JOINT TRANSPORTATION RESEARCH PROGRAM

INDIANA DEPARTMENT OF TRANSPORTATION AND PURDUE UNIVERSITY



PROJECT IMPLEMENTATION: CLASSIFICATION OF ORGANIC SOILS AND CLASSIFCATION OF MARLS—TRAINING OF INDOT PERSONNEL

Alain El Howayek

Graduate Research Assistant School of Civil Engineering, Purdue University

Antonio Bobet

Professor of Civil Engineering School of Civil Engineering, Purdue University

Sulaiman Dawood

Graduate Research Assistant School of Civil Engineering, Purdue University

Andrew Ferdon

Undergraduate Research Assistant School of Civil Engineering, Purdue University

Marika Santagata

Associate Professor of Civil Engineering School of Civil Engineering, Purdue University Corresponding Author

Nayyar Zia Siddiki

Supervisor, Geotechnical Operations
Office of Geotechnical Engineering, Indiana Department of Transportation

SPR-3517 Report Number: FHWA/IN/JTRP-2012/22 DOI: 10.5703/1288284314984

RECOMMENDED CITATION

El Howayek, A., A. Bobet, S. Dawood, A. Ferdon, M. Santagata, and N. Z. Siddiki. *Project Implementation: Classification of Organic Soils and Classification of Marls—Training of INDOT Personnel*. Publication FHWA/IN/JTRP-2012/22. Joint Transportation Research Program, Indiana Department of Transportation and Purdue University, West Lafayette, Indiana, 2012. doi: 10.5703/1288284314984.

CORRESPONDING AUTHORS

Associate Professor Marika Santagata School of Civil Engineering Purdue University (765) 494-0697 mks@purdue.edu

JOINT TRANSPORTATION RESEARCH PROGRAM

The Joint Transportation Research Program serves as a vehicle for INDOT collaboration with higher education institutions and industry in Indiana to facilitate innovation that results in continuous improvement in the planning, design, construction, operation, management and economic efficiency of the Indiana transportation infrastructure. https://engineering.purdue.edu/JTRP/index_html

Published reports of the Joint Transportation Research Program are available at: http://docs.lib.purdue.edu/jtrp/

NOTICE

The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views and policies of the Indiana Department of Transportation or the Federal Highway Administration. The report does not constitute a standard, specification or regulation.

TECHNICAL REPORT STANDARD TITLE PAGE

1. Report No. FHWA/IN/JTRP-2012/22	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle Project Implementation: Classification of Organic Soils and Classification of Marls— Training of INDOT Personnel		5. Report Date September 2012 6. Performing Organization Code	
7. Author(s) Alain El Howayek , Antonio Bobet , Sulaiman Dawood , Andrew Ferdon , Marika Santagata , Nayyar Siddiki		8. Performing Organization Report No. FHWA/IN/JTRP-2012/22	
9. Performing Organization Name and Address Joint Transportation Research Program Purdue University 550 Stadium Mall Drive West Lafayette, IN 47907-2051		10. Work Unit No.	
		11. Contract or Grant No. SPR-3517	
12. Sponsoring Agency Name and Address Indiana Department of Transportation State Office Building 100 North Senate Avenue Indianapolis, IN 46204		13. Type of Report and Period Covered Final Report 14. Sponsoring Agency Code	

15. Supplementary Notes

Prepared in cooperation with the Indiana Department of Transportation and Federal Highway Administration.

16. Abstract

This is an implementation project for the research completed as part of the following projects: SPR-3005 - Classification of Organic Soils and SPR-3227 – Classification of Marl Soils. The methods developed for the classification of both soils have been incorporated in INDOT standard specification 903.05 and 903.06 respectively. Both projects included recommendations for implementation that reflected input from the project PA and SAC. A specific recommendation from both projects was that INDOT soil technicians be trained to perform the required tests and classify soils based on the revised classification systems. This project was initiated to carry out the implementation of those recommendations.

The project scope includes development of training material for instruction about the performance of the revised classification tests and methods, training to pertinent INDOT personnel, integration of the revised classification system into INDOT's standards, and establishment of a resource database for future training of INDOT personnel.

Within the general scope outlined above, the specific objectives of the proposed work were to: a) administer training to select INDOT personnel and interested representatives from the geotechnical consulting/construction community; b) develop training materials to be used by INDOT to train additional personnel. These two general objectives were accomplished through four specific tasks: 1) Collection of Sample Soils for Testing and Classification; 2) Development of Training Material (a PowerPoint presentation with concise instructional handouts; supporting classification examples from a variety of soils; and a short manual summarizing the classification system for both soils with supporting examples); 3) Delivery of Training Sessions for INDOT personnel, as well as representatives from select geotechnical consultants and contractors; 4) Production of Training Video.

17. Key Words		18. Distribution Statement	t	
organic soils, marls, identification, classification, training program		No restrictions. This document is available to the public through the National Technical Information Service, Springfield, VA 22161.		
19. Security Classif. (of this report)	20. Security Classif. (of this page)		21. No. of Pages	22. Price
Unclassified	Unclassified		126	

EXECUTIVE SUMMARY

PROJECT IMPLEMENTATION: CLASSIFICATION OF ORGANIC SOILS AND CLASSIFICATION OF MARLS—TRAINING OF INDOT PERSONNEL

Introduction

This is an implementation project for the research completed as part of two projects: SPR-3005, Classification of Organic Soils, and SPR-3227, Classification of Marl Soils. The methods developed for the classification of both soils have been incorporated in Indiana Department of Transportation (INDOT) standard specifications 903.05 and 903.06, respectively. Both projects included recommendations for implementation that reflected input from the project administrator and study advisory committee. A specific recommendation from both projects was that INDOT soil technicians be trained to perform the required tests and to classify soils based on the revised classification systems. This project was initiated to carry out the implementation of those recommendations.

The project scope includes developing training materials, training pertinent INDOT personnel, integrating the revised classification system into INDOT's standards, and establishing a resource database for future training of INDOT personnel.

Findings

- The presence of organics in soils can create problems in geotechnical practice by increasing the soil's compressibility and creep potential, decreasing its maximum dry density and strength, and potentially interfering with the soil's stabilization or modification with cement, lime, and cement or lime byproducts.
- Such problems are recognized in current INDOT specifications, which have strict limits on the percentage of organic matter allowed for certain applications. Thus, identification of organic soils and quantification of the percentage of organic matter is critical in many engineering projects. The method

- that was previously employed by INDOT to determine organic content tends to overestimate the percentage of organic matter. This is problematic because misclassification of organic soils can lead to significant costs that could be avoided
- Marls typically have low dry density, very high moisture content, and low shear strength. As a result, they are considered problem soils and their correct identification and classification is critical in geotechnical engineering practice.
- Because of the generally unsatisfactory geotechnical properties of marls, INDOT specifications restrict the amount of calcium and magnesium carbonate that can be present in soils for a number of applications, similarly to how they restrict the presence of organic matter. The methodologies that are available for determining the calcium carbonate content are either very complex (e.g., the chemical determination of CaCO₃), or not sufficiently sensitive (e.g., the effervescent action of hydrochloric acid on the carbonate). As with organic soils, misclassification of marl soils can be costly.
- As a result, classification systems were developed to classify organics soils (SPR-3005) and marls (SPR-3227) more accurately and in a relatively easy manner.
- This project: (1) administers training to INDOT personnel and interested representatives from the geotechnical consulting/construction community, and (2) develops training materials to be used by INDOT to train additional personnel.

Implementation

This project was implemented based on four specific tasks:

- 1. Collection of sample soils for testing and classification.
- Development of training material, namely: a PowerPoint
 presentation with concise instructional handouts; supporting
 classification examples for a variety of soils; and a manual
 summarizing the classification system for both soils with
 supporting examples.
- Delivery of training sessions to INDOT personnel, as well as representatives from select geotechnical consulting firms and contractors.
- 4. Production of a training video.

CONTENTS

1. INTRODUCTION: BACKGROUND AND SCOPE
2. PROJECT OBJECTIVES
3. PROJECT TASKS
4. ACKNOWLEDGMENTS
5. CONCLUSIONS
APPENDIX 1. PowerPoint Presentation
APPENDIX 2. Lab Manual
APPENDIX 3. Short Procedure for Identification and Classification of Organic Soils
APPENDIX 4. Short Procedure for Identification and Classification of Marly Soils
APPENDIX 5. Classification Charts
APPENDIX 6. Classification Checklists
APPENDIX 7. Supporting Classification Examples
APPENDIX 8. Training Dates
APPENDIX 9 Feedback Form

LIST OF TABLES

Table	Page

4

Table 3.1 Sample soils used as supporting classification examples

LIST OF FIGURES

Figure	Page
Figure 3.1 Sample source 1	2
Figure 3.2 Sample source 2	3
Figure 3.3 Sample source 3	4

1. INTRODUCTION: BACKGROUND AND SCOPE

This is an implementation project for the research completed as part of two projects: SPR-3005, Classification of Organic Soils, and SPR-3227, Classification of Marl Soils. The methods developed for the classification of both soils have been incorporated in INDOT standard specification 903.05 and 903.06 respectively.

SPR-3005 addressed the classification of organic soils and the quantification of organic matter in soils. The study was motivated by the realization that the methods previously employed by INDOT to quantify organic matter content and the strict guidelines on organic content used to determine the acceptability of a soil for a given application could lead to incorrect classification of soils. This, in turn, could lead to erroneously considering a material unviable for a given application, and to unnecessary costs for material replacement/treatment.

The research conducted as part of SPR-3005 involved two main work streams: a review of the literature and a focused experimental effort. The former reviewed existing classification systems for organic soils, the effects of organic matter on the geotechnical properties of soils, and the methods for determination of organic content. The experimental component of the research involved performing loss on ignition tests, Atterberg limits, colorimetric tests, dry combustion tests, thermal analyses, and X-ray diffraction analyses on natural soils with varying organic content, as well as on laboratory prepared ("artificial") organic soils.

The work led to the proposition of a revised system for classifying soils in four groups (peats; organic soils; mineral soils with organic matter; and mineral soils) based on the percentage of organic matter estimated from the loss on ignition (LOI) in combination with the liquid limit ratio and the results of the colorimetric test. These methods were validated with tests on a variety of soils. It was found that based on the LOI results, some soils that might be considered unviable for roadway construction, did not instead contain significant amounts of organics. These observations were supported by in-laboratory chemical measurements.

SPR-3227 addressed the classification of marl soilssoft, carbonate-rich, low-organic, light gray colored clay or silt deposits (fine-grained soils only) that are formed by precipitation of calcite below an organic soil deposit. Marly soils, which are also often characterized by the presence of organic matter (4-20%), are not generally well described with existing soil classification systems, and the methodologies available for their identification in the laboratory or in the field are either not adequate or not effective. To address this, the project involved testing of marl samples obtained from three INDOT road construction projects. The experimental program included determinations of the CaCO₃ percentage using three different approaches (chemically; through thermogravimetric analysis (TGA); and through a "sequential" loss on ignition (LOI) test), as well as XRD analyses, pH tests, and Atterberg limit tests.

The experimental work: (a) re-endorsed the classification previously used by INDOT that classifies soils into five groups based on the % of CaCO₃ ("soil with trace of marl"; "soil with little marl"; "soil with some marl"; "marly soil"; "marl"); (b) validated the use of any of the methods above for measuring the % of CaCO₃ (with the sequential LOI test having the advantage of also providing an estimate of the organic content); and (c) proposed a simple classification procedure to identify a marl soil in the field, based on the color of the dry soil and its reaction with a 1M HCL solution.

Both SPR-3005 and SPR-3227 included recommendations for implementation that reflected input from the project administrator (PA) and study advisory committee (SAC). A specific recommendation from both projects was that INDOT soil technicians be trained to perform the required tests and classify soils based on the revised classification systems.

This project was initiated to carry out the implementation of those recommendations. The project scope includes development of training material for instruction in performance of the revised classification tests and methods, delivery of that training to pertinent INDOT personnel, integration of the revised classification system into INDOT's standards (specifications 903.05 and 903.06), and establishment of a resource database for future training of personnel.

2. PROJECT OBJECTIVES

Within the general scope outlined above, the specific objectives of the proposed work are to:

- Administer training to select INDOT personnel and interested representatives from the geotechnical consulting/construction community.
- 2. Develop training materials to be used by INDOT to train additional personnel.

3. PROJECT TASKS

The two objectives outlined above were accomplished through the completion of four specific tasks.

Task 1: Collection of Sample Soils for Testing and Classification

Task Description

Demonstration of the classification method and testing procedures required that several sample soils be obtained from different locations around Indiana. Thus, a small collection program was necessary to acquire the needed samples.

Task Completion

The first task for this project was to identify/test reference soils to be used as supporting classification examples. Specifically, efforts focused on finding reference

soils with different percentages of organics and calcium carbonate that fell in the following categories:

- 1. 1 to 2 organic soils with no CaCO₃
- 2. 1 to 2 marly soils with no organics
- 3. 1 to 2 soils with both organics and CaCO₃ (critical to examine the combined use of the two classification systems)
- 4. 1 mineral soil
- 1 soil that provided a false positive to the presence of organics based on the LOI test

In order to collect information on site locations that may have organic and/or marl soils with the characteristics listed above, fact-finding interviews were conducted with a number of persons, including INDOT personnel, private contractors, and consultants: Tom Coffey (Alt & Witzing Engineering); Michael Wigger and Darren Pleiman (Earth Exploration Inc.); Shawn Marcum (ATC Associates); Firooz Zandi (K&S Engineers Inc.); Radha Daita (H.C. Nutting. Terracon Co.); and Joey Franzino, Jonathan Paauwe and Youlanda Belew (INDOT). As a result of these efforts, samples were obtained from three different sources (Figures 3.1 through 3.3). From 12 samples tested, 7 were chosen to be used as reference soils for supporting classification examples (Table 3.1).

Additional samples were taken from a fourth site, part of section 3, segment 13 (Daviess, Indiana) of I-69, in conjunction with another currently ongoing JTRP Project (SPR-3639, Engineering Properties of Marls). Details on the sampling operations are provided in the report for that project.

Task 2: Development of Training Material

Task Description

Materials were to be developed providing adequate instruction in both the revised classification system (for organic and marl soils) and the testing methods necessary to perform the classification. The training material was to be designed in such a manner as to be conducive to administration in a small "classroom" setting, with a target training time of approximately 3 hours. Specifically, the materials were to include:

- A PowerPoint presentation supported by concise instructional handouts.
- 2. Supporting classification examples for a variety of soils.
- 3. A short manual summarizing the classification system for both soils with supporting examples.

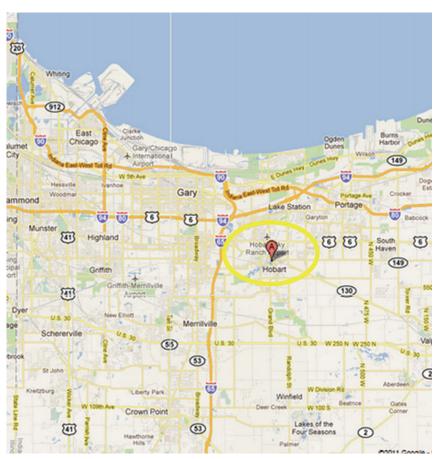


Figure 3.1 Sample source 1.

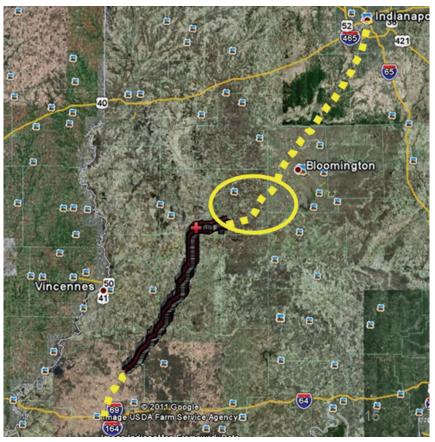


Figure 3.2 Sample source 2.

In addition to the hard copies provided to INDOT, digital copies of the aforementioned materials were to be uploaded to an internet repository for future access by INDOT personnel. The repository was also to include results from testing/classification of sample soils.

Task Completion

The following items were developed for the purpose of training INDOT personnel in the revised classification systems (for organic and marl soils) and the associated tests required for classifications. Copies of these items are included in the appendices of this report.

- PowerPoint Presentation—The presentation (Appendix 1) contains a short background section, which describes the need for a revised classification system for both organics and marls. The rest of the presentation is divided into "organic," "marl," and "combined" sections, which describe the required tests (LOI, colorimetric, and LLR for organic soils; sequential LOI for marls), outline the respective classification procedure, and present classification examples (using some the sample soils presented in Table 3.1).
- Lab Manual—A short manual was compiled (Appendix 2) that summarizes the objectives, procedures, and results from SPR-3005 and SPR-3227. The manual includes references for further inquiry.
- Short Procedure for Identification and Classification of Organic Soils—This document (Appendix 3) outlines the

- references, scope, apparatus, procedure, calculations, and report required for performance of the tests necessary for classification of organic soils (LOI, colorimetric, and LLR). It also includes sample data sheets for each of the tests.
- Short Procedure for Identification and Classification of Marly Soils—This document (Appendix 4) outlines the references, scope, apparatus, procedure, calculations, and report required for performance of the test necessary for classification of marly soils (sequential LOI). It also includes sample data sheets for the test.
- Classification Charts—These flowcharts (Appendix 5) demonstrate graphically the classification process for organic soils, marly soils, and combined (organic and marly) soils. They are necessary for the actual classification of soils (using the results from the tests in the Short Procedure above).
- Classification Checklists—These checklists (Appendix 6) provide bullet point steps for classification of organic soils, marly soils, and combined soils. They are to be used in conjunction with the Classification Charts (Appendix 5) as a quick reference for the classification procedure.
- Supporting Classification Examples—These items consist of sample data sheets (Appendix 7) with test results for the sample soils collected for demonstration of the testing procedure. They were designed to be used as accessory practice problems in the training sessions (see Task 3 below). However, they are also useful for classification practice, as the data sheets (containing raw data) can be used in conjunction with the PowerPoint presentation (containing the actual classification of the soils based on the data) for "self-study."



Figure 3.3 Sample source 3.

Task 3: Delivery of Training Sessions

Task Description

Training sessions were to cover the classification system and necessary testing methods, and to be administered to INDOT technicians, lab managers, geologists, engineers, and any other pertinent personnel, as well as representatives from select geotechnical consultants and contractors. Three to four sessions of approximately half a day (~4 hours) in length were to be held at several locations around the state, with the locations selected by INDOT.

Task Completion

A total of four training sessions were held at INDOT facilities around the state. First, a pilot session was held at INDOT's Indianapolis Materials Tests facility, where the attendees were primarily engineering staff and testing lab managers. This served as a trial run for the subsequent training sessions. Feedback was collected for improving the PowerPoint presentation, the handouts, and the delivery.

The remaining sessions were held at the Seymour District Office (which included representatives from Seymour and Indianapolis), the LaPorte District Office

TABLE 3.1 Sample soils used as supporting classification examples

Soil #	Soil name	LOI (%)	CaCO ₃ (%)	Source
1	"Soil with trace marl & organic matter"	8.0	2.4	Daviess, Indiana (EEI)
2	"Marl"	2.2	62.6	Daviess, Indiana (EEI)
3	"Soil with some marl"	2.4	21.1	Daviess, Indiana (EEI)
4	"Marly soil with organic matter"	7.3	26.2	Hobart, Indiana (EEI)
5	"Soil with some marl & organic matter"	6.8	23.9	Hobart, Indiana (EEI)
6	"Soil with trace marl"	2.3	3.2	Daviess, Indiana (EEI)
7	"Soil with trace marl—false positive"	3.6	4.7	ASTM CL

(which included representatives from LaPorte and Ft. Wayne), and finally at INDOT's Indianapolis Materials Tests facility, which included representatives from Indianapolis, Greenfield, and several geotechnical consultants and contractors. A complete listing of dates, locations, attendees, and affiliations, is included as Appendix 8.

The training sessions were delivered by Alain El Howayek, MSCE. First, a short background was provided on the necessity for improved classification systems for both organic and marly soils. Next, the test procedures required for each classification system were described. The colorimetric test in particular was demonstrated at each location, as not all attendees were familiar with its procedure. Following description of the required tests, the classification systems themselves were outlined. Finally, classification examples were demonstrated using actual test data (from the collected samples).

Attendees were issued an information packet upon arrival at the training sessions. The packets contained printouts of the PowerPoint presentation, "short procedures," classification charts, classification checklists, and supporting example data sheets. A CD was also included within each packet, containing electronic copies of the aforementioned items, as well as a copy of the lab manual summarizing SPR-3005 and SPR-3227. Feedback was collected following each training session through anonymous response forms and was used to improve and refine the sessions that followed. A copy of the feedback form is included as Appendix 9.

Task 4: Production of Training Video

Task Description

A short training video was to be prepared covering the materials from the training presentation, such that interested INDOT personnel could independently learn the classification system and testing procedures through self-study. The video was to include classification examples and demonstrations of the required tests. A hard disc copy of the video was to be delivered to INDOT for usage as necessary.

Task Completion

A short training video was developed covering the materials from the training presentation, such that INDOT personnel could learn the classification system and testing procedures through self-study. The video has

been uploaded to an online Joint Transportation Research Program repository at the following URL: http://dx.doi. org/10.5703/1288284315027. It includes a full description of the classification systems as well as examples and visual demonstrations of the required tests.

4. ACKNOWLEDGMENTS

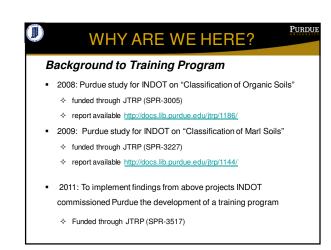
This training program was developed as part of SPR-3517. The principal investigators were Professors Marika Santagata and Antonio Bobet of Purdue University, and Mr. Nayyar Zia Siddiki of INDOT's Geotechnical Office. A number of people contributed to this work. The classification procedures were developed as part of two previous JTRP projects by Mr. Pao Tsung Huang, for organic soils, and by Dr. Chul Min Jung, for marly soils. A team of Purdue students was responsible for developing all training material for this implementation project. The team was headed by Mr. Alain El Howayek, and included Mr. Sulaiman Dawood, Mr. Andrew Ferdon, Mr. Alex Sangermano (voice on the video), and Mr. Michael Stockwell. Several members of INDOT have contributed to this training program, in particular Mr. Athar Khan, Manager of INDOT's Geotechnical Services, and Mr. Brian Dunbar, Mr. Ron Fine, Mr. Iqbal Khan, Dr. Tommy Nantung, and Mr. Mike Nelson, who participated in the pilot training program, and provided feedback on this presentation.

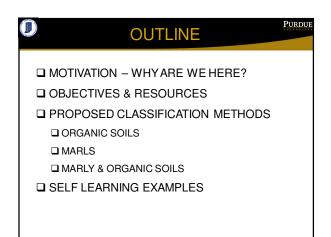
5. CONCLUSIONS

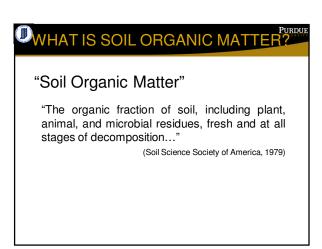
This implementation project, SPR-3517, completed the proposed objectives. Training was successfully administered to INDOT personnel and interested representatives from the geotechnical consulting/construction community on the revised classification methods for organic and marly soils developed in SPR-3005 and SPR-3227. Training materials were developed for use by INDOT in future training.

The accessory tasks were also successfully completed. Training materials were uploaded to an online repository for easy access by INDOT personnel. The INDOT Geotechnical Manual was updated to include the classifications systems developed in SPR-3005 and SPR-3227. A training video was produced and made available to INDOT for usage in future training sessions, and soil samples were collected from around the state for demonstration of the classification system.



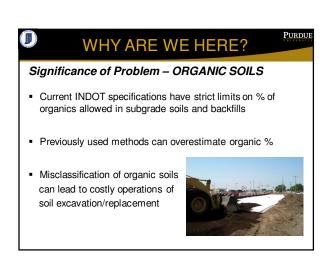




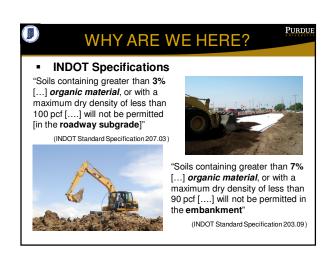


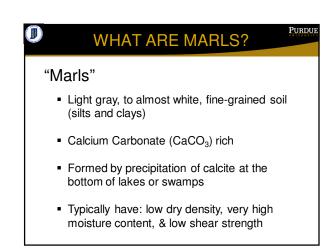


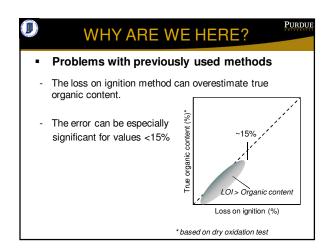


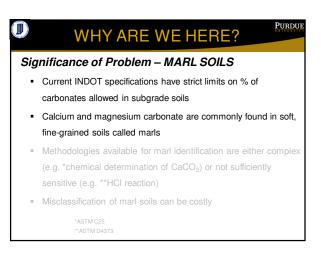


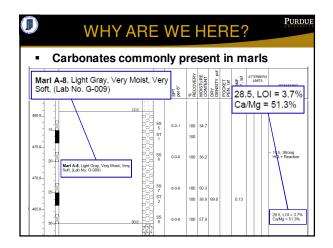


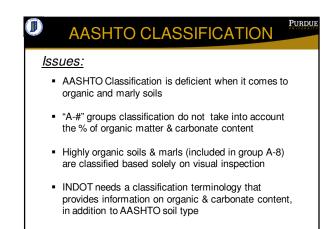




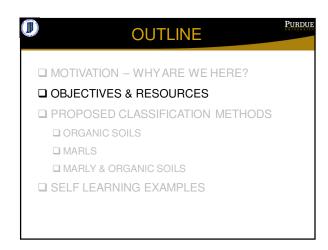


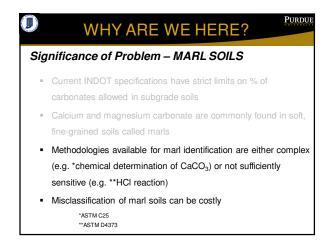


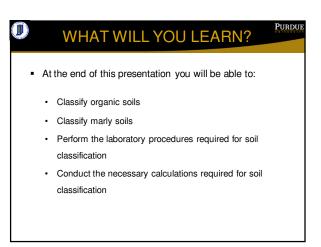


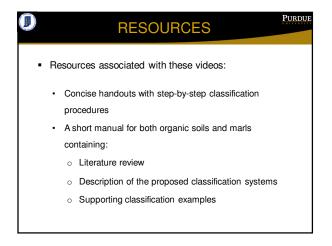


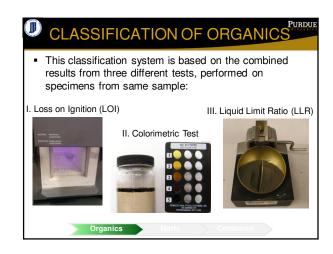


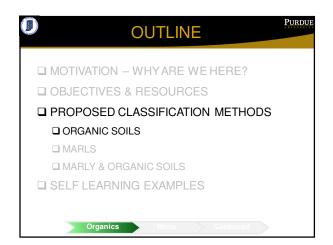


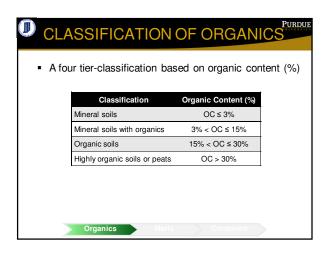


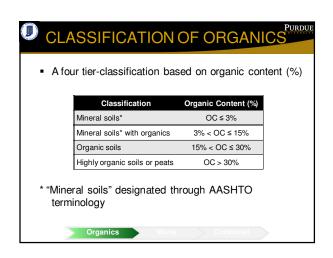


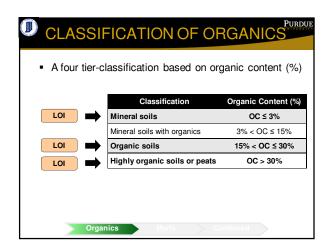


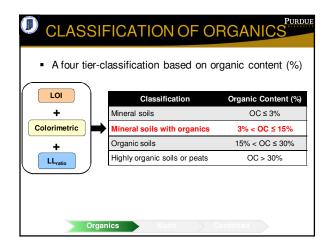


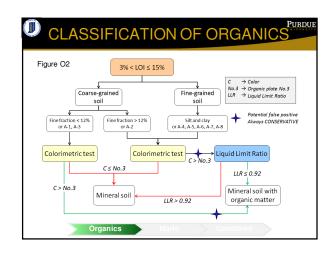


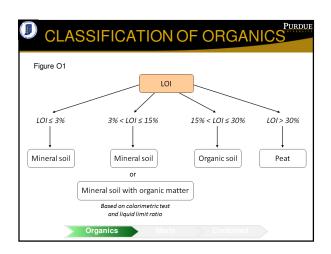


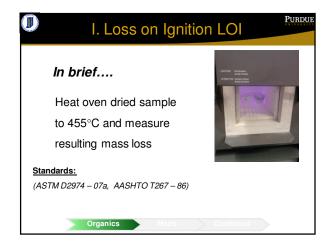


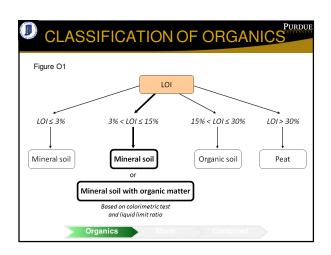






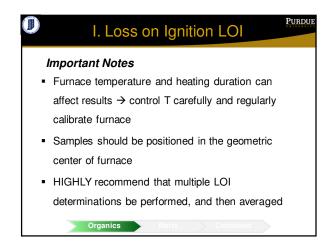




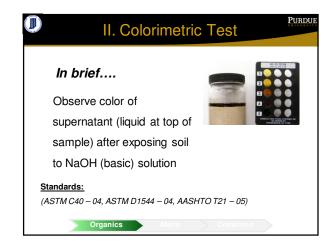


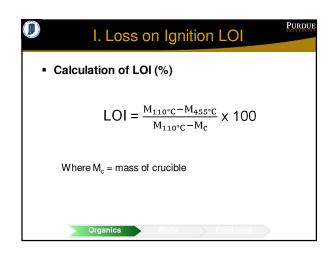


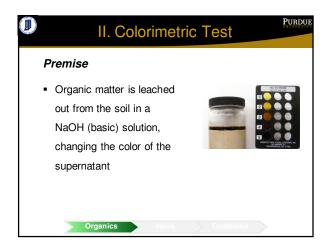


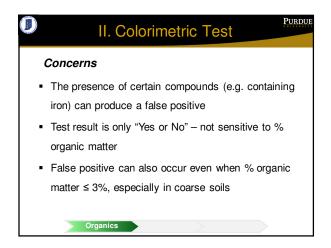


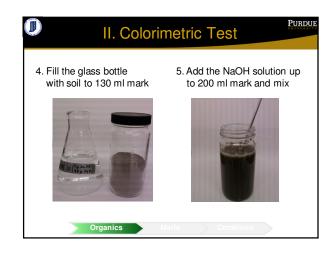


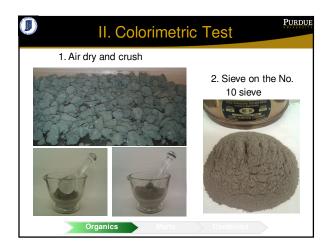


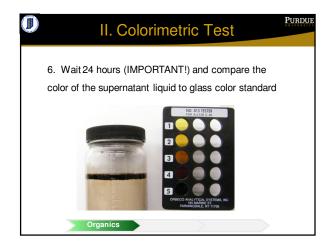


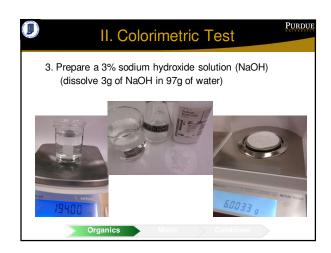


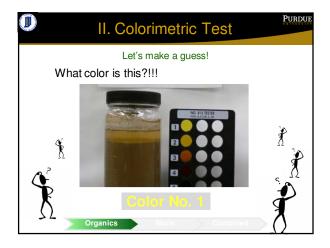




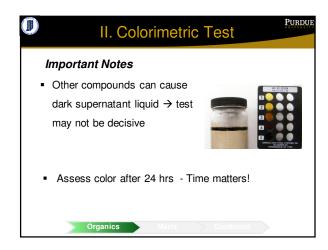




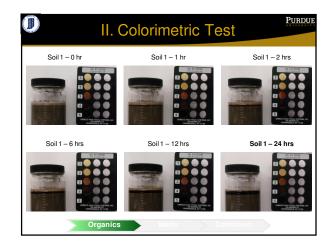


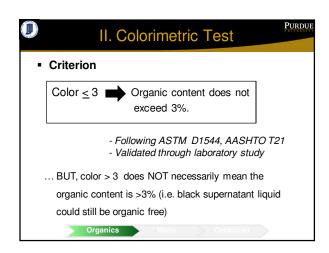


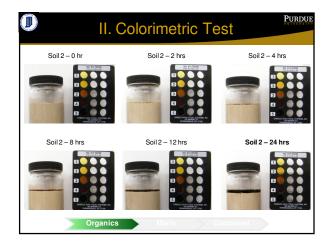


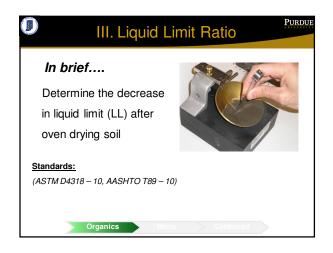




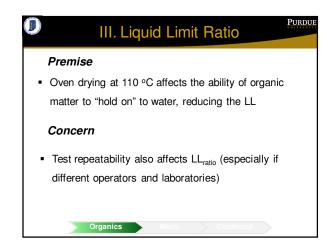






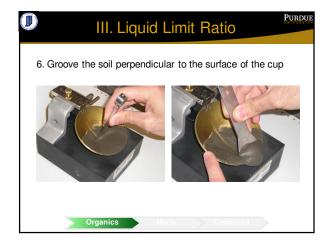


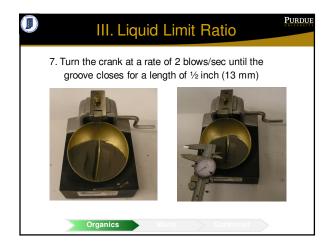


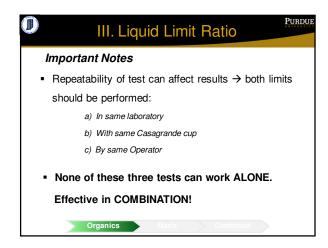


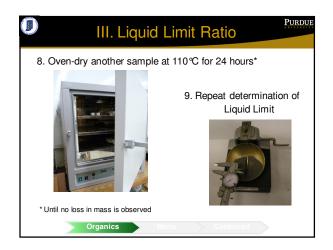


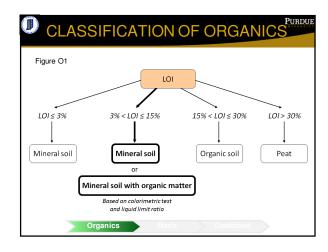


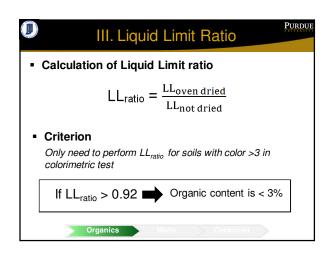


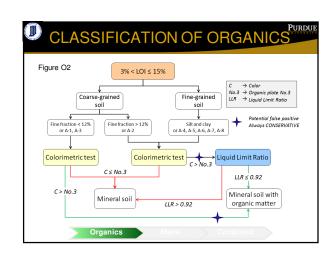


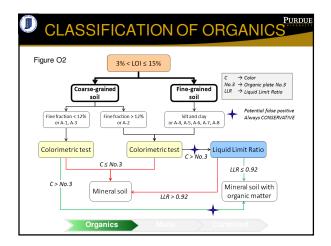


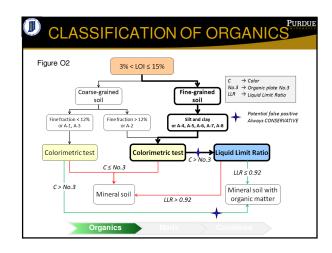


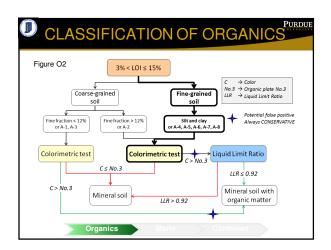


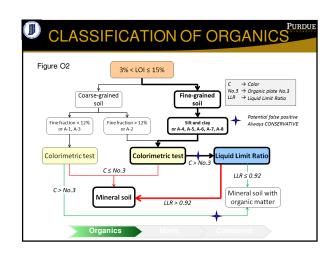


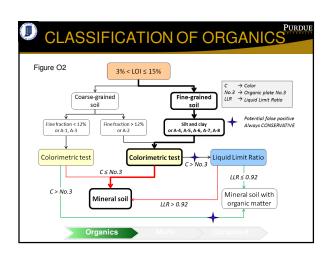


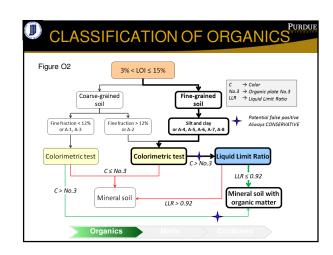


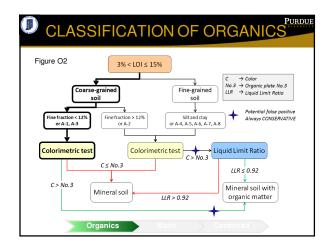


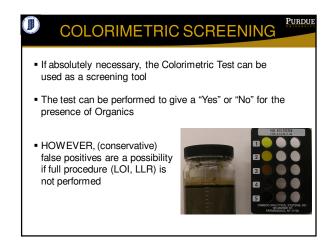


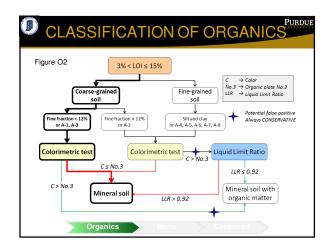




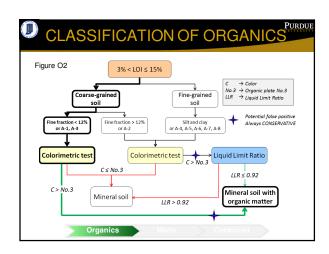




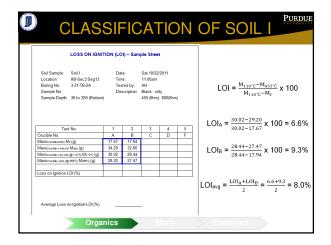


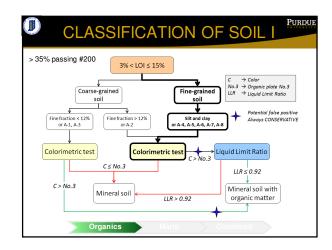


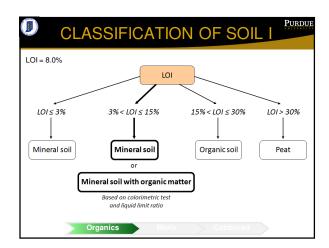




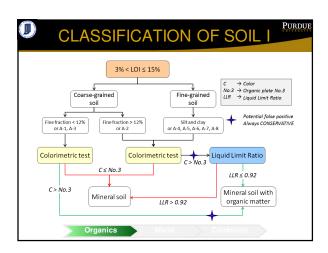


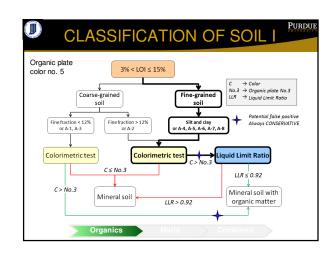


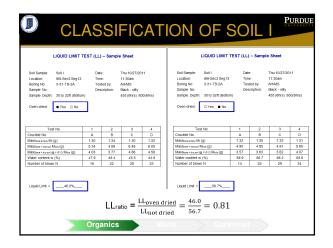


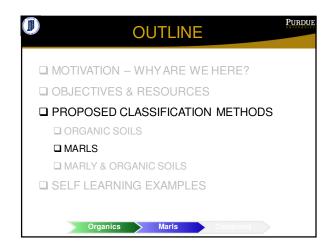


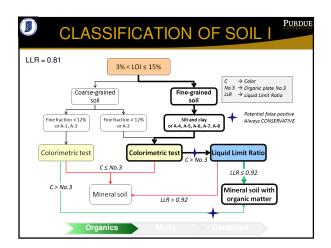


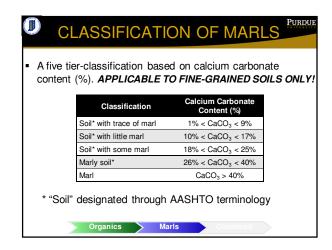


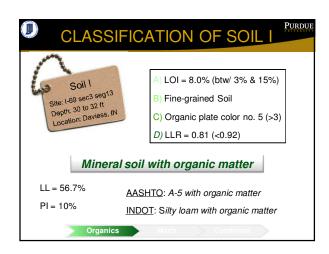


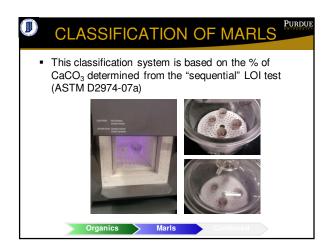


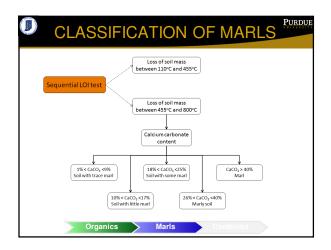


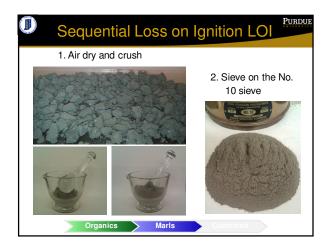


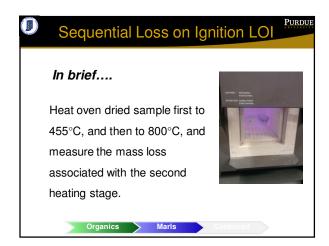


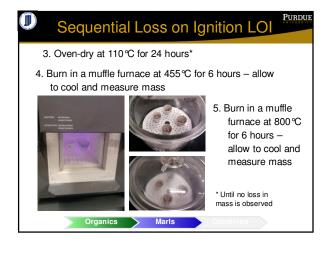


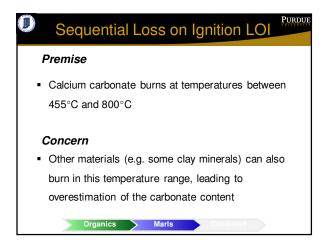


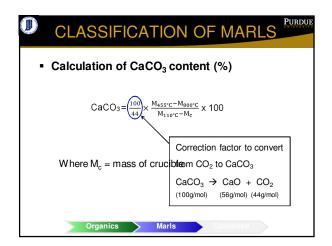


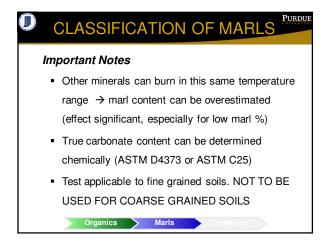


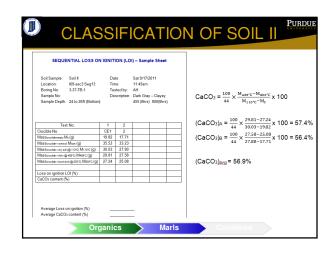


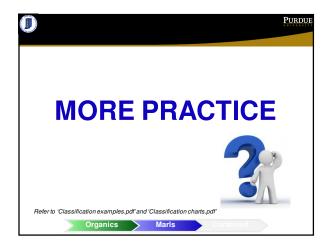


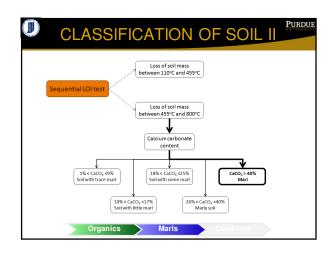




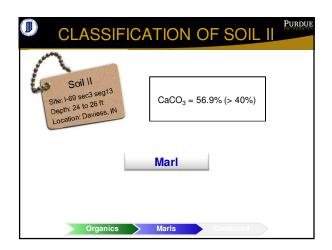




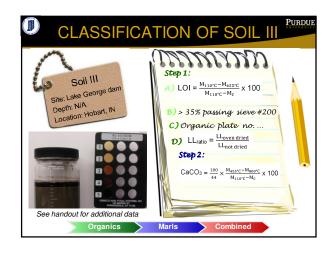


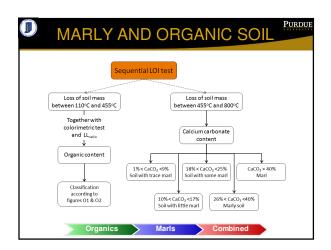


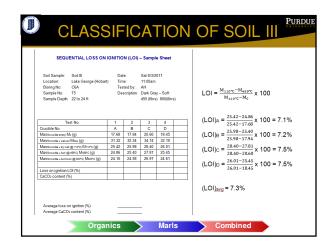


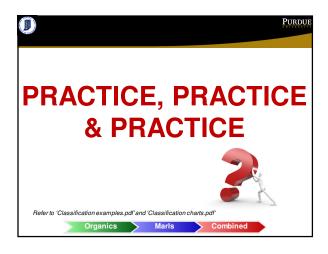


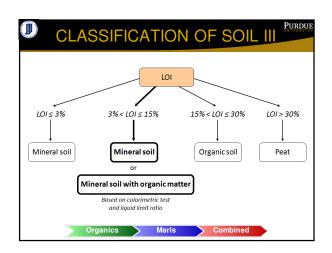




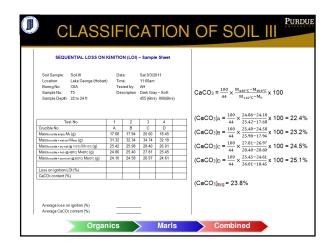


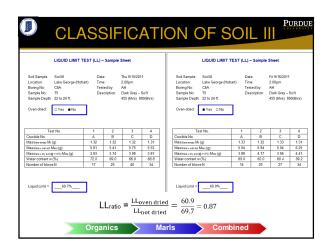


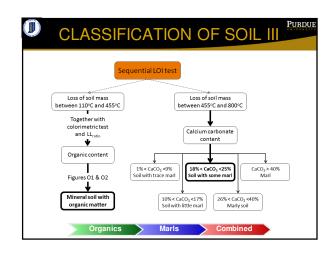


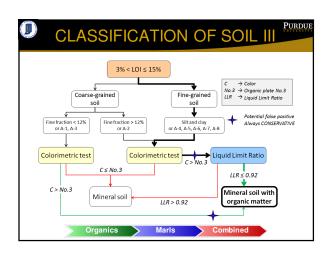


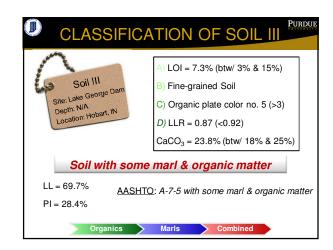


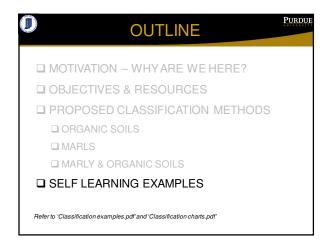


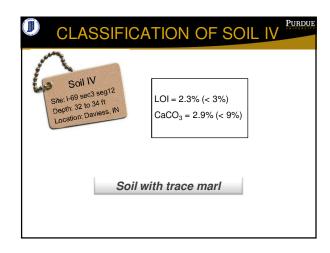


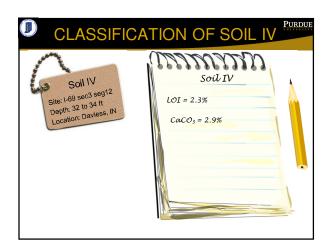


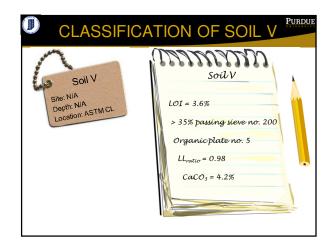


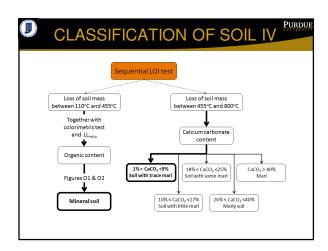


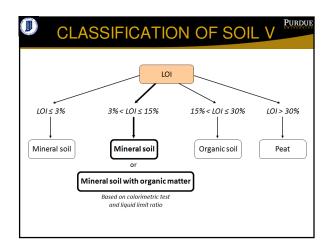


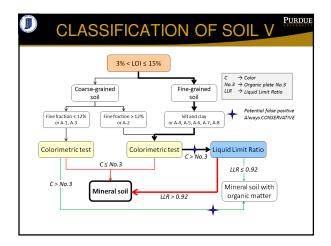


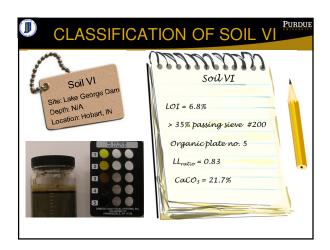


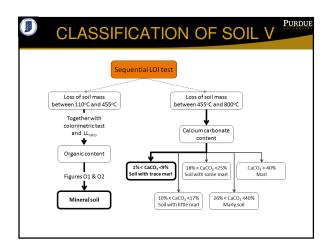


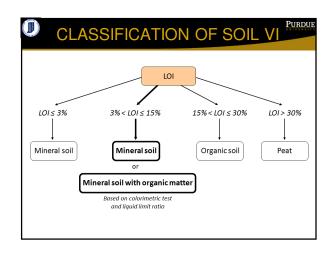


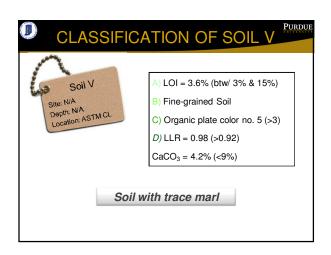


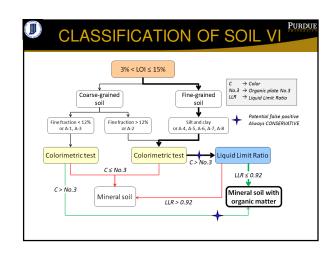


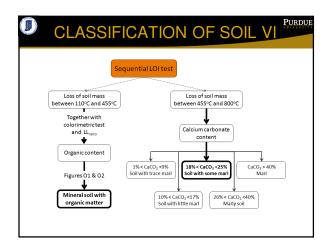


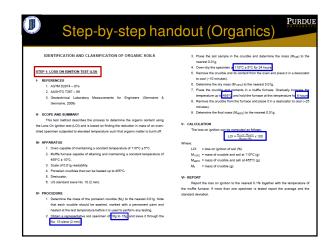


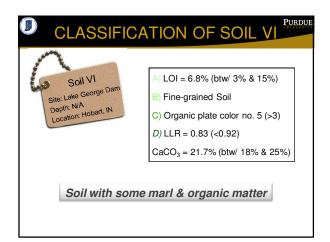


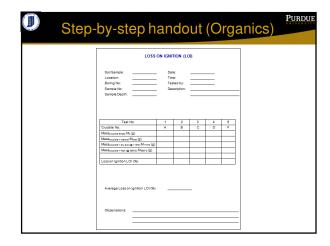


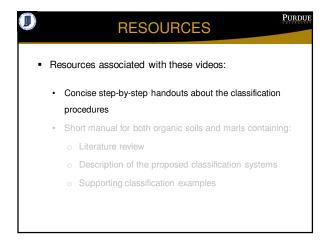


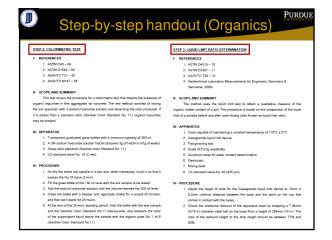


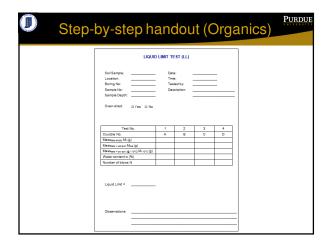


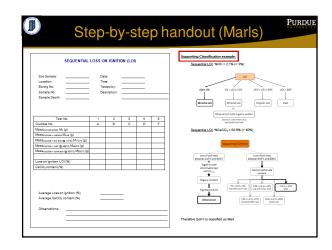


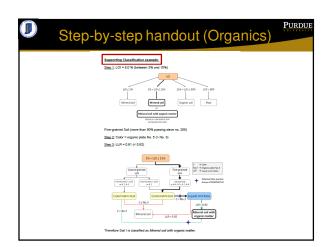


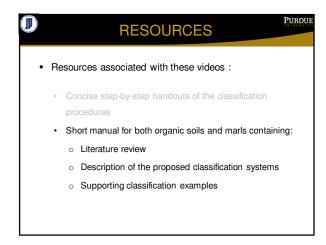


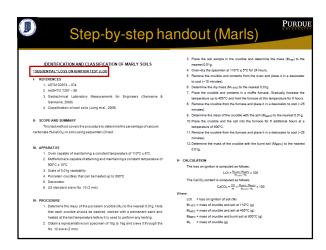


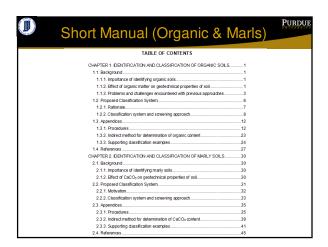


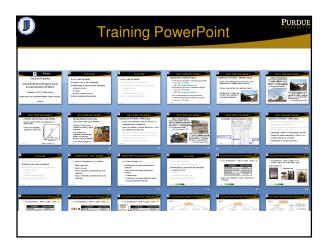


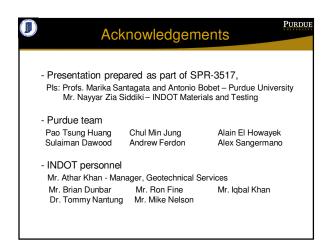














Lab Manual

Project Implementation:

Classification of Organic Soils and Classification of Marls Training of INDOT Personnel

Prepared by

Alain El Howayek
Graduate Research Assistant

Antonio Bobet Professor of Civil Engineering

Marika Santagata
Associate Professor of Civil Engineering

Last revised on September 15, 2012

TABLE OF CONTENTS

CHAPTER 1. IDENTIFICATION AND CLASSIFICATION OF ORGANIC SOILS	1
1.1. Background	1
1.1.1. Importance of identifying organic soils	1
1.1.2. Effect of organic matter on geotechnical properties of soils	1
1.1.3. Problems and challenges encountered with previous approaches	3
1.2. New Classification System	6
1.2.1. Rationale	7
1.2.2. Classification system and screening approach	8
1.3. Appendices	11
1.3.1. Procedures	11
1.3.2. Indirect method for determination of organic content	25
1.3.3. Supporting classification examples	26
1.4. References	29
CHAPTER 2. IDENTIFICATION AND CLASSIFICATION OF MARLY SOILS	32
2.1. Background	32
2.1.1. Importance of identifying marly soils	32
2.1.2. Effect of CaCO ₃ on geotechnical properties of soils	32
2.2. New Classification System	33
2.2.1. Motivation	34
2.2.2. Classification system and screening approach	35
2.3. Appendices	37
2.3.1. Procedures	37
2.3.2. Indirect method for determination of CaCO ₃ content	42
2.3.3. Supporting classification examples	44
2.4. References	48

LIST OF FIGURES

Figure 1-1: Loss on ignition versus organic content (Huang et al., 2009)4
Figure 1-2: Liquid limit ratio versus organic content (Huang et al., 2009)8
Figure 1-3: Soil classification based on organic content in revised classification system
(Huang et al., 2009)9
Figure 1-4: Approach for the classification of soils with $3\% < LOI \le 15\%$ (Huang et al.,
2009)
Figure 1-5 : Colorimetric test at different times after adding the NaOH solution 18
Figure 1-6: Supporting classification example - Soil 1
Figure 1-7: Supporting classification example - Soil 4
Figure 1-8: Supporting classification example - Soil 7
Figure 1-9: Supporting classification example - Soil 9
Figure 2-1: Approach for classifying soils in terms of calcium carbonate content based
on sequential LOI test (Jung et al., 2009)
Figure 2-2: Recommended field classification procedure for marly soils (Jung et al.,
2009)
Figure 2-3: Weight loss obtained from TGA test (Jung et al., 2009)44
Figure 2-4: Supporting classification example - Soil 1-1
Figure 2-5: Supporting classification example - Soil 1-9
Figure 2-6: Supporting classification example - Soil 2-1
Figure 2-7: Supporting classification example - Soil 3-2
Figure 2-8: Supporting classification example - Soil 3-4

LIST OF TABLES

Table 1-1: Summary of test results for natural soils (Huang et al., 2009)	5
Table 1-2: Criteria of organic soils classification (Huang et al., 2009)	7
Table 1-3: Supporting classification examples for organic soils	26
Table 2-1: Criteria of marly soils classification (Jung et al., 2009)	34
Table 2-2: Supporting classification examples for Marly soils	45

This manual summarizes the methods and procedures developed for the classification of organic and marly soils by researchers at Purdue University. This work was conducted as part of two research projects funded through the Joint Transportation Program (SPR: 3005 - Classification of organic soils; SPR: 3227 – Classification of marl soils). The methods have been incorporated in INDOT standard specification 903.05 and 903.06 respectively. Development of the manual and of the accompanying training material has been funded through JTRP under SPR: 3517.

CHAPTER 1. IDENTIFICATION AND CLASSIFICATION OF ORGANIC SOILS

1.1. Background

1.1.1. Importance of identifying organic soils

From a geotechnical engineering perspective the presence of organic matter in soils can often represent a concern due to its negative influence on the mechanical properties. The presence of organic matter is generally associated with higher compressibility and creep, often unsatisfactory strength characteristics, as well as interference of organic constituents with soil stabilization reactions. These concerns pertain not only to peats and highly organic soils, but may apply also to soils with relatively low (<10%) values of organic content. For this reason many agencies have strict limits on the maximum allowable organic content in subgrade soils and backfills, requiring that it falls below threshold values in the 2-7% range (1). The threshold value used by INDOT, standard specification 207.03 (2), is 3% organic content.

1.1.2. Effect of organic matter on geotechnical properties of soils

It is recognized that the presence of organic matter plays a critical role in affecting both the geotechnical index properties and engineering properties of soils. Its effects can be summarized as follows:

- Water content: Organic soils usually have very high water content. A more fibrous structure and/or a higher organic content result in large voids and the high cation exchange capacity of organic matter increases the attraction of water molecules; both characteristics result in high water content.
- 2. <u>Gas content</u>: The gas content of a soil is a very important parameter, which can change with time. The gas content influences permeability, consolidation rate,

and pore pressure generation (3). Organic matter may undergo chemical decomposition which is accompanied by the production of marsh gas with small amounts of nitrogen and carbon dioxide.

- 3. <u>Bulk density</u>: Typically, soils with higher organic content have low bulk density, especially when the fiber content is high (i.e. low degree of decomposition).
- 4. <u>Specific gravity</u>: The specific gravity of a soil tends to decrease as the organic content increases. Values of specific gravity less than 2.0 are an indication of a soil with high organic content (4).
- 5. <u>Atterberg limits</u>: In general both liquid limit and plastic limit increase with organic content due to the higher water adsorption capacity of organic matter.
- 6. <u>Shrinkage potential</u>: Shrinkage can be significant in soils with high organic content. For loose high organic soils, the volume change can reach 70% of their initial volume upon drying.
- 7. <u>Compaction behavior</u>: The maximum dry density decreases with organic content and the optimum moisture content increases as the organic content increases.
- 8. <u>Strength</u>: The strength of organic soils strongly depends on the organic content. As the organic content increases, the strength quickly decreases. However, the fibers in the soil (the fiber content is related to the degree of humification, i.e. decomposition of the soil), may produce a reinforcing effect on the soil matrix increasing the shear strength of the soil.
- 9. <u>Permeability</u>: The permeability of organic soils is much higher than inorganic soils. For example, the permeability of an organic soil with more than 75% organic content is 100 to 1000 times larger than typical values for inorganic clays. However, organic soils exhibit large deformations induced by creep. As a result the pore space in the soil may be drastically reduced with time, resulting in a dramatic decrease of permeability.
- 10. Compressibility: Peats and organic soils exhibit a much higher compressibility than other geotechnical materials *(5)*. First, organic soils have much higher natural water content and void ratio than inorganic soils; and second, organic soils have the highest values of C_α/C_c *(6, 7)*, which results in very high secondary consolidation (creep) deformations.

In summary, the geotechnical properties of organic soils depend on the following factors: 1) organic content; 2) type of organic matter; 3) degree of decomposition of the organic matter; and 4) void ratio. In general, as the organic content increases, water content, Atterberg limits, cation exchange capacity, and acidity all increase, whereas specific gravity, bulk density, plastic index, and efficiency of compaction decrease. In addition to organic content, the type of organic matter and the degree of decomposition of organic matter are two critical factors affecting the strength, permeability, and compressibility of organic soils. A more fibrous structure and a lower degree of decomposition usually lead to higher permeability and compressibility. The strength of an organic soil is reduced with the presence of organic matter; however, a fibrous structure, if present, may increase the shear strength and provide some tensile strength capacity. The void ratio depends on organic content, type of organic matter and degree of decomposition: a more fibrous structure, a higher organic content, and a lower degree of decomposition all lead to a more open structure, i.e. an increased void ratio. The void ratio controls the major properties of organic soils, especially compressibility. Their short, but large, primary consolidation and large secondary consolidation (creep) tend to create problems in civil engineering practice when organic soils are present.

1.1.3. Problems and challenges encountered with previous approaches

Methods previously used in practice for the identification of organic soils and for the quantification of organic matter have shortcomings when applied to soils with organic matter content less than ~10%. For such soils 1) the loss on ignition LOI often overestimates the true organic content, and 2) the criteria employed by the ASTM and ASHTO classification systems are generally insensitive to the presence of modest amounts of organic matter.

1.1.3.1. <u>Inaccuracy of LOI test for measuring true organic content</u>

The loss on ignition (LOI) test is the method most commonly employed in practice for assessing organic content, and measurements of the LOI are routinely

conducted to establish the suitability of a soil as a subgrade material and decide on the need for soil removal or treatment. Despite the simplicity and cost effectiveness of the LOI test, heating temperature and duration can significantly affect its results, and the presence of a number of inorganic constitutes (e.g. some hydrated alluminosilicates, gypsum) can lead to overestimate the soil's true organic content. The error can be especially significant in soils with organic content <10% (8, 9), potentially requiring unnecessary and costly operations of soil excavation and removal, or soil modification/stabilization.

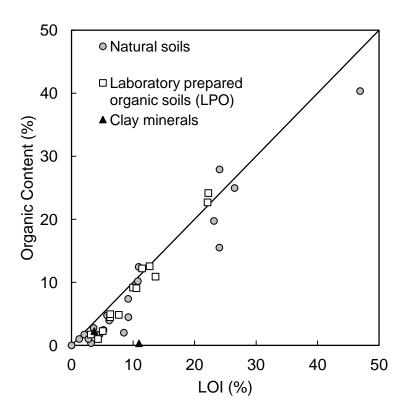


Figure 1-1: Loss on ignition versus organic content (10)

Measurements of the organic matter content obtained from the results of the dry combustion analysis, and herein considered representative of the "true" organic content of the soil are compared to the LOI data in Figure 1-1. The data presented in Table 1-1 and Figure 1-1 show that the LOI test may significantly overestimate the true organic content of some soils, particularly for low values of the organic content. This is clearly

an important concern if the loss on ignition method is to be used to identify and screen soils. For example, based on the LOI test, soils 1, 4, and 9 would not be considered adequate for use as subgrade soils in the State of Indiana, which employs a maximum threshold value for the organic content of 3%. The measurement of the true organic content using the dry combustion analysis shows instead that all three are viable subgrade geomaterials.

Table 1-1: Summary of test results for natural soils (10)

Soil ID	Classification (ASTM D2487)	LL	PL	PI	Liquid Limit Ratio	Colorimetric Test Result	Organic Content	LOI
	n/a	%	%	%	n/a	No.	%	%
Soil 1	CL	49.0	27.2	21.8	0.96	1	0.4	3.2
Soil 2	ML	36.9	28.0	8.9	0.93	3	1.7	2.1
Soil 3	ML	31.2	23.6	7.5	0.97	3	2.8	3.6
Soil 4	ML	46.7	35.0	11.6	0.88	3	2.4	5.2
Soil 5	CL	32.1	20.1	12.0	0.98	1+	1.0	1.2
Soil 6	ML	-	-	-	-	<1	0.0	0.0
Soil 7	OL	47.7	38.6	9.1	0.68	5	12.5	10.9
Soil 8	ML	41.7	33.8	7.8	0.85	4	4.8	5.8
Soil 9	CL	34.0	14.7	19.3	1.00	4	1.7	4.5
Soil 10	CH	60.5	19.3	41.2	0.99	1+	1.0	2.7
Soil 11	OH	151.1	123.0	28.1	0.58	-	38.5	43.2
Soil 12	MH	79.5	57.8	21.7	0.77	5	27.9	24.1
Soil 13	MH	94.2	67.4	26.8	0.79	4+	19.7	23.2
Soil 14	MH	112.8	58.9	53.8	0.87	4	15.5	24.0
Soil 15	MH	121.7	62.6	59.1	0.86	4	10.2	10.8
Soil 16	ОН	117.7	82.4	35.3	0.55	-	25.0	26.5
Soil 17	ML	39.1	25.2	13.9	0.96	4	4.0	6.1
Soil 18	MH	73.5	42.7	30.8	0.77	3	7.4	9.2
Soil 19	CL	48.6	25.1	23.5	0.82	3+	2.0	8.5
Soil 20	MH	67.2	39.9	27.3	0.92	5	4.5	9.2
Soil 21	MH	69.0	45.3	23.7	0.82	4	6.47	7.25
Soil 22	ML	48.0	32.3	15.7	0.80	3	0.00	2.71

1.1.3.2. Non-sensitivity of the ASTM and AASHTO classification systems to the presence of low organic matter

The Unified Soil Classification System *(11)* considers organic soils as a subgroup of fine-grained soils: silts and clays are classified as organic based on the reduction in liquid limit measured after oven drying the soil (if LL_{oven dried}/LL_{non dried} <0.75, a clay or a silt is termed organic and denoted as OL or OH depending on whether the LL is smaller or greater than 50). While there is no doubt that the presence of organic matter markedly affects the LL, the criterion does not discriminate between different levels of organic, and is not consistently sensitive to the presence of <10% amounts of organic matter. Note that ASTM D2487-10 *(11)* also considers highly organic soils, which it terms peats. Such soils are classified based on the prevalence of organic matter, their dark color and organic odor. Similarly, the AASHTO classification system considers only highly organic soils (peat or muck), which are included in group A-8, and classified based solely on visual inspection. The AASHTO system does not consider the impact of organic matter in any of the other groups.

For example, Table 1-1 shows that soils 8, 12, 13, 14, 15, 17, 18, 20, and 21 have a liquid limit ratio larger than 0.75. Thus they are classified as non organic soils according to ASTM D2487-10 (11). However, all these soils have organic content higher than 3% (and as high as 27.9%) and they would not be considered viable subgrade soils in the State of Indiana.

1.2. New Classification System

Prior to the work conducted as part of SPR-3005, the identification and classification of organic soils within INDOT relied on the loss on ignition method. As discussed in the previous section, this method can lead to incorrect classification of soils, especially given the strict guidelines on organic content used by INDOT to determine the acceptability of a soil for a given application (e.g. <3% for a subgrade soil). This, in turn, may lead to erroneously considering a material unviable for a given application, and generate unnecessary costs for material replacement/treatment. The new classification system, developed as part of SPR-3005 is a four tier-classification

that is based on the combined results of three different tests: loss on ignition (LOI), colorimetric test, and liquid limit ratio determination. It replaces the previous INDOT classification system, INDOT standard specification 903.05 (2), which relied on 5 tiers and was based exclusively on the result of the LOI test. Table 1-2 summarizes the four different categories for organic soils classification in the new classification system. Soils with organic content less than or equal to 3% are termed *mineral soils*. If the organic content is greater than 3% and less than or equal to 15%, soils are classified as *mineral soils with organics*. Once the organic content falls in the 15%-30% range, the term organic soils is employed. Finally, soils with organic content higher than 30% are termed *highly organic soils* or peats.

Table 1-2: Criteria of organic soils classification (10)

Classification	Organic Content OC (%)
Mineral soils*	OC ≤ 3%
Mineral soils* with organics	3% < OC ≤ 15%
Organic soils	15% < OC ≤ 30%
Highly organic soils or Peats	OC >30%

^{* &}quot;Mineral soils" designated through USCS/AASHTO terminology

1.2.1. Rationale

The rationale for the new classification system can be summarized as follows:

- 1. Soils with organic content less than 3% are usually considered as mineral soils in most existing classification systems, as the presence of 3% or less organic matter does not significantly change the soil's properties. Also, the Indiana specifications for roadway construction section 207.03 (2) require that "soils containing greater than 3% by dry weight calcium, magnesium carbonate or organic material [as determined based on the loss on ignition test in AAHSTO T267]... will not be permitted within the specified thickness of the subgrade". Therefore, 3% of organic content is an acceptable boundary for mineral soils.
- 2. The results of the Atterberg limit tests conducted by Huang et al. (10) show that when the liquid limit ratio is smaller than 0.75, the organic content of the given soil is around 15-18% (see Figure 1-2). Thus, soil with organic content less than

15% would be classified as inorganic based on the USCS. This is the basis for using a 15% organic content as a means to distinguish between organic soils and mineral soils with organic matter.

 30% of organic content is adopted to be the boundary between organic soil and highly organic soil (Peat) in many currently existing classification systems such as the Canadian System of Soil Classification (CSSC), as well as in the criteria previously used by INDOT (2).

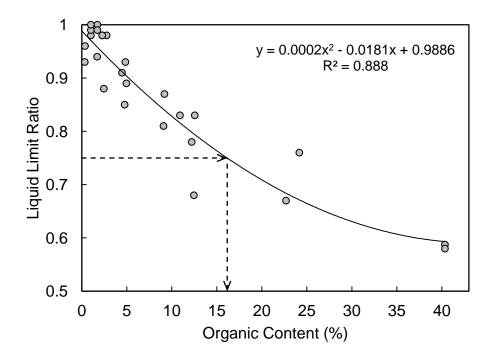


Figure 1-2: Liquid limit ratio versus organic content (10)

1.2.2. Classification system and screening approach

Figure 1-3 presents the recommended test procedure for classifying soils based on organic content in form of a flow chart. The LOI test is used first to provide a first assessment of the organic content. Figure 1-3 shows that based on the outcome of the LOI test it is possible to classify the soils in one of the four categories outlined above with the only uncertainty remaining in the case in which the LOI falls in the 3-15% range. In this case, the LOI may potentially overestimate the soil's true organic content

and thus an alternative screening approach based on the use of the colorimetric test and/or the liquid limit test is proposed.

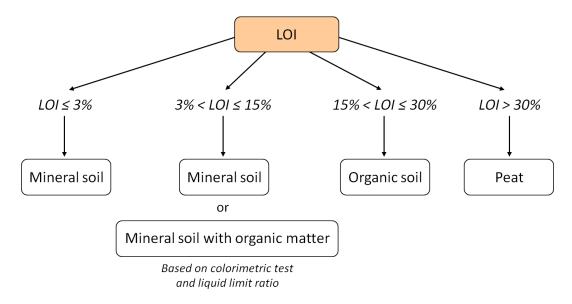


Figure 1-3: Soil classification based on organic content in revised classification system (10)

This second screening is summarized in Figure 1-4: for coarse-grained soil with fine fraction (i.e. passing #200 sieve) less than 12%, the colorimetric test is performed. If the color is lighter or equal to the organic plate No.3, the soil is considered to have negligible organic content, i.e. it is concluded that the LOI test overestimates the true organic content of the soil, which can be considered a mineral soil. Reliance on the results of the colorimetric test is based on the sensitivity of this test to the presence of organic matter in coarse-grained soils.

For fine soils and coarse soils with fine fraction greater than 12%, the colorimetric test also follows the LOI determination. If the color is lighter or equal than the organic plate No.3, the same conclusion as above is drawn, i.e. the soil is classified as a mineral soil. If the color is darker than No. 3 the screening process may be terminated if it is deemed acceptable that false positives may occur (i.e. that a soil may be erroneously considered as having organic content greater than 3%). If this not considered acceptable, the LL ratio is determined as a means to correct for these false positive results. Provided that the LL ratio is smaller than a given critical value (denoted

in Figure 1-4 as LL_{crit}), it can be concluded that a soil's organic content is higher than a threshold value of 3%. Based on the data for Indiana soils collected as part of SPR-3005, a value of LL_{crit} equal to 0.92 is recommended (see Figure 1-2).

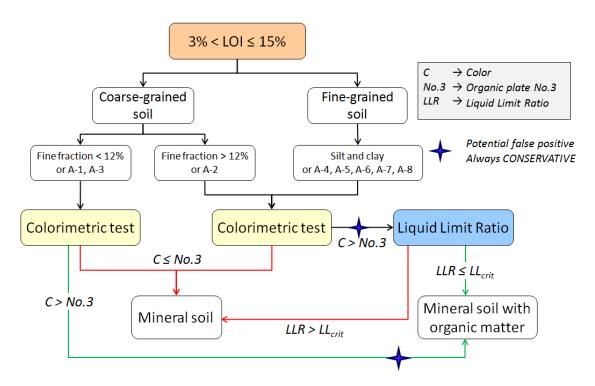


Figure 1-4: Approach for the classification of soils with 3% < LOI ≤ 15% (10)

When classification of the soil is not required, and a preliminary assessment of the presence of organic matter in a soil is required, the colorimetric test may be used as a "screening tool." In this case, the test provides a "yes"/"no" answer, i.e. if the color is less or equal than no.3, the soil can be assumed to have negligible organic content. If, instead, the color is greater than no. 3, it can be concluded that the organic content is likely to exceed 3%. Based on the data for Indiana soils examined as part of SPR-3005, no false negatives were observed (i.e. all organic soils were successfully detected). However, the method can generate false positives (i.e. color > no. 3 even for negligible % of organic matter), which may be resolved using the full procedure outlined in Figure 1-3 and Figure 1-4. Use of this screening method in coarse soils is discouraged.

1.3. Appendices

1.3.1. Procedures

1.3.1.1. Loss on Ignition Test (LOI)

a. References

- 1. Standard test methods for moisture, ash, and organic matter of peat and other organic soils (ASTM D2974 07a) (12)
- 2. Standard method of test for determination of organic content in soils by loss on ignition (AASHTO T267 86) (13)
- 3. Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009) (14)

b. Scope and Summary

This test method describes the process to estimate the organic content using the loss on ignition test (LOI) and is based on finding the reduction in mass of an oven-dried specimen subjected to elevated temperature such that organic matter is burnt off.

c. Background

Organic content can be determined using different methods. The LOI test is straightforward and is typically used in geotechnical laboratories. It measures the loss of mass by ignition when an oven-dried specimen (110°C) is placed in a furnace at much higher temperature (455°C), and assumes that this mass loss is entirely due to the oxidation of organic matter. Estimates of the organic content obtained from the LOI test usually exceed the true organic content because other processes (e.g. the dedydroxilation of some clay minerals) may be responsible for loss of mass at elevated temperatures. A more accurate method for determining the true organic content is the dry combustion test (see section 1.3.2).

d. Apparatus

1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.

- 2. Muffle furnace capable of attaining and maintaining a constant temperature of $455^{\circ}\text{C} \pm 10^{\circ}\text{C}$.
- 3. Scale with 0.01g readability.
- 4. Porcelain crucibles that can be heated up to 455°C.
- 5. Desiccator.
- 6. US standard sieve No. 10 (2 mm).

e. Procedure

- Determine the mass of the porcelain crucible (M_c) to the nearest 0.01g. Note that each crucible should be washed, marked with a permanent paint and heated at the test temperature before it is used to perform any test.
- 2. Obtain a representative soil specimen of 10g to 15g, and sieve it through the No. 10 sieve (2 mm) (13).
- 3. Place the soil sample in the crucible and determine the mass (M_{cws}) to the nearest 0.01g.
- 4. Oven-dry the specimen at 110°C ± 5°C for 24 hours (or until no mass loss is observed).
- 5. Remove the crucible and its content from the oven and place it in a desiccator to cool (~10 minutes).
- 6. Determine the dry mass $(M_{110^{\circ}C})$ to the nearest 0.01g.
- 7. Place the crucible and contents in a muffle furnace at 455°C for 6 hours.
- 8. Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).
- 9. Determine the final mass (M $_{455^{\circ}\text{C}}$) to the nearest 0.01g.

f. Calculation

The loss on ignition is computed as follows:

$$LOI = \frac{M_{110^{\circ}C} - M_{455^{\circ}C}}{M_{110^{\circ}C} - M_{C}} \times 100$$

Where:

LOI = loss on ignition of soil (%)

 $M_{110^{\circ}C}$ = mass of crucible and soil at 110°C (g)

 $M_{455^{\circ}C}$ = mass of crucible and ash at 455°C (g)

M_c = mass of crucible (g)

g. Report

Report the organic content to the nearest 0.1% together with the temperature of the muffle furnace. If more than one specimen is tested, report the average and the standard deviation of the values.

LOSS ON IGNITION (LOI) - Sample Sheet

Soil Sample: Soil I Date: Sat 10/22/2011

Location: I69-Sec3 Seg13 Time: 11:00am

Boring No: 3-31-TB-2A Tested by: AH

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2	3	4	5
Crucible No.	А	В	С	D	F
Mass _{crucible empty} M _c (g)	17.67	17.94			
Mass _{crucible + wet soil} M _{cws} (g)	34.29	32.80			
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)	30.02	28.44			
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.20	27.47			
Loss on Ignition LOI (%)	6.6	9.3			

Average Loss on Ignition LOI (%)	8.0
Observations:	
Observations.	

LOSS	ON IGNI	TION (LO	1)		
Soil Sample: Location: Boring No: Sample No: Sample Depth:		-			
Test No.	1	2	3	4	5
Crucible No.	Α	В	С	D	F
Mass _{crucible empty} M _c (g)					
Mass _{crucible + wet soil} M _{cws} (g)					
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)					
Mass _{crucible + Ash} (@ 455°C) M _{455°C} (g)					
Loss on Ignition LOI (%)					
Average Loss on Ignition LOI (%)					
Observations:					

1.3.1.2. Colorimetric test

a. References

1. Standard test method for organic impurities in fine aggregates for concrete (ASTM C40 – 04) (15)

- 2. Standard test method for color of transparent liquids (Gardner color scale) (ASTM D1544 04) (16)
- 3. Standard method of test for organic impurities in fine aggregates for concrete (AASHTO T21 05) (17)
- 4. Standard specification for materials for aggregate and soil-aggregate subbase, base, and surface courses (AASHTO M147 65) (18)

b. Scope and Summary

This test covers the procedure for a colorimetric test that detects the presence of organic impurities in soils. The test method consists of mixing the soil specimen with a sodium hydroxide solution and observing the color produced. If it is darker than a standard color (Gardner Color Standard No. 11), organic impurities may be present.

c. Background

The colorimetric test is one of several techniques that can be used to derive information on the presence of organic matter but without necessarily providing a quantitative assessment of the organic content of a soil. The test shows great sensitivity to the presence of organic matter in both fine and coarse soils. The colorimetric test is a relatively easy, economic and not time consuming method. However, the test can lead to false positives (i.e. the supernatant can turn dark even though no organic matter is present). It is recommended to use this technique in conjunction with other tests (e.g. LOI, liquid limit ratio) in order to identify organic content.

d. Apparatus

Transparent graduated glass bottles with a minimum capacity of 250 ml.

- A 3% sodium hydroxide solution NaOH (dissolve 3g of NaOH in 97g of water)
- 3. Glass color standard (Gardner color Standard No. 11).
- 4. US standard sieve No. 10 (2 mm).

e. Procedure

- Air-dry the entire soil sample in a pan and, when necessary, crush it so that it passes the No.10 sieve (2 mm) (the opening size of the No.10 sieve (2mm) corresponds to the definition of fine aggregate according to AASHTO M147 – 65 (18)).
- 2. Fill the glass bottle to the 130 ml level with the soil sample to be tested.
- 3. Add the sodium hydroxide solution until the volume reaches the 200 ml level.
- 4. Close the bottle with a stopper and vigorously shake for a couple of minutes, and then let it stand for 24 hours.
- 5. At the end of the 24-hour standing period, hold the bottle with the test sample and the Gardner Color Standard No.11 side-by-side, and compare the color of the supernatant liquid above the sample with the organic plate No. 1 to 5 (Gardner Color Standard No.11). Note that it is very critical to read the color after 24 hours since some soils show a light color few hours after adding the NaOH solution and darker after 24 hours (see Figure 1-5).



Figure 1-5: Colorimetric test at different times after adding the NaOH solution

f. Report

Report the organic plate number which is closest to the color of the supernatant. The color depends on the presence of organic matter. Specifically, according to the standard, "if the color of the supernatant liquid is darker than that of the standard color of solution or the glass color standard organic plate No. 3 (Gardner Color Standard No.11), the fine aggregate under test shall be considered to possibly contain injurious organic impurities".

1.3.1.3. Liquid limit ratio determination

a. References

- 1. Standard test methods for liquid limit, plastic limit, and plasticity index of soils (ASTM D4318 10) (19)
- Standard practice for classification of soils for engineering purposes
 (Unified Soil Classification System) (ASTM D2487 10) (11)
- 3. Standard method of test for determining the liquid limit of soils (AASHTO T89 10) (20)
- Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009) (14)

b. Scope and Summary

The method is based on the use of the liquid limit test to obtain a qualitative measure of the organic matter content of a soil. This can be obtained by comparing the liquid limit of a sample before and after oven-drying. The described method follows ASTM standard D4318 – 10 (19). The only deviation from the standard is the order of performing the determination of the blow counts at various water contents: while the standard suggests a dry to wet procedure (i.e. water is added to the soil before each blow count determination), a wet to dry procedure (using a fan to dry the soil) is instead recommended. It is acknowledged that the two procedures may cause slight differences in the results of liquid limit; however, the use of the latter procedure is reported to generate more repeatable data (14).

c. Background

The liquid limit of a soil is the water content at which the soil passes from a plastic to a liquid state. It is used for soil classification. In addition, a soil containing substantial amounts of organic matter shows a dramatic decrease in the liquid limit when oven-dried before testing. Therefore, a qualitative measure of organic content of a soil can be obtained by comparing the oven-dried liquid limit with the not oven-dried liquid limit. If the ratio (also known as liquid limit ratio) is less than 0.75, the soil is classified as an organic soil.

d. Apparatus

1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.

- 2. Casagrande liquid limit device.
- 3. Flat grooving tool.
- 4. Scale of 0.01g readability.
- 5. Aluminum tares for water content determination.
- 6. Desiccator.
- 7. Mixing bowl.
- 8. US standard sieve No. 40 (425 μm).

e. Procedure

- Adjust the height of drop for the Casagrande liquid limit device to 10mm ± 0.2mm (vertical distance between the base and the point on the cup that comes in contact with the base).
- 2. Check the resilience rebound of the apparatus base by dropping a 7.94mm (5/16 in) diameter steel ball on the base from a height of 254mm (10 in). The ratio of the rebound height to the drop height should be between 77% and 90%.
- 3. Sieve soil through US No. 40 sieve and obtain natural water content (never oven dry soil prior to tests).
- 4. Mix about 100 g of soil with distilled water to about 15 drop consistency, cover to prevent loss of moisture and place it in a humid room for 24 hours to temper.
- 5. Place soil in the Casagrande cup to a maximum depth of ½ inch. The soil should form a flat horizontal surface with the bottom lip of the cup. This can be checked by filling the cup on the strike position with water. Ensure that entrapped air is removed and that the flat surface is smooth.
- 6. Groove the soil with the flat grooving tool maintaining the tool perpendicular to the surface of the cup throughout its movement.

7. Lift and drop the cup by turning the crank at a rate of 2 blows/second until the groove closes for a length of 13 mm (½ inch) and record the number of blows.

- 8. Remove soil from cup and return to the dish. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.
- 9. Mix soil in a dish and repeat steps 5, 6, 7 and 8 until two consistent blow counts (± 1) are measured.
- 10. Remove about 10 g of paste perpendicular and across the closed groove, place in a tare of known mass and put it in the oven (110°C ± 5°C) for water content measurements.
- 11. Obtain four separate water content determinations between 15 and 35 blows by drying the soil slightly and repeating steps 5 through 10.
- 12. Plot the water content against log of number of blows, draw the flow curve and select the liquid limit as the intersection of this curve and the 25 blow line.
- 13. Prepare another soil sample by working the material through the US No. 40 sieve and oven-dry it for 24 hours at 110°C ± 5°C (or until no mass loss is observed).
- 14. Repeat steps 4 through 12 and determine the oven-dried liquid limit.

f. Calculation

The liquid limit ratio is computed as follows:

$$LL_{ratio} = \frac{LL_{oven dried}}{LL_{not dried}}$$

Where:

LL_{ratio} = Liquid limit ratio

LL_{oven dried} = Liquid limit for oven-dried soil

LL_{not dried} = Liquid limit for not oven-dried soil

g. Report

Report the average liquid limits before and after oven-drying along with the standard deviation. Also, provide the liquid limit ratio and note whether the specimen is an organic or inorganic soil.

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: □ Yes ■ No

Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.32	1.30	1.33	1.31
Mass _{tare + wet soil} M _{tws} (g)	4.90	4.95	4.91	5.60
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)	3.57	3.63	3.62	4.07
Water content w (%)	58.9	56.7	56.2	55.6
Number of blows N	14	25	29	34

Liquid Limit = _	56.7%
------------------	-------

Observations:			

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: ■ Yes □ No

Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.30	1.34	1.30	1.33
Mass _{tare + wet soil} M _{tws} (g)	5.34	4.89	6.48	6.05
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)	4.03	3.77	4.86	4.59
Water content w (%)	47.9	46.4	45.5	44.9
Number of blows N	16	22	28	33

Liquid Limit =	46.0%
----------------	-------

Observations:			

Soil Sample: Location: Boring No: Sample No: Sample Depth: Oven-dried:	Date: Time: Tested by: Description:			_
Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)				
Mass _{tare + wet soil} M _{tws} (g)				
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)				
Water content w (%) Number of blows N				
Liquid Limit =				
Observations:				

1.3.2. Indirect method for determination of organic content

a. Basic principle

An indirect method for the determination of organic content is based on measuring the concentration of total organic carbon (TOC) in soils. For any soil sample the organic content can be calculated by multiplying the total organic carbon content by a factor that reflects the carbon content of the soil's organic matter, which typically ranges between 48% and 58% (by weight) (21). Thus, in principle, the correction factor is soil and horizon specific. In practice, a correction factor of 1.724 (based on the assumption that organic matter contains 58% organic C) has been traditionally used (22). As shown in the equation below, this factor is used to estimate the true organic content.

$$OC (\%) = 1.724 \times C_{organic} (\%)$$

The total organic carbon (TOC) is determined by conducting the dry combustion test and the loss of carbon dioxide test as summarized in the next two subsections.

b. Total carbon content (Dry combustion test)

Dry combustion is considered to be the most reliable and accurate measurement of the total carbon content of a soil (22). The test consists in oxidizing organic carbon and thermally decomposing other carbonate minerals at high temperature (~950°C) in a resistance furnace. The total carbon content is then obtained through measurement of the CO₂ released from the elemental carbon. If there is no inorganic carbon, the total carbon provided by the dry combustion test is equal to the total organic carbon (TOC) of the soil. The potential presence of inorganic carbon can be assessed by pre-testing all soil samples by adding drops of a 3M hydrochloric acid (HCI) solution to a small soil sub-sample. If strong froth is observed, it is concluded that the soil contains inorganic carbon (e.g. calcite (CaCO₃) and/or dolomite (CaMg(CO₃)₂)), and an independent measure of the inorganic carbon content should be conducted using the procedure described in the following subsection. The total organic carbon content is then determined as the difference between the total carbon content given by the dry combustion test and the inorganic carbon content.

c. Inorganic carbon content (Loss of carbon dioxide - Gravimetric method)

This test is used to determine the inorganic carbon content of soils. The test consists of adding hydrochloric acid (HCI) to a soil sample and measuring the decrease in mass resulting from the release of CO₂ that is produced. Given that the release of CO₂ to the atmosphere is proportional to the carbonate content of the soil (23), the latter can then be determined from the measured CO₂. A soil sample of about 1g is placed in a flask with 10 ml of 3M hydrochloric acid (HCI), and measurements of the mass of the flask are conducted every 15 minutes until the change in mass is less than 1-2 mg. The carbon content can then be calculated from the following:

$$C (\%) = [CO_{2 \text{ lost }}(g) / Soil (g)] \times 0.2727 \times 100$$

1.3.3. Supporting classification examples

This section contains examples of the classification of soils containing organic matter based on the system and testing procedures proposed in CHAPTER 1. The table below summarizes the following: liquid limit, plastic limit, plasticity index, loss on ignition, results of colorimetric test, liquid limit ratio, and the corresponding classification. Figure 1-6 to Figure 1-9 present the classification of four different soil samples based on the results of LOI, colorimetric test and liquid limit ratio.

Table 1-3: Supporting classification examples for organic soils

Soil ID	LL	PL	PI	LOI	Colorimetric Test	Liquid Limit Ratio	Classification
	%	%	%	%	Color No.	=	%
Soil 1	49.0	27.2	21.8	3.2	1	0.96	Mineral soil*
Soil 4	46.7	35.0	11.6	5.2	3	0.88	Mineral soil*
Soil 5	32.1	20.1	12.0	1.2	1+	0.98	Mineral soil*
Soil 7	47.7	38.6	9.1	10.9	5	0.68	Mineral soil* with organic matter
Soil 9	34.0	14.7	19.3	4.5	4	1.00	Mineral soil*
Soil 11	151.1	123.0	28.1	43.2	-	0.58	Peat

^{* &}quot;Mineral soils" designated through USCS/AASHTO terminology (e.g. A-5)

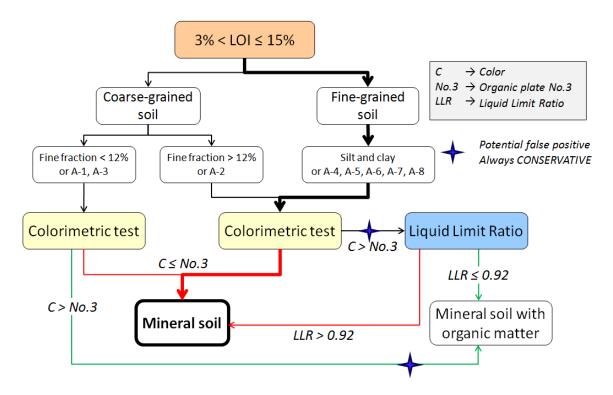


Figure 1-6: Supporting classification example - Soil 1

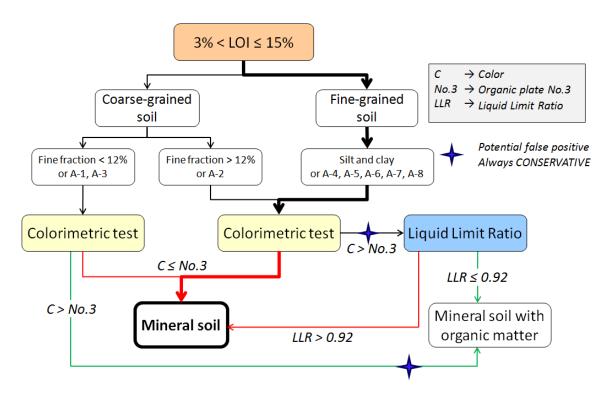


Figure 1-7: Supporting classification example - Soil 4

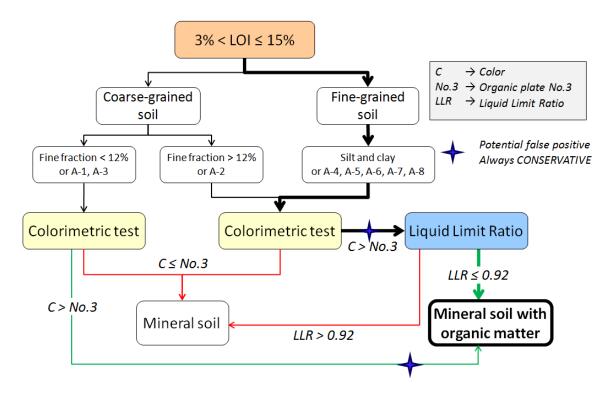


Figure 1-8: Supporting classification example - Soil 7

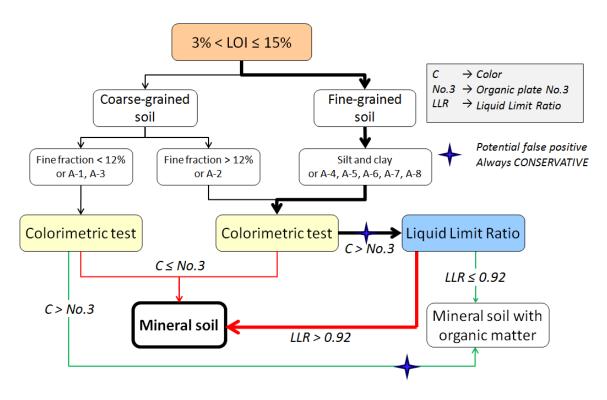


Figure 1-9: Supporting classification example - Soil 9

1.4. References

- 1. Chau, P.P. "Definition of muck." M.S.Thesis, Mississippi State University, Department of Civil Engineering, Mississippi State, MS, 1999.
- 2. INDOT. Geotechnical Manual. Indiana Department of Transportation, Indianapolis, 2008.
- 3. Lea, N.D. and C.O. Brawner. "Foundation and Pavement Design for Highway on Peat," Proc. Fortieth Convention, Can. Good Roads Association, Ottawa, 1959, pp. 406-424.
- 4. MacFarlane, I.C. (Ed.). *Muskeg Engineering Handbook*, University of Toronto Press, 1969.
- 5. Mesri, G, T.D. Stark, M.A. Ailouni, and C.S. Chen. "Secondary Compression of Peat with or without Surcharge," *Journal of Geotechnical and Geoenvironmental Engineering*, Vol. 123, No. 5, 1997, pp.411-421.
- 6. Mesri, G, and P.M. Godlewski. "Time and Stress-compressibility Interrelationship," *Journal of Geotechnical Engineering*, ASCE Vol. 103, No.5, 1977, pp.417-430.
- Mesri, G., T.D. Stark, and C.S. Chen. "Discussion of Cα/Cc Concept Applied to Compression of Peat", *Journal of Geotechnical Engineering*, ASCE Vol. 120, No.4, 1994, pp.764-767.
- 8. Christensen, B. T., and P. A. Malmros. "Loss-on-Ignition and Carbon Content in a Beech Forest Soil-Profile," Holarctic Ecology, 5(4), 1982, 376-380.
- 9. Howard, P. J. A., and D. M. Howard. "Use of Organic-Carbon and Loss-on-Ignition to Estimate Soil Organic-Matter in Different Soil Types and Horizons," Biology and Fertility of Soils, 9(4), 1990, 306-310.
- 10. Huang, P.T., M.C. Santagata, and A. Bobet. *Classification of Organic Soils*. Report FHWA/IN/JTRP-2009/ for the Joint Transportation Research Program, 2009.

11. ASTM Standard D2487-10. Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System), *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2010.

- 12. ASTM Standard D2974-07a. Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils. Annual book of ASTM Standards, ASTM international, West Conshohocken, PA, 2007.
- 13. AASHTO T267-86. Standard Method of Test for Determination of Organic Content in Soils by Loss on Ignition. AASHTO, 2004.
- 14. Germaine, J.T., and A.V. Germaine. *Geotechnical Laboratory Measurements for Engineers*, John Wiley & Sons, Inc., Hoboken, NJ, 2009.
- 15. ASTM Standard C40-04. Standard Test Methods for Organic Impurities in Fine aggregates for Concrete, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2004.
- ASTM Standard D1544-04. Standard Test Methods for Color of Transparent Fluids (Gardner Color Scale), Annual Book of ASTM Standards, ASTM International, West Conshohocken, PA, 2010
- 17. AASHTO Standard T21-05. Standard Test Methods for Organic Impurities in Fine aggregates for Concrete, American Association of State Highway and Transportation Officials, Washington D.C., 2005.
- 18. AASHTO Standard M147-65. Standard Specification for Materials for Aggregate and Soil-Aggregate Subbase, Base, and Surface Courses, American Association of State Highway and Transportation Officials, Washington D.C., 2004.
- 19. ASTM Standard D4318-10. Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2010.

 AASHTO Standard T89-10. Standard method of test for determining the liquid limit of soils, American Association of State Highway and Transportation Officials, Washington D.C., 2010.

- 21. Nelson, D.W. and L.E. Sommers. "Total Carbon, Organic Carbon, and Organic Matter," Methods of Soil Analysis Part 3. Chemical Methods Soil Science Society of America, 1996, pp. 961-1010.
- 22. Schumacher, B.A. "Methods for the Determination of Total Organic Carbon (TOC) in Soils and Sediments", Ecological Risk Assessment Support Center, Office of Research and Development, U.S. Environmental Protection Agency Report, 2002.
- 23. Loeppert, R.H. and D.L. Suarez. "Carbonate and Gypsum", *Methods of Soil Analysis: Part 3- Chemical Methods,* Soil Science Society of America, 1996, pp. 437-456.

CHAPTER 2. IDENTIFICATION AND CLASSIFICATION OF MARLY SOILS

2.1. Background

2.1.1. Importance of identifying marly soils

Marl soil deposits are encountered in the Midwest of the US, including the states of Indiana, Illinois, Michigan, and Ohio (1, 2, 3, and 4). The term marl has been used in the regional area to designate carbonate-rich, light gray to almost white silts and clays formed by precipitation of calcite at the bottom of lakes or swamps (1, 2, and 3). Marl soils sometimes contain noticeable amounts of fine sand (3). Marl deposits are encountered often below highly organic soil or peat deposits (1) and contain shell fragments (3). Marls are classified as an organic soil in accordance with the Ohio DOT soil classification system (4). According to the Indiana DOT soil classification system, a soil with a calcium carbonate content of 26% to 40% is classified as marly soil while a soil with a calcium carbonate content larger than 40% is classified as marl (2). One of the tests that the Indiana DOT uses to determine the calcium carbonate content in a soil is the chemical test, following ASTM C25. Both marly soils and marls fall into the ASSHTO soil class A-8 (2). Marl soils typically have low dry density, very high moisture content and low shear strength. This makes them "problem soils" and unsuitable for pavement subgrade, may be prone to slope instability and have low bearing capacity.

2.1.2. Effect of CaCO₃ on geotechnical properties of soils

The carbonate content of a soil affects its geotechnical engineering properties. It affects index properties (5, 6), the residual frictional angle of the soil (7), the general stress-strain response (8, 9, 10, and 11), and clay expansivity (12). This section summarizes the effect of CaCO₃ on some of these properties:

 Atterberg limits: In general both liquid limit and plastic limit decrease with carbonate content. In other words, as the carbonate content increases, marl soils tend to show less plastic behavior.

- 2. pH: As the CaCO₃ content of the soil increases, the pH tends to increase.
- 3. <u>Color</u>: As the percentage of calcium carbonate in the soil increases, the color of the soil changes from brown to light-gray, almost white.
- 4. <u>Cohesion</u>: With increasing carbonate content, the cohesion of the soil decreases.
- 5. <u>Permeability</u>: the permeability of a soil increases with the calcium carbonate content.

In summary, with increasing carbonate content, the LL, PL, PI, and activity of the soil decrease, the pH, permeability and friction angle increase while cohesion decreases. This trend however is applicable only for soils with no organic content. As discussed in section 1.1.2, the presence of organic matter strongly affects the soil properties and in some cases shows the opposite trend (e.g. Atterberg limits). Therefore, it can be concluded that the soil indices depend, to a large extent, on the CaCO₃ content when the soil does not contain any organic matter, but this trend becomes much weaker when the soil contains organic matter because the organic matter also significantly affects the soil indices. As a result, the geotechnical characteristics of marl soils depend on both organic content and CaCO₃ content.

2.2. New Classification System

Marl soils are usually categorized using classifications systems developed for clays and silts such as USCS (Unified Soil Classification System) and AASHTO. However, it may not be always appropriate to classify marl soils based only on their particle size distribution and consistency (7). The index properties of marls depend on the carbonate content and on the type and content of minerals in the clay (5). The research conducted as part of SPR-3227 re-endorsed the classification previously used by INDOT (2) that classifies soils into five groups based on the % of CaCO₃. Table 2-1

summarizes the five different categories for classification: soil with trace of marl, soil with little marl, soil with some marl, marly soil, and marl. It is recommended to use the "sequential" LOI for the determination of CaCO₃ (13). Note that this classification system is applicable only to fine-grained soils.

Classification	Calcium Carbonate Content (%)
Soil* with trace of marl	1% < CaCO ₃ < 9%
Soil* with little marl	10% < CaCO ₃ < 17%
Soil* with some marl	18% < CaCO ₃ < 25%
Marly soil*	26% < CaCO ₃ < 40%
Marl	CaCO ₃ > 40%

Table 2-1: Criteria of marly soils classification (13)

2.2.1. Motivation

The initiative for the work conducted as part of SPR-3227 came from the need of INDOT to have a workable classification system and accurate and yet economical laboratory tests to determine the percentage of calcium carbonate in soils. INDOT performs chemical tests in accordance with ASTM C25 to determine the calcium carbonate content in a soil. The chemical test is not easy to perform; as a result, it is not routinely performed by most geotechnical engineering companies in Indiana. It is important however to determine the CaCO₃ content in a soil since this is a parameter that may be needed to decide if the soil can be accepted for construction.

An experimental investigation was carried out by Jung et al. (13) to propose a simple, practical method, to identify and classify marl soils in the laboratory. The percentage of calcium carbonate (CaCO₃) of the soil was determined with three different methods: 1) TGA (Thermo-Gravimetric Analysis); 2) "sequential" LOI (Loss on Ignition); and 3) chemical reaction following ASTM C25. The authors validated the use of any of these three methods (with the sequential LOI having the advantage that both organic and calcium carbonate content of the soil can be determined using a conventional furnace).

^{* &}quot;soil" designated through USCS/AASHTO terminology

2.2.2. Classification system and screening approach

Laboratory:

The geotechnical engineering properties of marl soils depend on organic content and CaCO₃ content; therefore the soils should be classified in terms of both these parameters. Figure 2-1 summarizes the soil classification in terms of organic and calcium carbonate content in form of a flow chart based on the sequential LOI. In terms of organics, the soil is classified based on the organic classification system and the methods presented in section 1.2, whereas for calcium carbonate, the soil is classified based on classification system summarized in Table 2-1. Note that if the soil falls under the "mineral" category based on organic content, it is classified based on CaCO₃ content only. Otherwise, a dual classification is used (i.e. marly soil and mineral soil with organic matter).

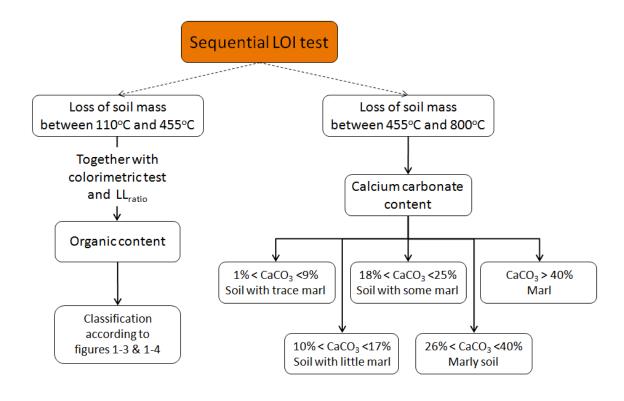


Figure 2-1: Approach for classifying soils in terms of calcium carbonate content based on sequential LOI test (13)

Field:

The color of the soil and its reaction with a 1M HCl solution may be used for a simple field classification. If a soil has a light gray color when dry, the soil can be potentially classified as marly soil or marl. If the soil has a different color, then the CaCO₃ content of the soil might be less than 20%.

The color determination must be complemented by a chemical test where a few drops of a 1M HCl solution are mixed with the soil. If effervescence is observed, this is an indication that the soil has a CaCO₃ content of at least 20%. The soil then can be classified as marl soil or marl. If no reaction is detected, the calcium carbonate content in the soil is smaller than 20%. A more precise determination of the CaCO₃ content, if needed, can be achieved by the sequential LOI test in the laboratory. Figure 2-2 is a schematic of the recommended field classification process using HCl reaction with the soil.

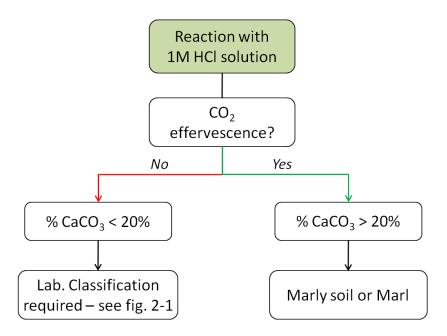


Figure 2-2: Recommended field classification procedure for marly soils (13)

2.3. Appendices

2.3.1. Procedures

2.3.1.1. "Sequential" loss on ignition Test (LOI)

a. References

- 1. Standard test methods for moisture, ash, and organic matter of peat and other organic soils (ASTM D2974 07a) (14)
- 2. Standard method of test for determination of organic content in soils by loss on ignition (AASHTO T267 86) (15)
- 3. Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009) (16)
- 4. Determination of calcium carbonate content in soils using sequential loss on ignition test (ITM 507) (17)
- 5. Classification of marl soils (13)

b. Scope and Summary

This test method covers the procedure to determine the percentage of calcium carbonate (%CaCO₃) in soils using sequential LOI test (17). The measurement is based on the fact that calcium carbonate decomposes into calcium oxide (CaO) and carbon dioxide (CO₂) in the range of 650°C to 800°C. The reduction in mass due to the release of CO₂ can be used to infer the calcium carbonate content.

c. Background

The loss on ignition (LOI) test can be used to determine the organic content and calcium carbonate content in the soil. In geotechnical engineering LOI tests have been used to measure organic content, heating the soil up to 455 °C, in accordance with AASHTO T267 – 86. Jung et al. (13) extended the LOI test in an attempt to determine

the calcium carbonate content in the soil, and as a simpler alternative to the chemical tests (discussed later).

d. Apparatus

- 1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.
- 2. Muffle furnace capable of attaining and maintaining a constant temperature of $800^{\circ}\text{C} \pm 10^{\circ}\text{C}$.
- 3. Scale of 0.01g readability.
- 4. Porcelain crucibles that can be heated up to 800°C.
- 5. Desiccator.
- 6. US standard sieve No. 10 (2 mm).

e. Procedure

- Determine the mass of the porcelain crucible (M_c) to the nearest 0.01g.
 Note that each crucible should be washed, marked with a permanent
 paint and heated at the test temperature before it is used to perform
 any testing.
- 2. Obtain a representative soil specimen of 10g to 15g and sieve it through the No. 10 sieve (2 mm) (15).
- 3. Place the soil sample in the crucible and determine the mass (M_{cws}) to the nearest 0.01g.
- 4. Oven-dry the specimen at 110°C ± 5°C for 24 hours (or until no mass loss is observed).
- Remove the crucible and contents from the oven and place it in a desiccator to cool (~10 minutes).
- 6. Determine the dry mass $(M_{110^{\circ}C})$ to the nearest 0.01g.
- 7. Place the crucible and contents in a muffle furnace at 455°C for 6 hours.
- 8. Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).

9. Determine the mass of the crucible with the ash $(M_{455^{\circ}C})$ to the nearest 0.01g.

- 10. Place the crucible and the soil into the furnace for 6 additional hours at a temperature of 800°C.
- 11. Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).
- 12. Determine the mass of the crucible with the burnt soil ($M_{800^{\circ}C}$) to the nearest 0.01g.

f. Calculation

The loss on ignition is computed as follows: (Refer to CHAPTER 1 for classification of organic soils)

$$LOI = \frac{M_{110^{\circ}C} - M_{455^{\circ}C}}{M_{110^{\circ}C} - M_{C}} \times 100$$

The CaCO₃ content is computed as follows:

CaCO₃ =
$$\frac{100}{44} \times \frac{M_{455^{\circ}C} - M_{800^{\circ}C}}{M_{110^{\circ}C} - M_{c}} \times 100$$

Where:

LOI = loss on ignition of soil (%)

 $M_{110^{\circ}C}$ = mass of crucible and soil at 110°C (g)

 $M_{455^{\circ}C}$ = mass of crucible and ash at 455°C (g)

 $M_{800^{\circ}C}$ = mass of crucible and burnt soil at 800°C (g)

 M_c = mass of crucible (g)

g. Report

Report the organic content and the percentage of calcium carbonate to the nearest 0.1% together with the temperatures of the muffle furnace. If more than one specimen is tested report the averages and the standard deviation.

Classify the soil based on both OC and CaCO₃ contents. If the soil falls under mineral" category based on organic content, it is classified based on CaCO₃ content only. Otherwise, dual classification shall be used (i.e. Marly soil and mineral soil with organic matter).

SEQUENTIAL LOSS ON IGNITION (LOI) – Sample Sheet

Soil Sample: Soil II Date: Sat 9/17/2011

Location: I69-sec3 Seg13 Time: 11:45am

Boring No: 3-37-TB-1 Tested by: AH

Sample No: Description: Dark Gray – Clayey

Sample Depth: 24 to 26ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2		
Crucible No.	CE1	2		
Mass _{crucible empty} M _c (g)	19.82	17.71		
Mass _{crucible + wet soil} M _{cws} (g)	35.53	33.23		
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)	30.03	27.80		
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.81	27.58		
Mass _{crucible} + burnt soil (@ 800°C) M _{800°C} (g)	27.24	25.08		
Loss on ignition LOI (%)	2.1	2.2		
CaCO ₃ content (%)	57.4	56.4		

Average Loss on i	• ,	2.1 56.9	
Observations: _			

Soil Sample: Location: Boring No: Sample No: Sample Depth:		-			
Test No.	1	2	3	4	5
Crucible No.	Α	В	С	D	F
Mass _{crucible empty} M _c (g)					
Mass _{crucible + wet soil} M _{cws} (g)					
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)					
Mass _{crucible + Ash} (@ 455°C) M _{455°C} (g)					
Mass _{crucible + burnt soil} (@ 800°C) M _{800°C} (g)					
Loss on ignition LOI (%)					
CaCO ₃ content (%)					
Average Loss on ignition (%) Average CaCO ₃ content (%) Observations:			-		

2.3.1.2. HCl reaction test

According to Soil Taxonomy (18), marl soils should react with dilute hydrochloric acid (HCl) to produce carbon dioxide (CO₂). Also, both standards, ASTM D4373 (19) and ASTM C25 (20), use 1.0 M HCl solution to neutralize the calcium carbonate in the soil. As a consequence, 1.0 M HCl solution can be used to detect the calcium carbonate in the soil by observing the effervescence (bubbling effect) that occurs with the production of CO₂.

2.3.2. Indirect method for determination of CaCO₃ content

2.3.2.1. Chemical determination of CaCO₃ content

The chemical tests follow ASTM C25 (20), which specifies a procedure to determine the neutralizing capacity of a calcareous material. About two grams of soil are placed into a 500-ml Erlenmeyer flask. 25 ml of 1.0 M hydrochloric acid (HCl) solution is added into the flask. About five minutes after the addition of the 1.0 M HCl solution the excess acid in the flask is titrated with 0.5 M sodium hydroxide (NaOH) solution using phenolphthalein as indicator. The volume of NaOH solution required for the titration of the excess acid is measured. The calcium carbonate content in the soil is:

% CaCO₃ =
$$\frac{5.0045(V_1N_1-V_2N_2)}{W} \times 100$$

Where

 V_1 = volume of the HCl solution used in ml

 N_1 = normality of the HCl solution

 V_2 = volume of the NaOH solution required for titration of excess acid in ml

 N_2 = normality of the NaOH solution

W = weight of the soil sample in grams

Note that the value obtained with the above equation is not the percentage of calcium carbonate (CaCO₃), but the percentage of calcium carbonate equivalent (C.C.E.). This is so because other carbonate species such as magnesite and dolomite as well as calcite (CaCO₃) can react chemically with the 1M HCl solution. In other words, the chemical test describes the amount of all carbonate species in terms of C.C.E.

2.3.2.2. Thermogravimetric analysis (TGA)

a. Basic principle

Thermogravimetric analysis (TGA) is a thermal analysis technique used to quantify the weight loss of materials with increasing temperature. The standard testing procedure for this test is contained in ASTM E1131 (21). A soil sample, typically 40mg in mass, is placed in a chamber, which, starting from room temperature, is heated to the desired temperature. The rate at which temperature is increased is typically 10-20°C/min, and pure Nitrogen is supplied at a rate of 50 ml/min. Different minerals decompose at well-defined temperatures. In the range of 650°C to 800°C, calcium carbonate (CaCO₃) decomposes into calcium oxide (CaO) and carbon dioxide (CO₂). As a consequence, the calcium carbonate content in a soil sample is determined from the weight loss of the soil between 650°C and 800°C as shown in the equation below:

$$%CaCO_3 = \frac{100}{44} \times \frac{M_{650} - M_{800}}{M_{110}} \times 100$$

Where

 M_{110} = mass of soil at 110°C (g)

 M_{650} = mass of soil at 650°C (g)

 M_{800} = mass of soil at 800°C (g)

b. Interpretation of data

Figure 2-3 shows an example of a TGA curve obtained from a test conducted by Jung et al. (13), employing a heating rate of 10°C/min. It is observed that the weight of the soil sample has a sharp decrease in the range of temperatures between 650°C and

750°C. This is within the range where CaCO₃ decomposes into CaO and CO₂, and so the weight loss represents the CaCO₃ content. The figure also includes the derivative of the weight loss with respect to time, which shows a clear peak around 740°C.

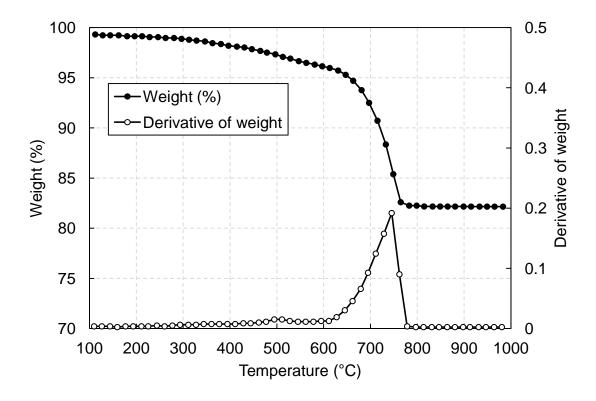


Figure 2-3: Weight loss obtained from TGA test (Jung et al., 2009)

2.3.3. Supporting classification examples

This section contains examples of the classification of marly soils containing CaCO₃ and organic matter based on the system and testing procedure proposed in Chapters 1 and 2. The table below summarizes the following: liquid limit, plasticity index, organic content (sequential LOI), Calcium carbonate content (sequential LOI), results of colorimetric test, liquid limit ratio, and the corresponding classification. Figure 2-4 to Figure 2-8 present the classification of five different soil samples based on the results of sequential LOI, colorimetric test and liquid limit ratio.

Table 2-2: Supp	orting cla	ssification	examples	for marly	√ soils

Soil ID	LL	PI	O.C. (LOI)	CaCO ₃ (LOI)	Colorimetric Test	Liquid Limit Ratio	Classification
_	%	%	%	%	Color No.	-	%
Soil 1-1	41	15	2.5	47	-	-	Marl
Soil 1-9	32	17	1.5	33	-	-	Marly soil*
Soil 2-1	73	23	17.3	41	-	-	Marl & organic soil
Soil 3-2	60	21	6.2*	21	-	-	Soil* with some marl+
Soil 3-4	68	24	15.5	11	-	-	Organic soil with little marl

[†]Organic content is between 3% and 15%. Need to conduct colorimetric test and liquid limit ratio to have a full classification for organics.

^{* &}quot;soil" designated through USCS/AASHTO terminology

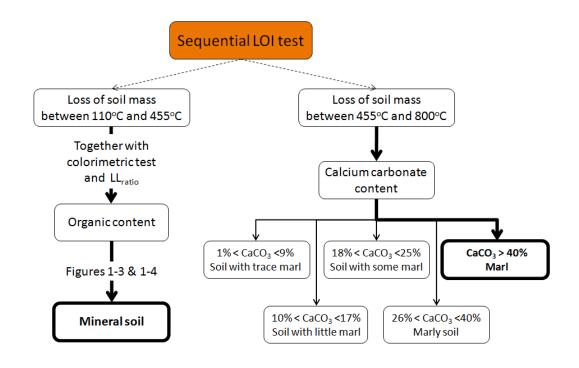


Figure 2-4: Supporting classification example - Soil 1-1

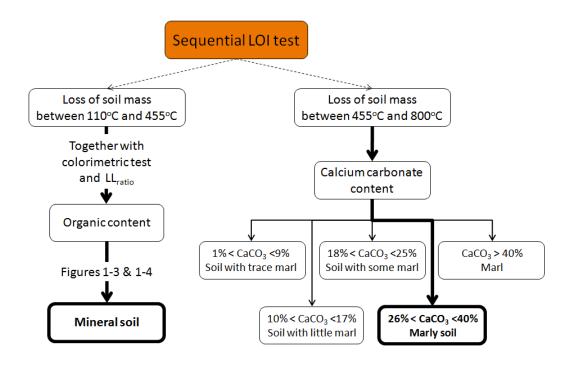


Figure 2-5: Supporting classification example - Soil 1-9

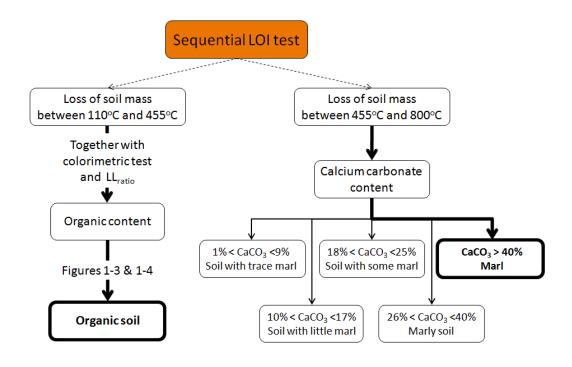


Figure 2-6: Supporting classification example - Soil 2-1

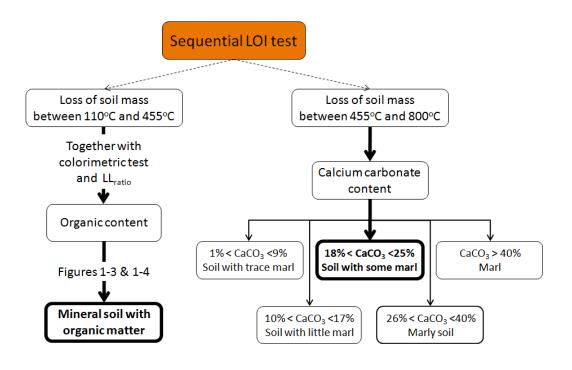


Figure 2-7: Supporting classification example - Soil 3-2

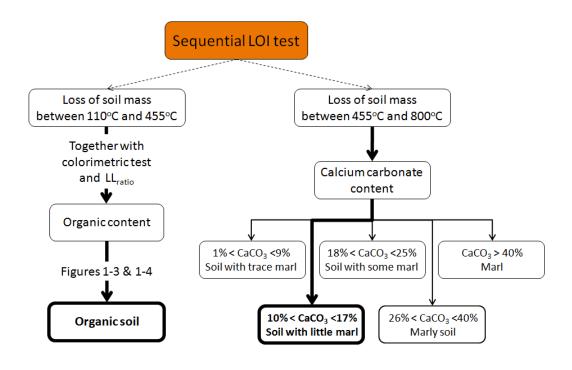


Figure 2-8: Supporting classification example - Soil 3-4

2.4. References

- 1. IDOT. Geotechnical Manual. Illinois Department of Transportation. Springfield, 1999.
- 2. INDOT. Geotechnical Manual. Indiana Department of Transportation. Indianapolis, 2008.
- MDOT. Uniform Field Soil Classification System (Modified Unified Description).
 Michigan Department of Transportation. Lansing, 2009.
- 4. ODOT. Specifications for Geotechnical Explorations. Ohio Department of Transportation. Columbus, 2010.
- 5. El Amrani, N., F. Lamas, C. Irigaray, and J. Chacon. Engineering geological characterization of neogene marls in the southeastern Granada Basin, Spain. *Engineering Geology*, Vol. 50, 1998, pp. 165–175.
- Lamas, F., C. Irigaray, J. Chacón. Geotechnical characterization of carbonate marls for the construction of impermeable dam cores. *Engineering Geology*. Vol. 66, 2002, pp. 283–294.
- 7. Tsiambaos, G. Correlation of mineralogy and index properties with residual strength of Iraklion marls. *Engineering Geology*. Vol. 30, 1991, pp. 357-369.
- 8. Fischer, A.G., S. Honjo, and R.E. Garrison. *Electron Micrography of Limestones and their Nanophosil*. Princeton University press, Princeton, N.J., 1967.
- 9. Mitchell, J.K. Soil Behaviour, vol. 1. Wiley, New York, 1975.
- Demars, K.R. Unique engineering properties and compression behavior of deepsea calcareous sediment. In: Demars, K.R., Chaney (Eds.), Geotechnical Properties Behaviour and Performance of Calcareous Soils, ASTM STP, Vol. 777. ASTM, Philadelphia, 1982, pp. 97–112.

11. Dapena, E. Influencia de la rigidez del material del núcleo de arcilla sobre el comportamiento de una presa de materials sueltos. Simposio Sobre Geotecnia de Presas de Materiales Sueltos. Zaragoza, 1993, pp. 217–226.

- 12. Konrad, J.M. Undrained response of loosely compacted sands during monotonic and cyclic compression tests. Geotechnique, Vol. 43, No. 1, 1993, pp. 69–89.
- 13. Jung, C., A. Bobet, and N. Z. Siddiki. Classification of Marl Soils. Publication FHWA/IN/JTRP-2009. Joint Transportation Research Program, Indiana Department of Transportation and Purdue University, West Lafayette, Indiana, 2009.
- 14. ASTM Standard D2974-07a. Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils. Annual book of ASTM Standards, ASTM international, West Conshohocken, PA., 2007.
- 15. AASHTO T267-86. Standard Method of Test for Determination of Organic Content in Soils by Loss on Ignition. AASHTO, 2004.
- 16. Germaine, J.T., and A.V. Germaine. *Geotechnical Laboratory Measurements for Engineers*, John Wiley & Sons, Inc., Hoboken, NJ, 2009.
- 17. INDOT. Determination of calcium carbonate content in soils using sequential loss on ignition test (ITM 507). Indianapolis, 2012.
- 18. USDA. Soil Taxonomy. United States Department of Agriculture, 1999.
- ASTM Standard D4373-02. Standard Test Method for Rapid Determination of Carbonate Content of Soils. *Annual book of ASTM Standards*, ASTM international, West Conshohocken, PA. 2002.
- 20. ASTM Standard C25-06. Standard Test Method for Chemical Analysis of Lime stone, Quicklime, and Hydrated Lime. *Annual book of ASTM Standards*, ASTM international, West Conshohocken, PA, 2006.

21. ASTM Standard E1131-08. Standard Test Methods for Compositional Analysis by Thermogravimetry, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2008.

IDENTIFICATION AND CLASSIFICATION OF ORGANIC SOILS

STEP 1: LOSS ON IGNITION TEST (LOI)

I- REFERENCES

- 1. ASTM D2974 07a
- 2. AASHTO T267 86
- Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009)

II- SCOPE AND SUMMARY

This test method describes the process to determine the organic content using the Loss on Ignition test (LOI) and is based on finding the reduction in mass of an ovendried specimen subjected to elevated temperature such that organic matter is burnt off.

III- APPARATUS

- 1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.
- 2. Muffle furnace capable of attaining and maintaining a constant temperature of 455°C ± 10°C.
- 3. Scale of 0.01g readability.
- 4. Porcelain crucibles that can be heated up to 455°C.
- 5. Desiccator.
- 6. US standard sieve No. 10 (2 mm).

IV- PROCEDURE

- 1. Determine the mass of the porcelain crucible (M_c) to the nearest 0.01g. Note that each crucible should be washed, marked with a permanent paint and heated at the test temperature before it is used to perform any testing.
- 2. Obtain a representative soil specimen of 10g to 15g, and sieve it through the No. 10 sieve (2 mm).

APPENDIX 3

- 3. Place the soil sample in the crucible and determine the mass (M_{cws}) to the nearest 0.01g.
- 4. Oven-dry the specimen at 110°C ± 5°C for 24 hours (or until no mass loss is observed).
- Remove the crucible and its content from the oven and place it in a desiccator to cool (~10 minutes).
- 6. Determine the dry mass $(M_{110^{\circ}C})$ to the nearest 0.01g.
- 7. Place the crucible and contents in a muffle furnace at 455°C for 6 hours.
- Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).
- 9. Determine the final mass (M_{455°C}) to the nearest 0.01g.

V- CALCULATION

The loss on ignition can be computed as follows:

$$LOI = \frac{M_{110^{\circ} C} - M_{455^{\circ} C}}{M_{110^{\circ} C} - M_{C}} \times 100$$

Where:

LOI = loss on ignition of soil (%)

 $M_{110^{\circ}C}$ = mass of crucible and soil at 110°C (g)

 $M_{455^{\circ}C}$ = mass of crucible and ash at 455°C (g)

M_c = mass of crucible (g)

VI- REPORT

Report the loss on ignition to the nearest 0.1% together with the temperature of the muffle furnace. If more than one specimen is tested report the average and the standard deviation.

LOSS ON IGNITION (LOI) - Sample Sheet

Soil Sample: Soil I	Date:	Sat 10/22/2011
---------------------	-------	----------------

Location: I69-Sec3 Seg13 Time: 11:00am

Boring No: 3-31-TB-2A Tested by: AH

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2	3	4	5
Crucible No.	А	В	С	D	F
Mass _{crucible empty} M _c (g)	17.67	17.94			
Mass _{crucible + wet soil} M _{cws} (g)	34.29	32.80			
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)	30.02	28.44			
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.20	27.47			
Loss on Ignition LOI (%)	6.6	9.3			

Average Loss on Ignition LOI (%)	8.0
Observations:	

STEP 2: COLORIMETRIC TEST

I- REFERENCES

- 1. ASTM C40 04
- 2. ASTM D1544 04
- 3. AASHTO T21 05
- 4. AASHTO M147 65

II- SCOPE AND SUMMARY

This test covers the procedure for a colorimetric test that detects the presence of organic impurities in fine aggregates for concrete. The test method consists of mixing the soil specimen with a sodium hydroxide solution and observing the color produced. If it is darker than a standard color (Gardner Color Standard No. 11), organic impurities may be present.

III- APPARATUS

- 1. Transparent graduated glass bottles with a minimum capacity of 250 ml.
- 2. A 3% sodium hydroxide solution NaOH (dissolve 3g of NaOH in 97g of water)
- 3. Glass color standard (Gardner color Standard No. 11).
- 4. US standard sieve No. 10 (2 mm).

IV- PROCEDURE

- 1. Air-dry the entire soil sample in a pan and, when necessary, crush it so that it passes the No.10 sieve (2 mm).
- 2. Fill the glass bottle to the 130 ml level with the soil sample to be tested.
- 3. Add the sodium hydroxide solution until the volume reaches the 200 ml level.
- 4. Close the bottle with a stopper and vigorously shake for a couple of minutes, and then let it stand for 24 hours.
- 5. At the end of the 24-hour standing period, hold the bottle with the test sample and the Gardner Color Standard No.11 side-by-side, and compare the color

APPENDIX 3

of the supernatant liquid above the sample with the organic plate No. 1 to 5 (Gardner Color Standard No.11).

V- REPORT

Report the organic plate number which is closest to the color of the supernatant. The color depends on the presence of organic matter. Specifically, according to the standard, "if the color of the supernatant liquid is darker than that of the standard color of solution or the glass color standard organic plate No. 3 (Gardner Color Standard No.11), the fine aggregate under test shall be considered to possibly contain injurious organic impurities".

STEP 3: LIQUID LIMIT RATIO DETERMINATION

I- REFERENCES

- 1. ASTM D4318 10
- 2. ASTM D2487 11
- 3. AASHTO T89 10
- Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009)

II- SCOPE AND SUMMARY

The method uses the liquid limit test to obtain a qualitative measure of the organic matter content of a soil. The procedure is based on the comparison of the liquid limit of a sample before and after oven-drying (also known as liquid limit ratio).

III- APPARATUS

- 1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.
- 2. Casagrande liquid limit device.
- 3. Flat grooving tool.
- 4. Scale of 0.01g readability.
- 5. Aluminum tares for water content determination.
- 6. Desiccator.
- 7. Mixing bowl.
- 8. US standard sieve No. 40 (425 μm).

IV- PROCEDURE

- Adjust the height of drop for the Casagrande liquid limit device to 10mm ± 0.2mm (vertical distance between the base and the point on the cup that comes in contact with the base).
- 2. Check the resilience rebound of the apparatus base by dropping a 7.94mm (5/16 in) diameter steel ball on the base from a height of 254mm (10 in). The

- ratio of the rebound height to the drop height should be between 77% and 90%.
- 3. Sieve soil through US No. 40 sieve and obtain natural water content (never oven dry soil prior to tests).
- Mix about 100 g of soil with distilled water to about 15 drop consistency, cover to prevent loss of moisture and place it in a humid room for 24 hours to temper.
- 5. Place soil in the Casagrande cup to a maximum depth of ½ inch. The soil should form a flat horizontal surface with the bottom lip of the cup. Ensure that entrapped air is removed and that the flat surface is smooth.
- 6. Groove the soil with the flat grooving tool maintaining the tool perpendicular to the surface of the cup throughout its movement.
- 7. Lift and drop the cup by turning the crank at a rate of 2 blows/second until the groove closes for a length of 13 mm (½ inch) and record the number of blows.
- 8. Remove soil from cup and return to the dish. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.
- 9. Mix soil in a dish and repeat steps 5, 6, 7 and 8 until two consistent blow counts (± 1) are measured.
- 10. Remove about 10 g of paste perpendicular and across the closed groove, place in a tare of known mass and put it in the oven (110°C ± 5°C) for water content measurements.
- 11. Obtain four separate water content determinations between 15 and 35 blows by drying the soil slightly and repeating steps 5 through 10.
- 12. Plot the water content against log of number of blows, draw the flow curve and select the liquid limit as the intersection of this curve and the 25 blow line.
- 13. Prepare another soil sample by working the material through the US No. 40 sieve and oven-dry it for 24 hours at 110°C ± 5°C (or until no mass loss is observed).
- 14. Repeat steps 4 through 12 and determine the oven-dried liquid limit.

V- CALCULATION

The liquid limit ratio can be computed as follows:

$$LL_{ratio} = \frac{LL_{oven dried}}{LL_{not dried}}$$

Where:

LL_{ratio} = Liquid limit ratio

 $LL_{oven dried}$ = Liquid limit for oven-dried soil

 $LL_{not dried}$ = Liquid limit for not oven-dried soil

VI- REPORT

Report the average liquid limits before and after oven-drying along with the standard deviation. Also, provide the liquid limit ratio and note whether the specimen is an organic or inorganic soil.

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: ☐ Yes ■ No

Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.32	1.30	1.33	1.31
Mass _{tare + wet soil} M _{tws} (g)	4.90	4.95	4.91	5.60
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)	3.57	3.63	3.62	4.07
Water content w (%)	58.9	56.7	56.2	55.6
Number of blows N	14	25	29	34

Liquid Limit =56.7%

Observations:

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: ■ Yes □ No

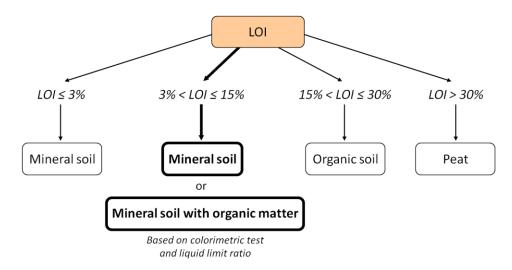
Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.30	1.34	1.30	1.33
Mass _{tare + wet soil} M _{tws} (g)	5.34	4.89	6.48	6.05
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)	4.03	3.77	4.86	4.59
Water content w (%)	47.9	46.4	45.5	44.9
Number of blows N	16	22	28	33

Liquid Limit =	46.0%
----------------	-------

Observations:

Supporting Classification example:

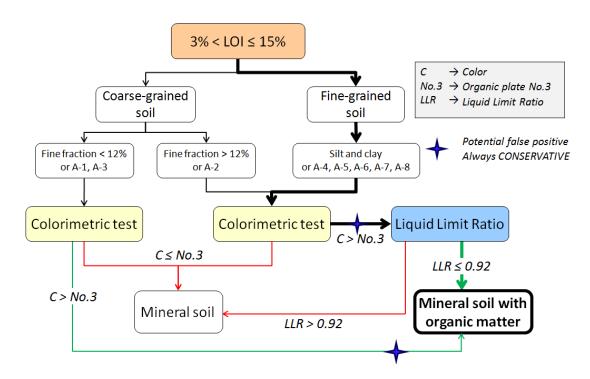
Step 1: LOI = 8.0 % (between 3% and 15%)



Fine-grained Soil (more than 35% passing sieve no. 200)

Step 2: Color = organic plate No. 5 (> No. 3)

<u>Step 3</u>: LLR = 0.81 (< 0.92)



Therefore Soil I is classified as Mineral soil with organic matter.

LOSS ON IGNITION (LOI)					
Soil Sample: Location: Boring No: Sample No: Sample Depth:		-			
Test No.	1	2	3	4	5
Crucible No.	А	В	С	D	F
Mass _{crucible empty} M _c (g)					
Mass _{crucible + wet soil} M _{cws} (g)					
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)					
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)					
Loss on Ignition LOI (%)					
Average Loss on Ignition LOI (%) Observations:					

LIQUID LIMIT TEST (LL)				
Soil Sample: Location: Boring No: Sample No: Sample Depth:	Date: Time: Tested by: Description:			
Oven-dried: ☐ Yes ☐ No				
Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)				
Mass _{tare + wet soil} M _{tws} (g)				
Mass _{tare + dry soil} (@ 110°C) M _{110°C} (g)				
Water content w (%)				
Number of blows N				
Liquid Limit =				
Observations:				

IDENTIFICATION AND CLASSIFICATION OF MARLY SOILS

"SEQUENTIAL" LOSS ON IGNITION TEST (LOI)

I- REFERENCES

- 1. ASTM D2974 07a
- 2. AASHTO T267 86
- Geotechnical Laboratory Measurements for Engineers (Germaine & Germaine, 2009)
- 4. Determination of calcium carbonate content in soils using sequential loss on ignition test (ITM 507)
- 5. Classification of marl soils (Jung et al., 2009)

II- SCOPE AND SUMMARY

This test method covers the procedure to determine the percentage of calcium carbonate (%CaCO₃) in soils using sequential LOI test.

III- APPARATUS

- 1. Oven capable of maintaining a constant temperature of 110°C ± 5°C.
- 2. Muffle furnace capable of attaining and maintaining a constant temperature of 800°C ± 10°C.
- 3. Scale of 0.01g readability.
- 4. Porcelain crucibles that can be heated up to 800°C.
- 5. Desiccator.
- 6. US standard sieve No. 10 (2 mm).

IV- PROCEDURE

Determine the mass of the porcelain crucible (M_c) to the nearest 0.01g. Note
that each crucible should be washed, marked with a permanent paint and
heated at the test temperature before it is used to perform any testing.

- 2. Obtain a representative soil specimen of 10g to 15g and sieve it through the No. 10 sieve (2 mm).
- 3. Place the soil sample in the crucible and determine the mass (M_{cws}) to the nearest 0.01g.
- 4. Oven-dry the specimen at 110°C ± 5°C for 24 hours (or until no mass loss is observed).
- 5. Remove the crucible and contents from the oven and place it in a desiccator to cool (~10 minutes).
- 6. Determine the dry mass $(M_{110^{\circ}C})$ to the nearest 0.01g.
- 7. Place the crucible and contents in a muffle furnace at 455°C for 6 hours.
- Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).
- 9. Determine the mass of the crucible with the ash $(M_{455^{\circ}C})$ to the nearest 0.01g.
- 10. Place the crucible and the soil into the furnace for 6 additional hours at a temperature of 800°C.
- 11. Remove the crucible from the furnace and place it in a desiccator to cool (~25 minutes).
- 12. Determine the mass of the crucible with the burnt soil ($M_{800^{\circ}C}$) to the nearest 0.01g.

V- CALCULATION

The loss on ignition is computed as follows:

$$LOI = \frac{M_{110^{\circ} C} - M_{455^{\circ} C}}{M_{110^{\circ} C} - M_{C}} \times 100$$

The CaCO₃ content is computed as follows:

$$CaCO_3 = \frac{100}{44} \times \frac{M_{455^{\circ}} C^{-}M_{800^{\circ}} C}{M_{110^{\circ}} C^{-}M_{c}} \times 100$$

Where:

LOI = loss on ignition of soil (%)

 $M_{110^{\circ}C}$ = mass of crucible and soil at 110°C (g)

 $M_{455^{\circ}C}$ = mass of crucible and ash at 455°C (g)

APPENDIX 4

 $M_{800^{\circ}C}$ = mass of crucible and burnt soil at 800°C (g) M_{c} = mass of crucible (g)

VI- REPORT

Report the organic content and the percentage of calcium carbonate to the nearest 0.1% together with the temperatures of the muffle furnace. If more than one specimen is tested report the averages and the standard deviation.

Classify the soil based on both OC and CaCO₃ contents. If the soil falls under "mineral" category based on organic content, it is classified based on CaCO₃ content only. Otherwise, dual classification shall be used (i.e. Marly soil and mineral soil with organic matter).

SEQUENTIAL LOSS ON IGNITION (LOI) – Sample Sheet

Soil Sample: Soil II	Date:	Sat 9/17/2011
----------------------	-------	---------------

Location: I69-sec3 Seg13 Time: 11:45am

Boring No: 3-37-TB-1 Tested by: AH

Sample No: Description: Dark Gray – Clayey

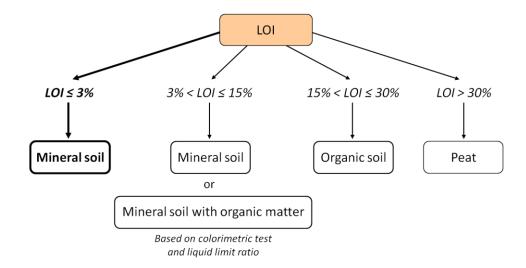
Sample Depth: 24 to 26ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2		
Crucible No.	CE1	2		
Mass _{crucible empty} M _c (g)	19.82	17.71		
Mass _{crucible + wet soil} M _{cws} (g)	35.53	33.23		
Mass _{crucible} + dry soil (@ 110°C) M _{110°} C (g)	30.03	27.80		
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.81	27.58		
Mass _{crucible} + burnt soil (@ 800°C) M _{800°C} (g)	27.24	25.08		
Loss on ignition LOI (%)	2.1	2.2		
CaCO ₃ content (%)	57.4	56.4		

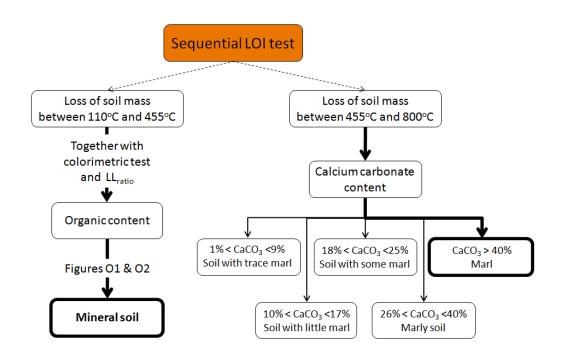
Average Loss on ignition (%) Average CaCO ₃ content (%)	2.1 56.9
Observations:	

Supporting Classification example:

Sequential LOI: %OC = 2.1% (< 3%)



<u>Sequential LOI</u>: $%CaCO_3 = 56.9\% (> 40\%)$



Therefore Soil II is classified as Marl

Soil Sample: Location: Boring No: Sample No: Sample Depth:		-			
Test No.	1	2	3	4	5
Crucible No.	А	В	С	D	F
Mass _{crucible empty} M _c (g)					
Mass _{crucible + wet soil} M _{cws} (g)					
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)					
Mass _{crucible + Ash} (@ 455°C) M _{455°C} (g)					
Mass _{crucible + burnt soil (@ 800°C)} M _{800°C} (g)					
Loss on ignition LOI (%)					
CaCO ₃ content (%)					
Average Loss on ignition (%)			-		
Average CaCO ₃ content (%)			-		
Observations:					

Organics

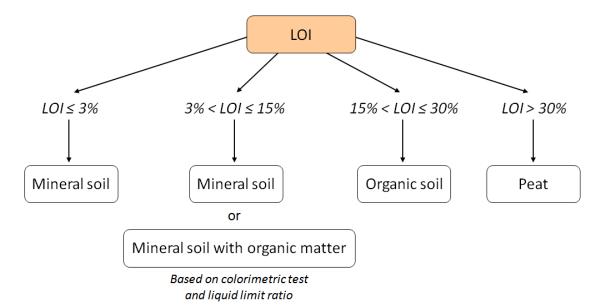


Figure O1

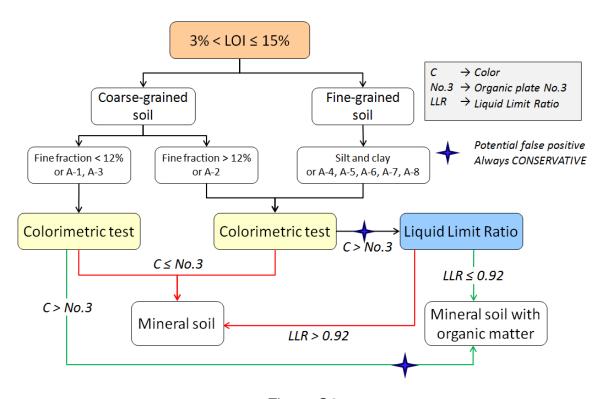
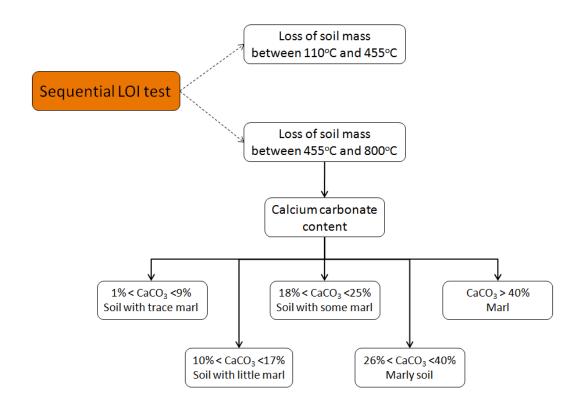
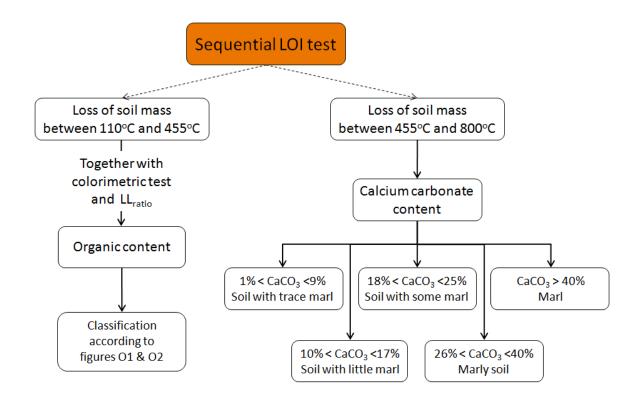


Figure O2

Marls



Combined (Organics & Marls)



"Organics" Classification Procedure & Checklist

____ 1. Perform the Loss on Ignition (LOI) test; (ASTM D2974–07a, AASHTO T267–86)

LOI =
$$\frac{M_{110^{\circ}C} - M_{455^{\circ}C}}{M_{110^{\circ}C} - M_{C}} \times 100$$

 M_c = mass of crucible

 $M_{110^{\circ}C}$ = mass after oven drying at 110°C

 $M_{455^{\circ}C}$ = mass after burning at 455°C

- ____ 2. Classify Soil based on Organic Content using Figure O1:
 - a) If LOI ≤ 3%, classify as "Mineral Soil"
 - b) If 3% < LOI ≤ 15%, GO TO STEP #3 BELOW (follow Figure O2)
 - c) If 15% < LOI ≤ 30%, classify as "Organic Soil"
 - d) If 30% < LOI, classify as "Peat"
- ____ 3. Perform Colorimetric Test; (ASTM C40 04, ASTM D1544 04, AASHTO T21 05)
- ____ 4. Classify Soil based on Organic Content using Figure O2:
 - a) If color $C \le 3$, classify only as "Mineral Soil"
 - b) If color C > 3 AND the soil is Coarse Grained with Fine Fraction < 12% (A-1 or A-3 soils), classify as "*Mineral Soil with Organic Matter*"
 - c) If color C > 3 AND the soil is Fine Grained or Coarse Grained with Fine Fraction >12% (A-2 soils), GO TO STEP #5 BELOW
- ____ 5. Perform Liquid Limit Ratio (LLR) Test; (ASTM D4318 10, AASHTO T89 10)

$$LL_{ratio} = \frac{LL_{oven dried}}{LL_{not dried}}$$

- ____ 6. Classify Soil based on Organic Content using Figure O2:
 - a) If LLR > 0.92, classify as "Mineral Soil"
 - b) If LLR ≤ 0.92, classify as "Mineral Soil with Organic Matter"

"Marls" Classification Procedure & Checklist

- ____ 1. Perform the Sequential Loss on Ignition (LOI) test
- ____ 2. Convert mass loss between 455°C and 800°C to CaCO₃ content using this equation:

CaCO₃ =
$$\frac{100}{44} \times \frac{M_{455^{\circ}C} - M_{800^{\circ}C}}{M_{110^{\circ}C} - M_{C}} \times 100$$

 M_c = mass of crucible

 $M_{110^{\circ}C}$ = mass after oven drying at 110°C

 $M_{455^{\circ}C}$ = mass after burning at 455°C

M_{800°C} = mass after burning at 800°C

- ____ 3. Classify Soil based on CaCO₃ content using "Marls" Flowchart:
 - a) If 1% < CaCO₃ < 9%, classify as "Soil with Trace Marl"
 - b) If 10% < CaCO₃ < 17%, classify as "Soil with Little Marl"
 - c) If 18% < CaCO₃ < 25%, classify as "Soil with Some Marl"
 - d) If $26\% < CaCO_3 < 40\%$, classify as "Marly Soil"
 - e) If 40% < CaCO₃, classify as "Marl"

"Combined (Organics & Marls)" Classification Procedure & Checklist

1. Perform the Sequential Loss on Ignition (LOI) test
2. Using LOI mass loss after burning at 455°C, follow "Organics" Classification Procedure to obtain Organic Content classification
3. Using LOI mass loss after burning at 800°C, follow "Marls" Classification Procedure to obtain Carbonate Content classification
4. Combine "Organic" and "Marl" classifications to obtain overall classification (se "Combined (Organics & Marls)" Flowchart)
- Example: if Organic Classification is "Mineral soil with organic matter", and if Carbonate Classification is "Marly soil", and if AASHTO Classification is "A-7-5", then classify as "Marly A-7-5 with organic matter"

LOSS ON IGNITION (LOI) - Sample Sheet

Soil Sample: Soil I Date: Sat 10/22/2011

Location: I69-Sec3 Seg13 Time: 11:00am

Boring No: 3-31-TB-2A Tested by: AH

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2	3	4	5
Crucible No.	А	В	С	D	F
Mass _{crucible empty} M _c (g)	17.67	17.94			
Mass _{crucible + wet soil} M _{cws} (g)	34.29	32.80			
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)	30.02	28.44			
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.20	27.47			
Loss on Ignition LOI (%)	6.6	9.3			

Average Loss on Ignition LOI (%)	8.0
Observations:	

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: ☐ Yes ■ No

Test No.	1	2	3	4
Crucible No.	А	В	С	D
Mass _{tare empty} M _t (g)	1.32	1.30	1.33	1.31
Mass _{tare + wet soil} M _{tws} (g)	4.90	4.95	4.91	5.60
Mass _{tare + dry soil} (@ 110°C) Mtds (g)	3.57	3.63	3.62	4.07
Water content w (%)	58.9	56.7	56.2	55.6
Number of blows N	14	25	29	34

Liquid Limit =	56.7%
----------------	-------

Observations:			

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil I Date: Thu 10/27/2011

Location: I69-Sec3 Seg13 Time: 11:30am
Boring No: 3-31-TB-2A Tested by: AH/MS

Sample No: Description: Black - silty

Sample Depth: 30 to 32ft (Bottom) 455 (6hrs) 800(6hrs)

Oven-dried: ■ Yes □ No

Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.30	1.34	1.30	1.33
Mass _{tare + wet soil} M _{tws} (g)	5.34	4.89	6.48	6.05
Mass _{tare + dry soil} (@ 110°C) Mtds (g)	4.03	3.77	4.86	4.59
Water content w (%)	47.9	46.4	45.5	44.9
Number of blows N	16	22	28	33

Observations:

SEQUENTIAL LOSS ON IGNITION (LOI) – Sample Sheet

Soil Sample:	Soil II	Date:	Sat 9/17/2011
--------------	---------	-------	---------------

Location: I69-sec3 Seg13 Time: 11:45am

Boring No: 3-37-TB-1 Tested by: AH

Sample No: Description: Dark Gray – Clayey

Sample Depth: 24 to 26ft (Bottom) 455 (6hrs) 800(6hrs)

Test No.	1	2		
Crucible No.	CE1	2		
Mass _{crucible empty} M _c (g)	19.82	17.71		
Mass _{crucible + wet soil} M _{cws} (g)	35.53	33.23		
Mass _{crucible} + dry soil (@ 110°C) M _{110°C} (g)	30.03	27.80		
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	29.81	27.58		
Mass _{crucible} + burnt soil (@ 800°C) M _{800°C} (g)	27.24	25.08		
Loss on ignition LOI (%)	2.1	2.2		
CaCO ₃ content (%)	57.4	56.4		

Average Loss on ignition (%) Average CaCO ₃ content (%)	2.1 56.9
Observations:	

SEQUENTIAL LOSS ON IGNITION (LOI) – Sample Sheet

Soil Sample: Sat 9/3/2011 Date: Sat 9/3/2011

Location: Lake George (Hobart) Time: 11:00am

Boring No: C6A Tested by: AH

Sample No: T5 Description: Dark Gray – Soft

Sample Depth: 22 to 24 ft 455 (6hrs) 800(6hrs)

Test No.	1	2	3	4	
Crucible No.	А	В	С	D	
Mass _{crucible empty} M _c (g)	17.68	17.94	20.60	18.45	
Mass _{crucible + wet soil} M _{cws} (g)	31.32	32.34	34.74	32.19	
Mass _{crucible + dry soil} (@ 110°C) M _{110°C} (g)	25.42	25.98	28.40	26.01	
Mass _{crucible + Ash (@ 455°C)} M _{455°C} (g)	24.86	25.40	27.81	25.45	
Mass _{crucible + burnt soil (@ 800°C)} M _{800°C} (g)	24.10	24.58	26.97	24.61	
Loss on ignition LOI (%)	7.1	7.2	7.5	7.5	
CaCO ₃ content (%)	22.4	23.2	24.5	25.1	

Average loss on	ignition (%)	7.3	
Average CaCO ₃	content (%)	23.8	
Observations:			
•			

LIQUID LIMIT TEST (LL) - Sample Sheet

Soil Sample: Soil III Date: Thu 9/15/2011

Location: Lake George (Hobart) Time: 2:00pm

Boring No: C6A Tested by: AH

Sample No: T5 Description: Dark Gray – Soft

Sample Depth: 22 to 24 ft 455 (6hrs) 800(6hrs)

Oven-dried: ☐ Yes ■ No

Test No.	1	2	3	4
Crucible No.	А	В	С	D
Mass _{tare empty} M _t (g)	1.32	1.32	1.32	1.31
Mass _{tare + wet soil} M _{tws} (g)	5.81	5.41	5.75	5.53
Mass _{tare + dry soil} (@ 110°C) M _{tds} (g)	3.93	3.74	3.98	3.81
Water content w (%)	72.0	69.0	66.9	68.8
Number of blows N	17	25	40	34

Liquid Limit =	69.7%
----------------	-------

Observations:

LIQUID LIMIT TEST (LL) – Sample Sheet

Soil Sample: Soil III Date: Fri 9/16/2011

Location: Lake George (Hobart) Time: 2:00pm

Boring No: C6A Tested by: AH

Sample No: T5 Description: Dark Gray – Soft

Sample Depth: 22 to 24 ft 455 (6hrs) 800(6hrs)

Oven-dried: ■ Yes □ No

Test No.	1	2	3	4
Crucible No.	Α	В	С	D
Mass _{tare empty} M _t (g)	1.33	1.32	1.33	1.31
Mass _{tare + wet soil} M _{tws} (g)	5.54	5.94	5.54	6.25
Mass _{tare + dry soil} (@ 110°C) M _{tds} (g)	3.88	4.17	3.96	4.41
Water content w (%)	65.0	62.0	60.4	59.2
Number of blows N	14	20	27	34

Liquid Limit =	60.9%
----------------	-------

Observations:

Self Learning Examples:

Soil IV

Site: I-69 sec3 seg12 Depth: 32 to 34 ft Location: Daviess, IN

LOI = 2.3%CaCO₃ = 2.9%

Soil V

Site: N/A Depth: N/A

Location: ASTM CL

LOI = 3.6%> 35% passing sieve # 200 Organic plate no. 5 LL_{ratio} = 0.98CaCO₃ = 4.2%

Soil VI

Site: Lake George Dam

Depth: N/A

116

Location: Hobart, IN

LOI = 6.8%> 35% passing sieve # 200 Organic plate no. 5 LL_{ratio} = 0.83CaCO₃ = 21.7%

Classification of Organic Soils and Classifications of Marls: Training Dates

Training Session 1 (Pilot Session)

February 16, 2012

INDOT Materials Testing Facility, Indianapolis, IN

Attendee	Affiliation
Nayyar Siddiki	INDOT, Geotech Dept.
Thomas Nantung	INDOT
Brian Dunbar	INDOT, Geotech Dept.
lqbal Khan	INDOT
Michael Nelson	INDOT, Greenfield District
Ron Fine	INDOT, Crawfordsville District
Antonio Bobet	Purdue University, Dept. of CE
Marika Santagata	Purdue University, Dept. of CE
Alain El Howayek	Purdue University, Dept. of CE
Sulaiman Dawood	Purdue University, Dept. of CE
Andrew Ferdon	Purdue University, Dept. of CE

Training Session 2

March 16, 2012

INDOT Seymour District Office, Seymour, IN

Attendee	Affiliation		
Nayyar Siddiki	INDOT, Geotech Dept.		
Brian Dunbar	INDOT, Geotech Dept.		
Bill Jarvis	INDOT, Seymour District		
Deloris Rieckers	INDOT, Seymour District		
Judy Turner	INDOT, Seymour District		
Chris Bell	INDOT, Seymour District		
Alain El Howayek	Purdue University, Dept. of CE		
Sulaiman Dawood	Purdue University, Dept. of CE		
Andrew Ferdon	Purdue University, Dept. of CE		

Training Session 3 March 30, 2012

INDOT Laporte District Office, Laporte, IN

Attendee	Affiliation		
Heather Woods	INDOT, Laporte District		
Mike Bramblett	INDOT, Laporte District		
Judith Hammons	INDOT, Laporte District		
Rhonda Giggy	INDOT, Laporte District		
Bob Dahman	INDOT, Ft. Wayne District		
Alain El Howayek	Purdue University, Dept. of CE		
Sulaiman Dawood	Purdue University, Dept. of CE		
Andrew Ferdon	Purdue University, Dept. of CE		

Training Session 4 April 3, 2012

INDOT Materials Testing Facility, Indianapolis, IN

Attendee	Affiliation
Jean Hiadari	INDOT
Heather Holder	INDOT
Donna Sipes	INDOT
Linda Spitsyna	INDOT
Brian Dunbar	INDOT, Geotech Dept.
Melvin Hall	INDOT
Youlanda Belew	INDOT
Michael Pritt	INDOT
Jackie Barnes	INDOT
Kulanand Jha	INDOT
David Jacobs	INDOT, Ft. Wayne District
Kellen Heavin	Alt & Witzig Engineering
Geoffrey Thompson	Earth Exploration Inc.
Matthew Brading	ATC
Kenneth Rush III	CTL Engineering, Inc.
Bill Dubois	Patriot Engineering
Abdul Khalaf	Chicago Testing Lab
Alain El Howayek	Purdue University, Dept. of CE
Sulaiman Dawood	Purdue University, Dept. of CE
Andrew Ferdon	Purdue University, Dept. of CE





Feedback Form

	Very Good	Good	Fair
Organization of content	\bigcirc	\bigcirc	\bigcirc
Clarity of presentation	\bigcirc	\bigcirc	\bigcirc
Depth of material covered	\circ	\bigcirc	\bigcirc
Language and Visual effects used	\circ	\bigcirc	\bigcirc
Ease in comprehending the classification table	\circ	\bigcirc	\bigcirc
Effectiveness of examples provided	\circ	\bigcirc	\bigcirc
Sufficiency of handouts	0	0	\circ
Met your expectation	0	0	\circ
Overall rating of the presentation	\circ	\bigcirc	\bigcirc
Please provide additional comments:			