

Florida Department of Environmental Protection

Bob Martinez Center
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

Charlie Crist
Governor

Jeff Kottkamp
Lt. Governor

Michael W. Sole
Secretary - Designee

January 8, 2007

Brian Timmins
Etec, LLC
6635 Northeast 59th Place
Portland, Oregon 97218

Re: **Dissolved Oxygen In Situ Treatment (DO-IT) System**

Dear Mr. Timmins:

The Bureau of Petroleum Storage Systems hereby reaffirms and updates its acceptance of the Dissolved Oxygen In Situ Treatment (DO-IT) System as an innovative technology for both in situ and ex situ bioremediation of petroleum and other suitable contaminants in groundwater and soil. This update supersedes earlier versions dated April 28, 1999 and May 5, 2005, and covers a change of company name from Enzyme Technologies Incorporated to Etec, LLC, and documents your indication that Custom Blend Nutrients (EZT-CBN) is also known as NutriMax.

As Etec has indicated, the DO-IT system is a mechanism for the delivery of oxygen and bioenhancement products to the subsurface for in situ remediation, or to a reactor or soil pile for ex situ applications. The system generates dissolved oxygen levels as high as 40 parts per million, and also uses the following six (6) proprietary bioenhancement products:

- o **Enzyme Accelerator (EZT-EA)**, a biosurfactant;
- o **PetroSolv (EZT-PS)**, a biosurfactant;
- o **Biostimulator (EZT-BST)**, an enzyme/biosurfactant;
- o **Multi-Enzyme Complexes (EZT-MZC)**, an enzyme formulation;
- o **Petroleum Hydrocarbon Bacterial Consortium (EZT-A2)**, a mixture of naturally-occurring, non-pathogenic microorganisms; and
- o **Custom Blend Nutrients (EZT-CBN)** also known as **NutriMax**.

This acceptance applies only to the use of these six products, for which Etec has submitted the results of proprietary chemical analyses, and for which the Bureau of Petroleum Storage Systems issues a proprietary analysis voucher as enclosure 1. For any additional products that are used, analyses are also required if they will be injected into the groundwater or may leach into it.

Additionally, this acceptance applies only to the jurisdiction of this Bureau, which is the cleanup of petroleum pursuant to Chapter 62-770, Florida Administrative Code (F.A.C.). Other bureaus or other government agencies may choose to recognize this acceptance if their needs and requirements are similar, but this Bureau is not responsible for applications beyond its jurisdiction.

The Bureau recognizes the DO-IT system as a viable method for the bioremediation of petroleum contaminated sites in Florida. There are no objections to its use provided: (a) the considerations of this letter

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are taken into account; (b) a site-specific Remedial Action Plan is approved by the Department; and (c) the requirements of Rule 62-522.300(2)(c), F.A.C., are observed.

While the Department of Environmental Protection does not provide endorsement of specific or brand name remediation products or processes, it does recognize the need to determine their acceptability from an environmental standpoint with respect to applicable rules and regulations, and the interests of public health and safety. Vendors must then market the products and processes on their own merits regarding performance, cost, and safety in comparison to competing alternatives in the marketplace. In no way, however, shall this regulatory acceptance letter be construed as certification of product performance. Additionally, the Department emphasizes a distinction between regulatory "acceptance" and approval. Products and processes are accepted, not approved. For the DO-IT system, the major regulatory considerations are set forth in enclosure 2.

Those who prepare Remedial Action Plans may include a copy of this letter in the appendix of plans they submit, and call attention to it in the text of their document. In this way, technical reviewers throughout the state will be informed that you have contacted the Department of Environmental Protection to inquire about DO-IT's acceptability. To aid those reviewers, the Bureau of Petroleum Storage Systems provides supplemental information as enclosure 3.

Even though it may be convenient to have an acceptance letter for inclusion in the appendix of a plan, as suggested above, the Bureau would like to emphasize that it is not a requirement for a particular remediation product or process to have an official acceptance letter in order to be proposed in a site-specific Remedial Action Plan. The plan, however, must contain sufficient information about the product or process to show that it meets all applicable and appropriate rules and regulations, especially those of the Florida Administrative Code pertaining to groundwater and underground injection control.

The Department reserves the right to revoke its acceptance of a product if it has been falsely represented. Additionally, Department acceptance of any product or process does not imply it has been deemed applicable for all cleanup situations, or that it is preferred over other treatment or cleanup techniques in any particular case. A site specific evaluation of applicability and cost-effectiveness must be considered for any product or process, whether conventional or innovative, and adequate site-specific design details must be provided in Remedial Action Plans proposing the product or process. You may contact me at (850) 877-1133, extension 3722, if there are any questions.

Sincerely,

Rick Ruscito
Rick Ruscito, P.E.
Ecology and Environment, Inc.
Bureau of Petroleum Storage Systems
Petroleum Cleanup Section 6

Rebecca S. Lockenbach
Rebecca S. Lockenbach
FDEP Section Leader
Bureau of Petroleum Storage Systems
Petroleum Cleanup Section 6

Brian Timmins
January 8, 2007
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enc: (1) Proprietary Chemical Analysis Voucher
(2) Regulatory Information
(3) Supplemental Information
(4) Underground Injection Control Memorandum

c: T. Conrardy - FDEP/Tallahassee

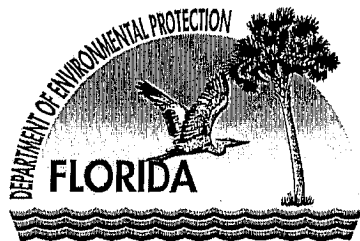
History

Inn_027.doc - 4/28/99

Inn_027a.doc - 5/5/05

Inn_027b.doc - 1/8/07

DO-IT SYSTEM PROPRIETARY CHEMICAL ANALYSIS VOUCHER



Florida Department of Environmental Protection

Bob Martinez Center
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

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January 8, 2007

Brian Timmins
Etec, LLC
6635 Northeast 59th Place
Portland, Oregon 97218

Re: **Proprietary Chemical Analysis Voucher**

Dear Mr. Timmins:

The Bureau of Petroleum Storage Systems hereby acknowledges receipt of a confidential disclosure dated March 8, 1999, by Enzyme Technologies Incorporated (now known as Etec, LLC as of September 8, 2006) regarding the proprietary analyses of the following products associated with its Dissolved Oxygen In Situ Treatment (DO-IT) system:

- o **Enzyme Accelerator (EZT-EA)**, a biosurfactant;
- o **PetroSolv (EZT-PS)**, a biosurfactant;
- o **Biostimulator (EZT-BST)**, an enzyme/biosurfactant;
- o **Multi-Enzyme Complexes (EZT-MZC)**, an enzyme formulation;
- o **Petroleum Hydrocarbon Bacterial Consortium (EZT-A2)**, a mixture of naturally-occurring, non-pathogenic microorganisms; and
- o **Custom Blend Nutrients (EZT-CBN)** also known as **NutriMax**.

Having reviewed the confidential disclosure, we hereby vouch for the analyses, and indicate that pH is the only measured parameter which does not meet the drinking water standards of Chapter 62-550, Florida Administrative Code (F.A.C.), which are referenced by Chapter 62-528, F.A.C., as an underground injection control requirement. None of the above products has a pH that falls within the acceptable range of 6.5 to 8.5 for injection into an aquifer.

In order to use these products for in situ aquifer remediation, a temporary injection zone of discharge must be permitted for the pH. Effective August 27, 2001, a temporary injection zone of discharge can be obtained by Rule 62-522.300(2)(c), F.A.C., for a secondary groundwater parameter such as pH, rather than by way of a variance. Each site-specific Remedial Action Plan that proposes the use of the above products must include information that is required by this rule. Enforceable approval orders for Remedial Action Plans containing this information, when issued by the Department, will not only constitute approval of the site-specific plan, but will also constitute permission for a temporary injection zone of discharge.

For underground injection control purposes, remediation plans proposing the DO-IT system products must disclose the volume and composition of the fluid to be injected into an aquifer. Since the composition is proprietary, it will suffice to indicate the overall concentration of the solution and then provide a footnote indicating that a one-time confidential disclosure regarding the proprietary analyses has been

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DO-IT Voucher

submitted to the Department and accepted. Reference should be made to the original March 8, 1999 disclosure, and a copy of this voucher should be included as an appendix in the plan.

Remediation plan reviewers for petroleum contaminated site applications involving the DO-IT system and its associated products may contact Rick Ruscito at (850) 877-1133, extension 3722.

Sincerely,

Rick Ruscito

Rick Ruscito, P.E.
Ecology and Environment, Inc.
Bureau of Petroleum Storage Systems
Petroleum Cleanup Section 6

Rebecca S. Lockenbach

Rebecca S. Lockenbach
FDEP Section Leader
Bureau of Petroleum Storage Systems
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REGULATORY INFORMATION

- a. Groundwater cleanup standards: The onus shall be on Etec and users of the DO-IT system to observe all applicable regulations, particularly those regarding underground injection control and groundwater quality. All applicable groundwater contaminant standards shall be met at the time of project completion, for the contaminants of concern, any residuals associated with the ingredients of DO-IT system products, and any byproducts produced as a result of chemical or biochemical reactions involving those ingredients. The following chapters of the Florida Administrative Code are cited: Chapter 62-550, F.A.C., for primary and secondary water quality standards; Chapter 62-520, F.A.C. for groundwater classes and standards; Chapter 62-522, F.A.C., for groundwater permitting and monitoring requirements; Chapter 62-528, F.A.C., for underground injection control, particularly Part V, for Class V, Group 4 aquifer remediation projects; and Chapter 62-770, F.A.C., for petroleum cleanup criteria.

A noteworthy aspect of the minimum criteria set forth in Chapter 62-520, F.A.C., is that it requires groundwater to be free from substances which are harmful to plants, animals, and organisms, and free from substances that are carcinogenic, mutagenic, teratogenic or toxic to human beings. In effect, these "free from" requirements form a catchall. They close what would otherwise be a loophole in the regulations by preventing injection of a potentially harmful product in the event that any of its ingredients is not regulated as a specific primary or secondary drinking water contaminant.

- b. Injection well permit: The issuance of a site specific Remedial Action Plan Approval Order, by either the Bureau of Petroleum Storage Systems or the Bureau of Waste Cleanup, for injection-type aquifer remediation constitutes the granting of a Class V injection well permit.
- c. Groundwater injection standards: For in situ aquifer remediation, the composition of an injected fluid must meet the drinking water standards set forth in Chapter 62-550, F.A.C., pursuant to underground injection control Rule 62-528.600(2)(d), F.A.C. The proprietary chemical analysis of enclosure 1 indicates that the six products associated with the DO-IT system meet this requirement, with the exception of pH. A temporary injection zone of discharge must be permitted for the pH before DO-IT products can be used for in situ aquifer remediation.
- d. Zone of discharge by rule: In order for the DO-IT system products to be used for in situ aquifer remediation, a temporary injection zone of discharge must be permitted for the pH. Effective August 27, 2001, a temporary injection zone of discharge can be obtained by Rule 62-522.300(2)(c), F.A.C., for a secondary groundwater parameter such as pH, rather than by way of a variance. Each site-specific Remedial Action Plan that proposes the use of the above products must include information that is required by this rule. The required information is as follow: (a) an indication that the DO-IT products to be used do not meet the secondary groundwater standard for pH, which is range 6.5 to 8.5; (b) the size of the temporary injection zone of discharge (usually the radius of influence for each injection point); (c) the amount of time that a temporary zone of discharge will be needed; and (d) plans for the monitoring of groundwater pH. Enforceable

approval orders for Remedial Action Plans containing this information, when issued by the Department, will not only constitute approval of the site-specific plan but will also constitute the permission for a temporary injection zone of discharge.

- e. Utilization of wells: If a remediation site happens to have an abundance of monitoring wells, then the Department has no objection to the use of some wells for the application of DO-IT system products. However, no "designated" monitoring well, dedicated to the tracking of remediation progress (by sampling) shall be used to apply DO-IT system products. This will avoid premature conclusions that the entire site meets cleanup goals. By making sure that designated tracking wells are not also used for treatment, there will be more assurance that the treatment process has permeated the entire site and that it did not remain localized to the area immediately surrounding each injection well.
- f. Groundwater monitoring:
 - 1. Active remediation petroleum monitoring: During the period of active remediation, groundwater shall be monitored for contaminants of concern in accordance with the requirements set forth in Section 62-770.700, F.A.C. Two noteworthy rules within that section are 62-770.700(3)(i), F.A.C., for frequency of sampling, and 62-770.700(5)(f), F.A.C., which requires a sampling schedule for bioremediation. For non-petroleum cleanups, the monitoring should be conducted in accordance with the provisions of an approved Remedial Action Plan.
 - 2. Post remediation petroleum monitoring: At least one (1) year of quarterly post remediation groundwater monitoring for contaminants of concern shall be conducted at a minimum of two (2) wells, one located in the area of maximum petroleum contamination, the other downgradient of the area of maximum petroleum contamination, pursuant to Section 62-770.750, F.A.C. For non-petroleum cleanups, the monitoring should be conducted in accordance with the provisions of an approved Remedial Action Plan.
 - 3. Underground injection control monitoring: As indicated in paragraph *d* above, in order to comply with Rule 62-522.300(2)(c), F.A.C., site-specific Remedial Action Plans proposing the use of DO-IT system products must include a groundwater monitoring plan for pH.
- g. Underground injection control inventory: Remedial Action Plans prescribing in situ aquifer injection-type remediation shall include information pursuant to Rule 62-528.630(2)(c)1 through 6, F.A.C., for the inventory purposes of underground injection control. Per Rule 62-528.630(2)(c), F.A.C., aquifer remediation projects involving injection wells may be authorized under the provisions of a Remedial Action Plan, provided the construction, operation, and monitoring requirements of Chapter 62-528, F.A.C., are met. A memorandum outlining the inventory information about injection-type aquifer remediation plans to be transmitted by Department reviewers to the Underground Injection Control Section is provided as enclosure 4.

h. Operation:

1. Avoidance of migration: For in situ injection-type aquifer remediation projects, injection of DO-IT products shall be performed in such a way, and at such a rate and volume, that no undesirable migration of either the product's ingredients or the contaminants of concern in the aquifer results, pursuant to Rule 62-528.630(3), F.A.C.
 2. Underground injection control operating permit: Although an operating permit is not required for aquifer remediation wells pursuant to Rule 62-528.640(1)(b), and 62-528.640(1)(c), F.A.C., since no movement of the petroleum contamination plume is expected to accompany the DO-IT treatment process, the Department requests that the information items listed in Rule 62-528.640(1)(b), F.A.C., be considered and included in Remedial Action Plan proposals as a matter of good and thorough design practice. Briefly summarized, they are: quality of water in the aquifer; quality of the injected fluid; existing and potential uses of the affected aquifer; and well construction details. Additionally, each Remedial Action Plan should clearly indicate the total volume of fluid and the concentration of products that will be injected.
 3. Operating parameter measurements: Rule 62-770.700(9)(h), F.A.C., sets forth frequency requirements for the measurement of bioremediation operating parameters such as dissolved oxygen levels, rates of nutrient addition, temperature, etc. It also includes an option for reduction in the frequency or discontinuation of some measurements in situations when appropriate.
- i. Abandonment of wells: Upon issuance of a petroleum Site Rehabilitation Completion Order, or a declaration of "No Further Action", injection wells shall be abandoned pursuant to Section 62-528.645, F.A.C. The Underground Injection Control Section of the Department shall be notified so that the injection wells can be removed from the inventory-tracking list.

SUPPLEMENTAL INFORMATION

- a. Distinguishing features: Etec, developer of both the DO-IT system and its associated products, indicates that diffusion of contaminants through the cell walls of bacteria is a rate-limiting step in the bioremediation process, especially when large hydrocarbon molecules such as those found in diesel, crude oil, and polynuclear aromatic hydrocarbons (PAH) are involved. It is only after these hydrocarbons pass through the cell walls that they are broken down by enzymes produced by the bacteria. Etec manufactures these same enzymes in a concentrated form. When they are applied to contaminated soil or groundwater they immediately begin to break down the hydrocarbons, thereby hastening the start of the contaminant degradation process. Additionally, to ensure that sufficient oxygen is available for biodegradation to occur in situ, Etec uses an aboveground air separator to produce pure oxygen, to saturate its injection fluids with as much as 40 parts per million oxygen.
- b. Department of Environmental Protection reviewers of in situ injection-type aquifer remediation plans, regardless of whether in Tallahassee or district offices, must fill in the blanks on the enclosure 4 memorandum, whose subject is "Proposed Injection Well(s) for In Situ Aquifer Remediation at a Petroleum Remedial Action Site". The completed form must be submitted to the Underground Injection Control Section at 2600 Blair Stone Road, Tallahassee, Florida 32399-2400.

Only reviewers within the Department and its district offices may approve in situ injection-type remediation plans in which the approval constitutes the issuance of a Class V injection permit; local programs are not authorized to grant such approvals. The reason: although an arrangement between the US Environmental Protection Agency and the Department delegates underground injection control authority to the Department, it does not allow the Department to delegate that authority any further. This includes delegation to the Department's contracted remediation review agencies such as those operated by the counties and other local governments. These contracted counties and local governments, however, upon completion of an injection-type, in situ aquifer Remedial Action Plan review, can arrange for the Department headquarters in Tallahassee to issue an enforceable order.

- c. Pilot study: For bioremediation, per Rule 62-770.700(2), F.A.C., a pilot study proposal shall be submitted for review, and a pilot test shall be performed prior to designing a treatment system. If conditions or the situation at a site do not warrant a pilot study, then a proposal explaining the rationale for the decision not to perform a pilot study shall be submitted for review.
- d. Bacteria: It is generally reported (on a total weight basis) that bacteria are approximately 70 to 80 percent water. On a dry weight basis, approximately 95 percent of the composition is represented by 5 elements: carbon, oxygen, nitrogen, hydrogen, and phosphorus. At a petroleum remediation site, it is intended that the source of carbon for the growth of bacteria will come from the petroleum hydrocarbons themselves. Naturally-occurring organic carbon at a site can also serve as a carbon source for bacteria. Depending on site's specific conditions, the remaining four elements must either be available naturally, or added as macronutrients in order to

stimulate bioremediation. Micronutrients must also be present for bacteria to grow.

Enzymes Technologies Incorporated has indicated that its Petroleum Hydrocarbon Bacterial Consortium product EZT-A2 contains only microorganisms that are naturally-occurring (not genetically engineered) and non-pathogenic (do not cause disease).

- e. Degradation products: Carbon dioxide and water are the ultimate products of aerobic and most anaerobic biodegradations of hydrocarbons. In the case of methanogenesis, an anaerobic process, carbon dioxide and methane are produced. The intermediate products of aerobic degradation may include simple acids, alcohols, and fatty acids. Aerobic processes use oxygen as an electron acceptor to produce carbon dioxide and water.
- f. Parameters: The following parameters may be useful in determining the potential for bioremediation at a site, or whether bioremediation is already occurring. They were selected from a list that appears in the publication "In Situ Treatment Technology" by E. Nyer et al., Lewis Publishers, 1996. The parameters are: dissolved oxygen; redox potential; pH; temperature; specific conductance; volatile organic compounds; nitrate; nitrite; ammonia nitrogen; manganese (total and dissolved); iron (total, dissolved, and ferrous); sulfate; sulfide; and total organic carbon. Gaseous parameters include: carbon dioxide, oxygen, nitrogen, and methane. Other parameters that may be helpful are chemical oxygen demand, biochemical oxygen demand, and total organic carbon. Those who prepare bioremediation plans, and their reviewers should determine which parameters, if any, should be investigated on a site specific basis. Etec has indicated that DO-IT system applications may also include measurement of phosphorus and occasional bacteria plate counts.
- g. Dosage: Etec has indicated that the exact dosage of its products will vary from site to site, depending on the volume of contaminated media and the mass of contaminants present. Therefore, no rule-of-thumb guidance is offered by the Bureau of Petroleum Storage Systems on this subject in the case of these products. Each site specific Remedial Action Plan, however, must indicate the amount of products to be used in order to comply with underground injection control requirements.
- h. Limitations: Direct application of the products for in situ treatment of free product is not recommended, although they may be used to augment free product recovery efforts and to treat water which has become contaminated as a result of contact with free product. Also, the DO-IT system may not be suitable for in situ treatment of low permeability clay. If there are any doubts about the suitability of the process for a particular site, then a measurement of the permeability or a pilot test may be helpful in making a decision. Etec has also indicated that permeability is not a factor in the ex situ treatment of clay since excavation increases the porosity.

**Florida Department of
Environmental Protection**

Memorandum

TO: Richard Deuerling, Mail Station 3530
Division of Water Resource Management
Underground Injection Control Section
Florida Department of Environmental Protection
2600 Blair Stone Road, Tallahassee, FL 32399-2400

FROM: _____ [see Note 1.]

DATE: _____

SUBJ: **Proposed Injection Well(s) for In Situ Aquifer
Remediation at a Petroleum Remedial Action Site**

Pursuant to paragraph 62-528.630(2)(c), F.A.C., inventory information is hereby provided regarding the proposed construction of temporary injection well(s) for the purpose of in situ aquifer remediation at a petroleum-contaminated site.

Site name: _____
Site address: _____
City/County: _____
Latitude/Longitude: _____
FDEP Facility Number: _____

Site owner's name: _____
Site owner's address: _____

Well contractor's name: _____ [see Note 2.]
Well contractor's address: _____

Brief description of the in situ injection-type aquifer remediation project:

Summary of major design considerations and features of the project:

Areal extent of contamination (square feet): _____
Number of injection wells: ____

Composition of injected fluid [see Note 3.]

(ingredient, wt. %): _____

Injection volume per well (gallons): _____

Single or multiple injection events: _____

Injection volume total (all wells, all

events): _____

A site map showing the areal extent of the groundwater contamination plume, and the location and spacing of injection wells and associated monitoring wells is attached.

The following is a summary description of the affected aquifer:

Name of aquifer: _____

Depth to groundwater (feet): _____

Aquifer thickness (feet): _____

The injection well(s) features are summarized below, and/or a schematic of the injection well(s) is attached.

Direct-push or HSA/Mud rotary (*circle the appropriate well type*)

Diameter of well(s) (i.e., riser pipe & screen) (inches): _____

Total depth of well(s) (feet): _____

Screened interval: _____ to _____ feet below land surface

Grouted interval: _____ to _____ feet below land surface

Casing diameter, if applicable (inches): _____

Cased depth, if applicable: _____ to _____ feet below land surface

Casing material, if applicable: _____

The in situ injection-type aquifer remediation plan for this petroleum contaminated site is intended to meet the groundwater petroleum cleanup criteria set forth in Chapter 62-770, F.A.C. Additionally, all other groundwater standards will be met at the time of project completion for any residuals associated with the ingredients of the injected remediation products, and any by-products or intermediates produced as a result of the chemical or biochemical transformation of those ingredients or the contaminating petroleum during their use. Applicable primary and secondary drinking water standards are set forth in Chapter 62-550, F.A.C., and additional groundwater quality criteria are set forth in Chapters 62-520 and 62-777, F.A.C.

The remediation plan estimates that site remediation will take _____ months. We will notify you if there are any modifications to the remediation strategy which will affect the injection well design or the chemical composition and volume of the injected remediation product(s).

The proposed remediation plan was approved on _____ by an enforceable approval order. A copy is attached. The remediation system installation is expected to commence within 60 days. Please call me at _____ if you require additional information.

Note 1. Local programs are not authorized to approve underground injections into aquifers. Reason: Per agreement with the USEPA, the FDEP cannot delegate this authority. Local programs, after reviewing a Remedial Action Plan or an injection proposal document, should prepare the Approval Order and route it to Tallahassee for Bureau Chief's signature, and then complete this form. This form is primarily for use by state and local program technical reviewers, but petroleum remediation contractors may fill in all blanks except those labeled "FROM" and "DATE" on page 1, and the approval date and telephone number blanks in the last paragraph. Those blanks should be completed only by a state or local program reviewer.

Note 2. If an injection well installation contractor has not yet been selected, then indicate the name and address of the project's general remediation contractor/consultant.

Note 3. Complete chemical analysis of injected fluid is required by Chapter 62-528, F.A.C. Proprietary formulations shall make confidential disclosure. Injected fluids must meet drinking water standards of Chapter 62-550, F.A.C., unless an exemption or variance has been granted.