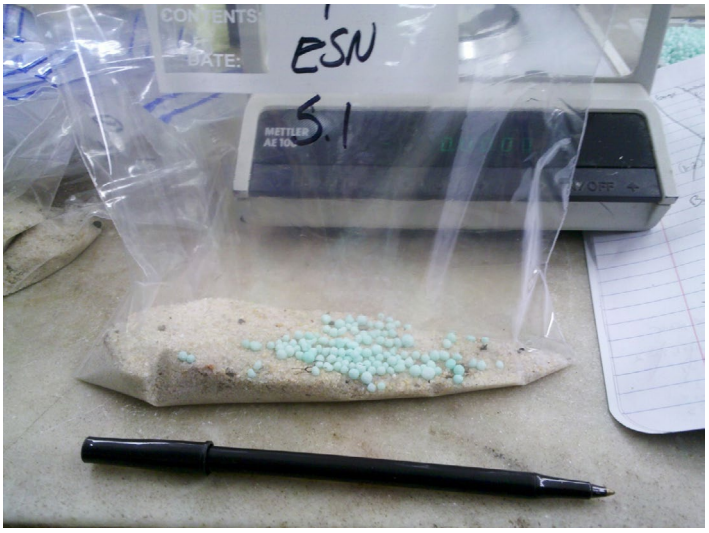

Figure 5. A plastic bag filled with sand and a CRF equivalent to 0.035 oz nitrogen for use in the plastic bag method.
Credits: Luther Carson

Field Methods

Field methods can be used to determine how CRFs will release under actual field conditions. The field method should subject the CRF to an environment similar to CRFs applied in vegetable production systems (Wilson et al., 2009). Ideally, CRF-N release matches crop N uptake and releases N throughout the entire vegetable production cycle (Lammel, 2005). Nitrogen release should be measured throughout the entire crop cycle, or until 75% of the N is released or recovered (Trenkel, 1997).

1. Pouch method: Pouches are made of fiberglass mesh screen that allows movement of moisture to the CRF prill (Figure 6). For the release curves to accurately reflect environmental conditions, the pouch materials must not interfere with water movement to CRF prills. Polymer coated urea incubated in polypropylene mesh pouches with 1.2 mm^2 openings had significantly greater N release than pouches constructed from weed block material with 0.07 mm^2 openings (Wilson et al., 2009). Pouch dimensions range from 2 × 2.4 to 5×5 inches with CRF sample sizes ranging from 0.05 to 0.18 oz N (Gandeza et al., 1991; Haase et al., 2007; Jacobs et al., 2003; Medina, 2006; Wilson et al., 2009; Zvomuya et al., 2003). Soil can be included into the pouch with the CRF sample (Broschat, 2005; Gandeza et al., 1991). Pouch placement in the field should follow growers' production practices, i.e. buried under vegetable beds

with plastic mulch or in open potato hills (Broschat, 2005; Medina et al., 2008; Medina et al., 2009; Wilson et al., 2009; Zvomuya et al., 2003). Pouches can be collected at pre-determined times during the vegetable production cycle and remaining N in the CRF can be determined. Medina et al. (2008) found that CRFs performed differently in citrus groves with different row orientation (north to south vs. east to west) due to the different wetting and drying patterns found in the groves. Differences due to grove-row orientation show that the pouch method allows CRF prills to be subjected to real field environments.

Figure 6. A 6 × 6 inch pouch containing controlled-release fertilizer equivalent to 0.12 oz nitrogen ready to be installed in the field.
Credits: Luther Carson

2. Pot-in-pot method: This method consists of two 8-inch pots nested together separated by a 3/4 inch spacer (Figure 7). The interior pots with screened drain holes are filled with soil and 0.17 to 0.22 oz CRF samples. Covered pots are buried in a potato hill with 1 inch of the bottom pot above the soil surface (Simonne and Hutchinson, 2005). Incubated pots are leached with water at pre-arranged dates, and the following day leachate volumes are collected and measured.

Figure 7. Two 8-inch pots nested together separated by a 3/4 inch spacer. The interior pot has screened drain holes and is filled with soil and a 0.17 to 0.22 oz CRF sample.
Credits: Joel Mendez

The pot-in-pot method and the column method measures N released from the CRF rather than N remaining in the prills. Measuring released N takes into consideration soil microbial activity on the N, thus being representative of plant available N (Simonne and Hutchinson, 2005). The project research objectives will determine the importance of measuring N released in leachate, while requiring additional labor, or measuring N remaining in the CRF prill. Important factors to consider are that environmental field conditions can be highly variable, and CRFs are temperature dependent, therefore field studies must include all growing seasons and multiple years (Fraisse et al., 2010). For vegetable production, both CRF field methods can be viable methods for measuring CRF-N successfully.

Correlations between the Methods

Controlled-release fertilizer nutrient release differs in free water, water saturated sand, and sand at field capacity (Du et al., 2006). Temperature-controlled incubation methods that do not correlate with other methods can offer only restricted practical use for commercial vegetable production because the results will not reflect nutrient release obtained under field conditions. Sartain et al. (2004) compared the accelerated TCIM extraction of polymer SCU with a column extraction method at room temperature and found that the accelerated TCIM may be able to predict N release from column incubations accurately. Results from TCIM have not been correlated with any field method. In field conditions, there are several factors to consider, such as release time, temperature, moisture, placement, rate and cultural practices, making the correlation difficult to achieve (Sartain et al., 2004).

Procedures to Measure Nitrogen

With all CRF research methods (laboratory, growth chamber, greenhouse, and field methods) N concentration in leachate or in the CRF prills needs to be measured after incubation. Methods to measure N include:

- **Total Kjeldahl N (TKN):** TKN is the standard and most popular method. It is a time-consuming laboratory procedure, which includes concentrated sulfuric acid and sodium hydroxide. All CRF-N sources and research methodologies may use TKN to measure N concentrations (Gandeza et al., 1991; Greenberg et al., 1985; Haase et al., 2007; Zvomuya et al., 2003).

- **Prill weight loss:** This is a quick procedure where the mass of dried-incubated prills are subtracted from the original dry prill mass (Salman et al., 1989; Savant et al., 1982). Unfortunately, this method may only be used with pouch-incubated urea CRF. Each type of ion diffuses out of the CRF prill at a different rate; therefore, it cannot be assumed that the ion ratio inside the incubated CRF prill and the non-incubated CRF prill are equal. For example, a potassium nitrate fertilizer is composed of 50% K^+ ions and 50% NO_3^- ions. Nitrate releases more quickly than K^+, thus K^+ will represent a larger portion of the nutrients in the prill near the end of a trial (Broschat and Moore, 2007).
- **Combustion and colorimetric N determination with an autoanalyzer:** This method uses a solution, so both methods may be used with any of the CRF research methods or N sources (Pack et al., 2006 and Wilson et al., 2009).
- **Ion specific electrodes:** This method may be used to measure N in leachate and solubilized (homogenized) CRF prills; however, free urea cannot be measured using these electrodes unless the urease enzyme is added and the solution is incubated (Broschat, 2005 and Guilbault et al., 1969).

Wilson et al. (2009) compared prill weight loss to combustion methods and found that both were equally reliable methods for measuring N release.

Cost of Laboratory Analysis

The accelerated TCIM is preferred when compared to the standard TCIM method due to savings on time and labor costs. Column extractions can be used to test new CRFs before going to the field from controlled environments, but column extractions can be time consuming with associated high cost. Field methods will be the preferred research tools by vegetable growers until the accelerated TCIM has been correlated and calibrated to field studies with a positive crop response, thus determining a CRF's suitability for vegetable production in a shorter amount of time.

Laboratory analysis of N remaining in CRF prills or in leachate varies in cost. Prill weight loss costs the least per sample to measure N, but this method can only be used with pouch-incubated CRF urea. Ion specific electrodes are the next most inexpensive method, but each electrode only measures one N species, therefore more than one electrode must be used to measure total inorganic N. Both organic-N and ammonium-N (NH_4-N) may be measured using TKN but it costs more to conduct than the prill weight loss or the ion specific electrode method. Total Kjedahl N does not measure nitrate-N (NO_3-N), but modified methods are available to measure NO_3-N along with NH_4-N and Organic-N (Latimer, 2010). Selection of a laboratory that uses the modified method will reduce the number of analytical tests required to measure total fertilizer N. Colorimetric measurements for NO_3-N or NH_4-N need separate analyses. Using both colorimetric N analyses would be more expensive than all methods but TKN. The combustion method costs around the same amount as TKN; neither method provides N species information like colorimetric analysis. Since the prill weight loss method and the combustion method are equally acceptable, it would be fiscally responsible to use prill weight loss to determine N release, when CRF type allows. Depending on the amount of information needed regarding N types, multiple methods can be used to measure N released for CRF on vegetable production systems.

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Table 1. **Nitrogen (N) determination method to use with different incubation methods, type of N, and controlled-release fertilizer (CRF) N source.**

N determination method	CRF incubation methods[z]	Type of N measured	CRF-N source
Total Kjeldahl N	L,G,F	Organic-N, NH_4-N, and with modification NO_3-N	NH_4-N, NO_3-N and urea
Prill weight loss	F (pouch method only)	None – measures change in mass	urea only
Combustion	L,G,F	NH_4-N, NO_3-N, and urea-N	NH_4-N, NO_3-N and urea
Colorimetric	L,G,F	NO_3-N, or NH_4-N	NH_4-N, NO_3-N and urea
Ion specific electrodes	L,G,F	NH_4-N, NO_3-N and urea but it must be transformed with urease	NH_4-N, NO_3-N and urea

[z]L = Laboratory, G = growth chamber and greenhouse, and F = field methods.
[y]Abbreviations: ammonium-N, NH_4-N; nitrate-N, NO_3-N

Table 2. **Cost of laboratory analysis for nitrogen (N) content remaining in CRF prills in a field trial consisting of six replications, five treatments and eleven sampling dates (Medina et al., 2008).**

N determination method	Cost/unit ($)	Total cost ($)[w]
Total Kjeldahl N (TKN)[z]	8.25 to 13.20	2,723 to 4,356
Prill weight loss[x]	0.17 to 0.20	56 to 66
Combustion	7.5 to 11.4	2,475 to 3,762
Colorimetric (NO_3-N or NH_4-N)[y]	6.00 to 10.00 7.00 to 10.00 11.6 to 12.5	1,980 to 3,300 (NO_3-N) 2,310 to 3,300 (NH_4-N) 3,828 to 4,125 (Both)[u]
Ion Specific Electrode	0.25 to 0.33	83 to 110

[z]The laboratory method unit cost for TKN, combustion, and colorimetric are a range of prices given on analytical laboratories websites (University of Minnesota Research Analytical Laboratory, St. Paul, MN; Cornell Nutrient Analysis Laboratory, Ithaca, NY; University of California Davis Analytical Laboratory, Davis, CA; and Penn State Agricultural Analytical Services Lab, University Park, PA) where the applicable laboratory tests are performed.
[y]NO_3-N, nitrate nitrogen; NO_4-N, ammonium nitrogen
[x]Unit cost for prill weight loss and ion specific electrode methods are based on a laboratory technician measuring 50 to 60 and 30 to 40 samples per hour, respectively, at an hourly rate of $10/hr and availability of laboratory equipment (D.E. Lucas, Personal Communication).
[w]Total costs estimates, collected in Jan. 2012, do not include shipping and do not include drying time, which may be necessary for some methods, with inconsequential labor costs to evaluate dryness.
[u]This cost represents the price set by labs when both NO_3-N or NH_4-N are measured.