# Detecting Phosphorus with Plasma Emission Spectroscopy May Require Unique Field Calibration Data

By Antonio P. Mallarino and T. Scott Murrell

Using inductively coupled plasma emission spectroscopy (ICP) to measure phosphorus (P) in the Mehlich 3 (M3) extractant produces higher test results than the traditional colorimetric procedure (COL), requiring the creation of new soil test interpretation categories. The M3-ICP procedure should be considered a different soil P test than M3-COL. Failing to do so could result in large recommendation errors and significant underestimation of supplemental P needs.

In a soil testing laboratory, one of the first steps in generating a soil test P value is to get a portion of the total soil P into solution that is proportional to the soil P available to plants. To do this, a small measure of soil from a submitted sample is mixed with a solution of different chemicals, termed an extractant. In the Midwestern U.S., three extractants are commonly used for P: Bray P-1, Olsen, and M3. Mehlich 3 is gaining popularity among laboratories, because it can be used to extract more elements than just P and it produces reliable results across a wider range of pH levels than does Bray P-1.

Once P is extracted, the amount dissolved must be measured. There are two commonly used techniques for doing this: 1) COL, and 2) ICP. In the past, soil testing laboratories used only the molecular absorbance method. Recently, however, many laboratories have begun using ICP. This article briefly describes these detection methods and how they influence soil test interpretations appropriate for the M3 extractant.

## COL

**A standard P detection method for many soil P extractants is COL.** After a soil test extractant has dissolved P from a soil sample, the solution is filtered to obtain a clear solution. More chemicals are then added that react with dissolved orthophosphate P ($H_2PO_4^-$ or $HPO_4^{2-}$) and turn the solution blue. The blue solution is then placed in a clear cell in a spectrophotometer. This instrument measures the absorption of energy by P molecules (**Figure 1a**). Light of a specific wavelength, coming from a radiation source, is directed at the sample. Some of the radiation is absorbed by the molecules in the sample. Radiation not absorbed passes through the sample and is captured by a detector. As P concentration in the sample increases, more radiation is absorbed, reducing the intensity of the radiation transmitted to the detector. Consequently, measuring the intensity of transmitted radiation allows P



**The blue solution** is characteristic of the traditional colorimetric procedure for P detection.

Note: In this article, the classic M3 colorimetric procedure is referred to as M3-COL, while a M3 procedure that uses ICP is referred to as M3-ICP.
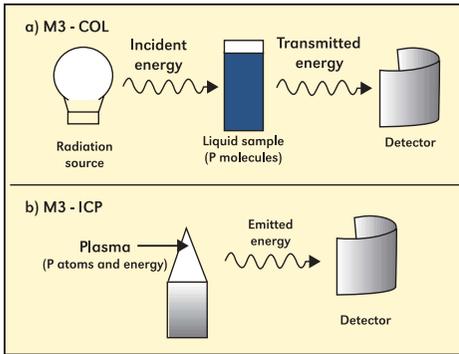
**Figure 1.** Simplified diagrams show how P is quantified in the Mehlich 3 extractant using a) the colorimetric procedure (M3-COL), and b) inductively coupled plasma emission spectroscopy (M3-ICP).

concentration to be quantified.

### ICP

**Another means of measuring P in any extractant solution is ICP.** In this method, the sample is heated to such an extreme temperature that all molecules decompose into atoms to form a gas. The source of extreme heat is an inductively coupled plasma (**Figure 1b**). The high energy plasma excites electrons in the P atom to a higher energy state. This excited state doesn't last long, and soon the electrons return to their original energy states. During this return, energy is emitted from the sample and hits a detector. The more P that is in the sample, the greater the emitted energy detected. In this method, detected energy comes from all P compounds, not just orthophosphate P.

### What the Procedures Measure

These two methods do not measure the same things. In M3-COL, only orthophosphate P is measured. Most of the P extracted by soil tests is in the orthophosphate form, but there are also other P compounds that may be present, such as simple organic P compounds and P associated with very small soil particles that sometimes pass filters. These additional compounds are not detected in M3-COL, but can be detected in M3-ICP. **Consequently, P measured using an M3-ICP tends to be greater than P measured by M3-COL, even using the same soil sample or extracted solution.**

Because the M3 extractant measures several other elements also, some laboratories use ICP for measuring P in a M3 extractant, but use COL when measuring P from Bray P-1 or Olsen extractants. As of 2003, more than 60% of the soil testing labs in the North American Proficiency Testing Program were requesting proficiency testing for M3-ICP, although fewer labs use M3-ICP for testing farmers' samples.

### Need for Field Calibration Experiments

For any soil test method to have meaning, values generated must be calibrated to crop yield response in the field. With M3-ICP and M3-COL methods measuring different things, many wondered if different field calibration data were needed for each procedure. To answer this question, field calibration research for corn was conducted across 78 site-years. These sites represented 17 soil series in which row crop production predominates in Iowa. Thirty-one trials evaluated four P fertilization rates for corn managed with plow and/or disk tillage; 13 trials evaluated three P fertilization rates applied either broadcast or banded for no-till corn; and 15 trials evaluated three P rates applied either broadcast or banded for ridge-till corn. Corn grain yield and soil samples were collected in each site-year.

Corn grain yield data were expressed as relative responses to P. Relative response was calculated for each site-year by dividing the average yield of the control plots (no P applied) by the average yield of the treatment with the highest P rate. This fraction was then multiplied by 100 to express relative response as a percentage.

Two sub-samples were taken from each soil sample. Each sub-sample was mixed with the M3 extractant. One aliquot of each sub-sample was put through the M3-COL procedure and a second aliquot was subjected to M3-ICP analysis.

The average soil P measured by M3-ICP and M3-COL across all sites was 31 and 19 parts per million (ppm), respectively. These averages show that for the same samples,

M3-ICP measured more P than M3-COL. **Figure 2** shows the relationship between P measured by M3-ICP and M3-COL for all site-years. The high $r^2$ value indicates that both analyses are well correlated and highly significant. If both procedures measured the same amount of P, then most of the points would fall along the 1:1 line. However, most points fall above this line, demonstrating the higher quantity of P detected by M3-ICP across the range of soil test P levels.

**Figure 3** shows corn grain calibration data from the field experiments. Grain yields across all site-years ranged from 87 to 210 bu/A. **Figures 3a and 3b** show the relationships between relative corn grain yield responses to applied P and P measured by either M3-COL or M3-ICP, respectively.

In both graphs, the optimum ranges are defined as soil test levels most profitable to
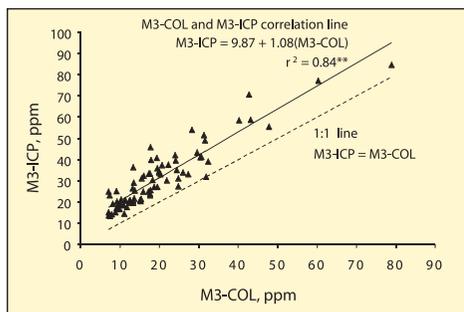
**Figure 2.** Correlation between M3 P measured by the colorimetric procedure (M3-COL) and inductively coupled plasma emission spectroscopy (M3-ICP).

maintain over time with regular P applications.

These results, combined with crop response and economic models, led to the formation of new soil test interpretation ranges for the M3-ICP test, shown in **Table 1**. This table shows that higher soil test P values are used to define wider soil test categories when M3-ICP is used.

The categorization of high and low subsoil P levels is based on previous research showing that lower P levels are needed at the surface when subsoil P levels are higher. Recom-
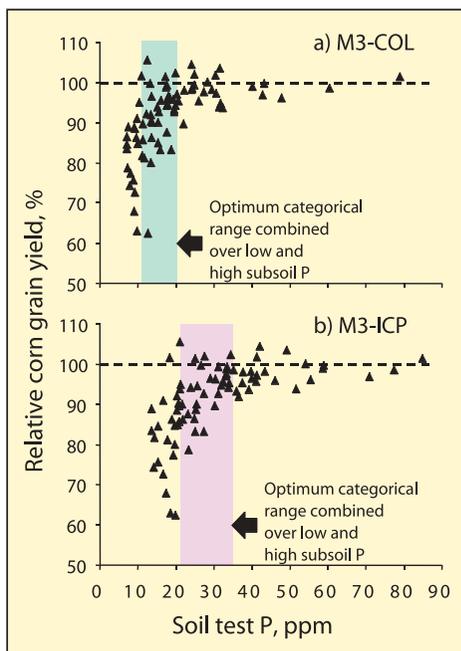
**Figure 3.** Relationship between relative yield response of corn grain and M3 P measured by the colorimetric procedure (M3-COL) and inductively coupled plasma emission spectroscopy (M3-ICP).

mendations in **Table 1** are based on soil samples taken to a 6 in. depth.

These new ranges took effect in 2002 and were published in the Iowa State University Cooperative Extension Bulletin PM 1688 entitled "A General Guide for Crop Nutrient and Limestone Recommendations in Iowa." It may be downloaded at

**Table 1.** Updated Iowa State University soil test P interpretation classes for P measured by M3-COL and M3-ICP.

| Soil test | Very low | Low | Optimum | High | Very high |
|---|---|---|---|---|---|
| | | | - - - - - - - - - - - - - - ppm - - - - - - - - - - - - - - | | | |
| | | | Low subsoil P | | | |
| M3-COL | 0-8 | 9-15 | 16-20 | 21-30 | 31+ |
| M3-ICP | 0-15 | 16-25 | 26-35 | 36-45 | 46+ |
| | | | High subsoil P | | | |
| M3-COL | 0-5 | 6-10 | 11-15 | 16-20 | 21+ |
| M3-ICP | 0-10 | 11-20 | 21-30 | 31-40 | 41+ |

# New Publication Offers Insight about Food Quality/Safety Concerns

**W**hile consumers today have ready access to perhaps the most nutritious, safe, and affordable food products in history, many continue to have doubts and questions about food safety, quality, and other issues.

A new publication from PPI/PPIC and the Foundation for Agronomic Research (FAR) addresses this situation. The booklet is titled *There's WHAT in My Food?* Presented primarily through a question/answer format, the 24-page publication includes colorful illustrations in a friendly style to appeal especially to non-farm audiences.

For those involved in agriculture and fertilizer industry work, this publication can be another useful and effective tool in providing a fresh, positive message to consumers.
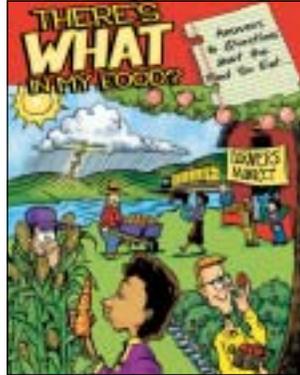
The booklet is available for purchase at $2.00 per copy (plus shipping), with discounts for larger quantities. Some sample pages of the booklet may be viewed as PDF files on the Institute's website through this link: www.ppi-ppic.org/food. An order form is also available there as a PDF file.

For additional information or to order, contact: Circulation Department, PPI, 655 Engineering Drive, Suite 110, Norcross, GA 30092-2837. Phone: (770) 825-8082; fax (770) 448-0439. ᴮᶜ

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>www.extension.iastate.edu/Publications/PM1688.pdf<.

Although not discussed in this article, differences between M3-COL and M3-ICP for soybeans were analogous to those discussed for corn grain.

### Summary

Using M3-ICP resulted in higher soil test levels than those produced with M3-COL. The additional P measured with the M3-ICP test varied greatly across Iowa soils and cannot be accurately predicted from results of the M3-COL test. This required that new field calibration data be collected for the M3-ICP test. Results from field trials showed that when M3-ICP was used to measure P concentration in the M3 extractant, new soil test interpretation classes were needed. Laboratories should clearly inform their clients of which P detection method is being used with the M3 extractant. ᴮᶜ

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