

# Review of Applicability of EPA's Part 503 Biosolids Rule on Phosphorus Minerals recovered at Water Resource Recovery Facilities

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# Background

Nutrient recovery from Water Resource Recovery Facilities (WRRF) yields valuable products that have applications in agricultural, horticultural, and industrial markets. Nutrients such as nitrogen and phosphorus have long been reclaimed as components of WRRF-generated biosolids, products typically with varying total solids content and high organic matter content. The generation and use of biosolids products - referred to under the broader term 'sewage sludge' - are regulated under the United States Environmental Protection Agency's (EPA) Part 503 rule of the Code of Federal Regulations, which will herein be referred to as simply "Part 503".

More recently, technologies that precipitate nutrients such as nitrogen and/or phosphorus as relatively pure mineral compounds have been implemented by WRRFs in the United States. Various technologies of this type continue to be commercialized and adopted as tightening limits on effluent nutrient loading have swayed the water resource recovery Industry's interest in high-efficiency nutrient recovery solutions.

As a result of the chemistry and physics of how these high-efficiency nutrient recovery technologies precipitate and dewater their products, these systems yield mineral compounds with low organic content, very different in chemical composition and biological behavior from products falling under the definition of sewage sludge. CNP's MagPrex® technology, with its recovered struvite, is a prime example of this adoption of newer methods to remove nutrients from wastewater streams and meet ever tighter limits on nutrient loading for agricultural, industrial and municipal effluents.

Recovery of high-purity minerals such as struvite can offer WRRFs a source of revenue and further help the industry attain a vision of resource recovery from wastewater. After recovery at a WRRF, these products can be sold with or without further processing as valuable consumer products, such as fertilizers with clear applications in agriculture, forestry, horticulture, and home gardening. Because the primary federal guideline used in the municipal wastewater industry for products produced at WRRFs is the Part 503 rule - which was written for products of much different physical and biological characteristics than these types of high-purity precipitated minerals – regulators and the wastewater treatment industry as a whole have yet to devise and enforce a set of regulations which are appropriate for these new products.

The applicability of Part 503 on WRRF-recovered struvite will be further discussed in this document, and CNP offers its stance on what it considers to be the appropriate regulatory guidelines for struvite recovered by MagPrex installations at WRRFs. This document is intended to provide CNP's understanding of the Part 503 rule, existing industry discussion around the applicability of the Part 503 rule on recovered minerals, and the qualities of struvite that has been recovered from existing pilot or full-scale MagPrex installations. This document is not intended to provide definitive and specific recommendations for WRRFs considering implementing an MagPrex installation or any other nutrient recovery system, as site-by-site physical and operational factors must be considered before determining an appropriate regulatory approach for struvite produced at a particular WRRF.

## Struvite: Chemical, Physical, Biological Properties

The pure struvite mineral - Ammonium Magnesium Phosphate (AMP) or Magnesium Ammonium Phosphate Hexahydrate ( $NH_4MgPO_4\cdot 6H_2O$ ), also known in the WRRF industry as Magnesium Ammonium Phosphate (MAP, not be confused with the common fertilizer, monoammonium phosphate) - is a transparent to yellowish white phosphate crystal consisting of 9.0% magnesium, 5.7% nitrogen, and 12.6% phosphorus. The struvite mineral



contains six water molecules that constitute the hydration shell of the magnesium atom in the crystal unit cell and are therefore essential to the integrity of the struvite's crystalline structure (see molecular structure, Figure 2). Struvite is precipitated and recovered by the MagPrex process from high-ammonium, high-phosphate streams post anaerobic digestion at WRRFs, with the addition of magnesium (Figure 1).



Figure 1: Struvite harvested by the MagPrex system under controlled conditions

Pure struvite has a particle density of 1.7 Mg m<sup>-3</sup> and a typical bulk density of 1 Mg m<sup>-3</sup>. Bulk density of MagPrex-recovered struvite has been documented as 0.99 Mg m<sup>-3</sup>. Based on compositions of nitrogen, magnesium, and phosphorus exhibited in samples harvested by four different MagPrex installations, laboratory analyses have shown the material is 87.4% pure mineral struvite, on average.

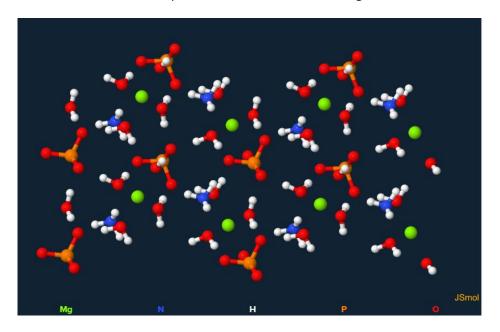


Figure 2. Molecular structure of Struvite lattice, courtesy of the Virtual Museum of Minerals and Molecules, University of Wisconsin – Madison. Ammonium, blue-white molecules; magnesium, green atoms; phosphate, orange-red molecules; water, red-white molecules.



Struvite is a sparingly soluble phosphate salt, and this combined with initial feed conditions can affect the formation of the crystals in a variety of ways. According to Shih and Yan (2018)

"Struvite has low solubility in water, with a solubility product constant [Ksp] of  $10^{-13.26}$ . In contrast, it is readily soluble in acidic environments:  $0.033 \text{ g} \ 100^{-1} \text{ mL}^{-1}$  in 0.001 M HCl and  $0.178 \text{ g} \ 100^{-1} \text{ mL}^{-1}$  in 0.01 M HCl at  $25^{\circ}$ C. [M]orphology of struvite particles differs under various formation conditions and includes a needle-like shape, a cube-like shape, and irregular shapes. According to the summary of Rahman et al. (2014), the length of struvite particles varied from 15  $\mu$ m (Zhang et al., 2009) to 3.5 mm. [P]hosphate concentration in the inflow together with the retention time of particles in reactors had a significant effect on the particle size of the struvite products obtained."

Struvite has variable particle size, dependent on feed characteristics (pH and P concentration) and the hydraulic retention time of the feed in the MagPrex reactor. Natural struvite is largely a biogenic mineral which forms mostly in the urinary and digestive tracts of live animals, in animal excreta or wastewater handling systems, or during the decomposition of proteinaceous compounds under conditions of high available Mg and phosphate and high pH.

Typically, once dry, struvite is inert and does not allow for growth of microorganisms such as fungi or bacteria and it behaves mostly like sand while slowly dissolving if placed in a moist environment, with acceleration of the dissolution as a function of acidity.

Partly due to its high purity and partly because of its crystalline nature, air-dried struvite recovered from WRRFs has little tendency to produce molds or odors.

### Thermal Decomposition of Struvite

The six water molecules of the struvite mineral constitute 44.08% of the mineral's total mass and are proven to evaporate when struvite is exposed to heat. Exposure of pure struvite to temperatures as low as 55° C has been shown to extract crystal water from the struvite molecule, decomposing the mineral lattice and inherently contributing to mass loss in variable amounts, depending on temperature, exposure time, and rate of incremental heating (Bhuiyan et al., 2008; Frost et. al, 2004).

2In addition to crystal mass loss attributable to the dehydration of six crystal waters of struvite, 7.24% of the struvite mineral mass is composed of ammonium (NH<sub>4</sub>) which is also susceptible to off-gassing as NH<sub>3</sub> when exposed to heat. Frost et. al (2004) found mass loss of ammonia beginning at a temperature of 39.5° C, with peak mass loss occurring at 84° C when struvite was heated at 2° C min<sup>-1</sup> for 180 minutes.

Studies on thermal decomposition of WRRF-recovered struvite (87% pure; average for 4 different WRRFs) conducted by CNP and partner company Nutrient Recovery and Upcycling (NRU) showed mass loss trends consistent with other peer reviewed studies published on the topic. Notably, MagPrex-derived struvite exposed to 105° C – the upper temperature advised by SM 2540 G described below – for about one hour yielded an average mass loss of 46% during two trials. The combined masses of ammonia (17 grams per mole) and six hydrated water molecules (18 grams per mole) constitutes 51.42% (142 grams) of the total mass of struvite (245.3 grams per mol). For a crystal made of pure struvite, the total theoretical crystal mass loss can be over 50% of the initial mass when exposed to 105° C for an extended period of time.



# Applicability of Part 503

The Part 503 rule sets standards for quality control of dewatered sludge and related products derived from sewage sludge, offering classifications of Class B, Class A, and Class A Exceptional Quality (Class A EQ), with the highest quality material being Class A EQ products. The components of quality assurance can be summarized by a three-part methodology: a) analysis of heavy metals contamination; b) pathogenic potential elimination; and c) vector attraction reduction. These standards are set to ensure safe and appropriate disposal or use of sewage sludges. Each of the three components of quality assurance, and the implications of their application to struvite, are described in further detail later in this document.

### Standard Method 2540 G

One primary consideration for regulating mineral products recovered at WRRFs under the Part 503 rule is the applicability of standard laboratory methods used for sewage sludges on these types of mineral products. Perhaps the most significant is the Standard Method 2540 G for measuring total, fixed and volatile solids in solid and semi-solid samples. By this method, determination of total solids for solid samples is conducted by placing the sample in an oven at 103° to 105° C overnight, followed by repeat drying in intervals of one hour until weight change is less than 4% or 50 mg (SM 2540 G, 1997). This method is applied to quality control analyses of materials regulated under Part 503 and inherently affects reported laboratory results for analytes reported on a dry weight basis, such as pollutant concentrations and pathogenic analyses.

The thermal decomposition of struvite described above not only degrades the fertilizer quality of the struvite mineral, it also makes the application of Standard Method 2540 G inappropriate in the determination of solids measurements since there is mass loss attributable to the loss of struvite's crystalline ammonia and/or crystal water molecule in addition to mass loss due to free water that is not part of the struvite molecule.

### **Pollutant Concentration Limits**

Part 503 sets limits for pollutant concentrations (heavy metals concentrations) allowable in products intended to be sold or given away in a container and intended for land application. Table 1 exhibits the limits to be met for Class A EQ products, for reference.

**Pollutant** Monthly average concentration (milligrams per kilogram)<sup>1</sup> Arsenic 41 Cadmium 39 1500 Copper 300 Lead Mercury 17 Nickel 420 Selenium 100

Table 1: Pollutant Concentrations

Zinc

[58 FR 9387, Feb. 19, 1993, as amended at 64 FR 42571, Aug. 4, 1999]

Reporting for the regulated pollutants is required on a dry weight basis. As discussed, laboratory reported concentrations on a dry weight (total solids) basis for analytes in struvite will inherently yield higher-than-actual

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<sup>&</sup>lt;sup>1</sup>Dry weight basis.



results due to the confounding mass loss of ammonia and crystal water at 105° C. Though all struvite sampled from previous MagPrex pilot installations have yielded pollutant concentrations significantly below the provided limits, it is worth noting that actual concentrations will be variably lower than reported by analytical laboratories on a dry weight basis.

### Pathogenic Potential

The third component of quality control for materials regulated by Part 503 and intended for land application promotes reduction in pathogenic growth potential. Material must exhibit either fecal coliform density of less than 1000 Most Probable Number (MPN) per gram of total solids (dry weight basis) or *Salmonella*, sp. bacteria density of less than three Most Probable Number per four grams of total solids (dry weight basis).

The regulation offers six alternatives for treatment of Class A material to meet the pathogenic indicator density requirements. Of the alternatives, the only reasonable treatment relating to MagPrex-derived struvite is that by pasteurization (exposure to a temperature at minimum 70° C for a minimum of 30 minutes) or a different time and temperature exposure as determined by the following equation:

$$D = \frac{131700000}{10^{0.14t}}$$

Where D = time in days; t = temperature in degrees Celsius. By this equation, minimum temperature must be 50° C and minimum time period must be 20 minutes.

Consistent with the previous discussions of this report, meeting heat exposure requirements will result in loss of crystal waters and ammonia. It may be possible to expose struvite to heat up to 100° C for a very short time with less significant mass loss not attributable to surface moisture. However, based on internal testing the minimum exposure time of 20 minutes required by Part 503 would result in significant crystal mass loss.

Given the mass loss implications of exposing struvite to high heat and that the thermal treatment requirement is the most applicable Class A pathogenic reduction solution to the MagPrex process, meeting the pathogenic reduction method requirements as currently written in Part 503 is a difficult if not impossible challenge. Further, as noted previously, MagPrex-derived struvite is a primarily mineral material with trace amounts of the organic materials. Potential for pathogenic growth after harvest from the MagPrex reactor and significant removal of surface moisture is highly unlikely, meaning that upon evidence that MagPrex-derived struvite exhibits appropriate density of either fecal coliform or *Salmonella*, sp. bacteria, the material should not be expected to harbor pathogenic regrowth after it is containerized or otherwise prepared for give away or sale.

#### **Vector Attraction Reduction**

Part 503 mandates vector attraction reduction to be met for all materials applied to land, lawn, home, garden, etc. Specifically, any one of the first eight listed vector attraction options provided in Part 503 must be met for material applied to a lawn or home garden and/or sold or given away in a bag or other container for land application. These first eight vector attraction options are listed and summarized below:

- Option 1: Reduce the mass of volatile solids by a minimum of 38%
- Option 2: Demonstrate vector attraction reduction with additional anaerobic digestion in a bench-scale unit



- Option 3: Demonstrate vector attraction reduction with additional aerobic digestion in a bench-scale unit
- Option 4: Meet a specific oxygen uptake rate for aerobically treated biosolids
- Option 5: Use aerobic processes at greater than 40° C (average temperature 45° C) for 14 days or longer (e.g., during biosolids composting)
- Option 6: Add alkaline materials to raise the pH under specified conditions
- Option 7: Reduce moisture content of biosolids that do not contain unstabilized solids from other than primary treatment to at least 75% solids
- Option 8: Reduce moisture content of biosolids with unstabilized solids to at least 90%

Of the above, option 7 is the only potentially applicable requirement to the struvite recovery process by MagPrex. Yet, due to loss of crystal waters, determining total solids by the standard method will result in inaccurately high moisture content results which impacts the verification of Vector Attraction Reduction properties mandated by Part 503. Furthermore, the crystal water constituting 44.08% of the struvite mineral represents water with no significant water activity. Struvite is a primarily inorganic precipitate whose mineral nature is itself inhibitory to vector attraction and/or microbial growth. Therefore, should the Part 503 vector attraction reduction requirements be applied to struvite as currently written, there is a need for the acceptance of a new standard method for materials like struvite which cannot accurately be tested for solids by using the standard method of drying.

#### Discussion

The standards for quality control, beneficial re-use of biosolids, and methodology for monitoring quality of products governed by Part 503 are legislated for applicability to sewage sludge specifically. Sewage sludge is defined by Part 503 in the following way:

Sewage sludge is solid, semi-solid, or liquid residue generated during the treatment of domestic sewage in a treatment works. Sewage sludge includes, but is not limited to, domestic septage; scum or solids removed in primary, secondary, or advanced wastewater treatment processes; and a material derived from sewage sludge. Sewage sludge does not include ash generated during the firing of sewage sludge in a sewage sludge incinerator or grit and screenings generated during preliminary treatment of domestic sewage in a treatment works. (Clean Water Act, 1999)

Under this definition, EPA considers products extracted from sewage sludge that are land applied, land disposed, or incinerated, to be within the scope of Part 503. EPA does consider products extracted from sewage sludge not being land applied, land disposed, or incinerated but rather sold into a commodity market to be outside of the scope of Part 503, as explained in a 2017 letter from EPA to the National Association of Clean Water Agencies (NACWA) (Gilinsky, 2017).

Further discussed in that 2017 letter is the following excerpt:

EPA recognizes that some products originating from sewage sludge could conceivably be so heavily refined or processed that a significant transformation or change in quality has occurred to the extent that it would be unreasonable to describe those products as "material derived from sewage sludge"



[and] because such products would not meet the definition of "sewage sludge," they would be outside the scope of Part 503. (Gilinsky, 2017)

Without further specification on what EPA considers a 'significant transformation or change in quality' for materials originating from sewage sludge, CNP offers its stance that struvite recovered from the MagPrex process is indeed formed and treated to be significantly transformed from the digest fed to the MagPrex process. The influent to the MagPrex process would be considered post-digested sewage sludge and provides constituent orthophosphate, ammonia, and some magnesium that, along with augmented magnesium chloride forms the struvite crystals in the MagPrex reactor. Yet, the result of the struvite precipitation reaction is a primarily inorganic crystalline material much different in chemical structure and biological behavior from the high-organics digest fed to the system.

This transformation can be further discussed in considering how biosolids are classified. Though not defined explicitly in Part 503, biosolids are defined in EPA's Plain English Guide to Part 503 as "primarily organic solid product(s) produced by wastewater treatment processes that can be beneficially recycled" (EPA, 1994). Notable in this definition is the phrase "primarily organic".

The average purity of MagPrex-recovered struvite being 87.4% implies that the combined amount of inorganic impurities and any organic matter constitute about 12% the total mass of the material, making the product harvested by MagPrex a primarily inorganic crystalline compound, distinguishing it from biosolids products on the basis of organic content.

The following conclusions can be drawn from this discussion of what EPA has published on the applicability of Part 503 to minerals like struvite:

- a) Struvite generated by the MagPrex process and sold into a commodity market can be considered outside of the scope of Part 503;
- b) Struvite is significantly transformed from sewage sludge so that the change in quality and primarily inorganic nature allows it to fall outside of the definitions of sewage sludge and biosolids, thus allowing struvite to fall outside of the scope of Part 503.

# **CNP** Approach

CNP agrees with and promotes Part 503's intention of diligently monitoring the quality of materials recovered from WRRFs to ensure the safety and well-being of end users, crops and household plants receiving an application of struvite fertilizer, and immediate and surrounding environments where the struvite may be landapplied. However, the standard methods used in Part 503 do not allow for accurate testing of struvite and therefore limit the rule's applicability to these types of precipitated minerals.

CNP partner company, NRU, is willing to offer assistance in the development of new standard methods for minerals like struvite to be analyzed for solids content. NRU scientists have extensive knowledge of mineral crystallography and laboratory methods for total solids and are confident that such a method could be developed, potentially in collaboration with EPA scientists if enough interest exists in the future. Until this progress is made, the use of standard methods for applying the standards of Part 503, developed without



consideration of their applicability to struvite, to the regulation of minerals like struvite recovered from WWRFs cannot be appropriate.

If struvite produced at a WRRF is intended for land application, struvite must be tested regularly on a schedule based on tonnage produced to meet the pollutant concentration limits for heavy metals provided in Part 503, to exhibit elimination of pathogenic potential, and to ensure free moisture contents of less than 10%. These standards must be proven using a new standardized laboratory methodology. CNP encourages WRRFs implementing an MagPrex installation to submit a quality control plan to local or state regulating agents to exhibit how quality standards will be measured and met, and it is advisable to request an initiation of a site-specific process for Part 503 exemption with an alternative standard that shows due diligence in the pursuit of adequate product quality and public safety.

Upon exhibited proof that a WRRF is producing a material which meets quality control standards - by appropriate methodology recognized by CNP and NRU personnel - CNP and/or NRU would be interested in a material acquisition scenario. In such a situation, this interest would be extended regardless of a commitment by the WRRF to comply with Part 503, but with evidence that the material meets safety characteristics similar to those embodied by Part 503. At that point, CNP or NRU may set further standards for quality assurance beyond those established for Class A EQ products by Part 503. For example, further quality assurance may include a minimum nitrogen or phosphorus concentration, a limit for organics (or carbon) content in the struvite material, acceptable particle size distribution, or other fertilizer quality measures which are outside of the scope of Part 503.

It is the intention and hope of CNP and NRU that nutrient recovery technologies like MagPrex can successfully offer nutrients reclaimed from WRRFs as valuable products in the fertilizer industry and elsewhere. To meet this goal, it is imperative for the industry to hold such reclaimed nutrients to a high standard of quality control by methods which ensure accurate analysis that can reliably demonstrate compliance with such a standard. With intentional coordination between scientists, laboratories, regulators, clients, and other key industry players, CNP and NRU are confident that a new set of standard methods can be implemented and enforced to ensure the intent of safe land application of the Part 503 rule is maintained for precipitated minerals like struvite or a new standard can be drafted and approved for these materials if the final consensus is that such materials are outside of the sewage sludge regulation contained in Part 503.



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# Appendix A: EPA Letter to NACWA



# UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON D.C., 20460

Office of Water

JAN 1 2 2017

Mr. Adam Krantz, CEO National Association of Clean Water Agencies 1816 Jefferson Place N.W. Washington D.C. 20036

We have had discussions with the National Association of Clean Water Agencies (NACWA) and some of their members regarding the applicability of the 40 C.F.R. 503 Standards for the Use or Disposal of Sewage Sludge to various scenarios involving the recovery of useful resources from wastewater.

As a general matter, EPA considers products extracted from sewage sludge that are not land applied, land disposed, or incinerated, but instead sold into a commodity market, outside the scope of Part 503. For example, a company could potentially extract precious metals or rare earth elements from wastewater. Such products would not be subject to Part 503 if they were resold as commodities instead of "applied to the land, placed on a surface disposal site, or fired in a sewage sludge incinerator." This is consistent with EPA's existing guidance on Part 503, which makes clear that Part 503 "establishes requirements for the final use or disposal of sewage sludge [biosolids] when biosolids are: applied to land . . . placed on a surface disposal site . . . or fired in a biosolids incinerator." See A Plain English Guide to the EPA Part 503 Biosolids Rule, at p. 6 (September 1994).

The situation becomes more complicated when the product is intended to be land applied, surface disposed, or incinerated as contemplated by Part 503. Part 503 regulates "sewage sludge" and it defines the term to include "material derived from sewage sludge." But EPA recognizes that some products originating from sewage sludge could conceivably be so heavily refined or processed that a significant transformation or change in quality has occurred to the extent that it would be unreasonable to describe those products as "material derived from sewage sludge." Because such products would not meet the definition of "sewage sludge," they would be outside the scope of Part 503. EPA cannot, at this time, offer any general statements about what types of products may not be "derived from sewage sludge." But EPA is willing to consider on an individual case-by-case basis whether a particular product recovered from sewage sludge is beyond the scope of Part 503.

If NACWA or its member facilities wish to inquire of EPA its views about whether a particular product is "derived from sewage sludge," EPA would consider all of the following information to be helpful:



- In order for EPA to determine whether the product will be protective of public health
  and the environment, evidence demonstrating that the product meets the pollutant
  concentrations for high quality sewage sludge in Table 3 of 40 CFR 503.13, the more
  stringent Class A pathogen requirements, and one of the first eight vector attraction
  requirements. These standards are known collectively as the exceptional quality or
  Class A EQ requirements;
- Data and information indicating that the process used to generate the product will
  continue to ensure that the product meets the Class A EQ requirements over time. For
  example, the requestor may include a proposed schedule for the voluntary submission
  to EPA of data and evidence concerning the composition of the product. Or the
  requestor may include other relevant information about the requestor's quality
  assurance and quality control procedures; and
- A reasonable rationale for believing that the product is outside the scope of what EPA intended to regulate under Part 503 when it adopted the regulations. For example, are there indications in the legislative history of the Clean Water Act, the administrative record of the Part 503 regulations, or prior EPA guidance and statements that the regulations were not intended to apply to the product? Or do the chemical purity, commercial applications, and/or the existence of other applicable state, federal, or international regulations suggest that regulation under Part 503 would be redundant?

Please note that this response is not a rule or final agency action; nor does it establish any binding legal obligations on EPA or the regulated community. EPA may adjust its views as changing circumstances warrant or as otherwise appropriate. Products not regulated under Part 503, of course, remain subject to any other applicable federal regulations.

If you have additional questions on this matter, or wish to make an inquiry of and submit information for a specific product, please contact Betsy Southerland by email (Southerland.Elizabeth@epa.gov) or by phone (202-566-0328).

Regards,

Ellen Gilinsky, Ph.D.

Associate Deputy Assistant Administrator

Ellen Dlinsky