DETERMINATION OF TOTAL ORGANIC CARBON (TOC) USING THE SHIMADZU SSM-5000A SOLID SAMPLE MODULE

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1. SCOPE AND APPLICATION

1.1. This SOP details the analysis of Total Organic Carbon (TOC) in solid and waste matrices. The test covered by this SOP is S-TOC.

1.2. Sample results for Total Carbon (TC), Inorganic Carbon (IC) and TOC are reported as percent carbon. The maximum amount of carbon that can be measured in one aliquot of sample is 18 mg.

1.3. The method detection limit (MDL) of 0.12 % and the practical quantitation limit (PQL) of 0.30 % carbon for TC and IC are based on the analysis of 500 mg of sample.

2. SUMMARY OF THE METHOD

2.1. Samples are pulverized and dried prior to analysis. See DEP SOP MT-064.

2.2. Samples are separately analyzed for their TC and IC content. The IC fraction is subtracted from the TC result to determine the TOC content.

2.3. TC analysis: The sample is combusted at 900 °C in a column containing a platinum and cobalt oxide catalyst. Under these conditions, all carbon compounds in the sample are converted to carbon dioxide, which is detected by an infrared detector.

2.4. IC analysis: The inorganic component of the sample is oxidized with concentrated phosphoric acid at 200 °C to form carbon dioxide, which is detected by an infrared detector.

2.5. Interferences - Samples containing large amounts of alkaline metal carbonates or phosphates may require the addition of tungsten oxide (WO_3). Tungsten oxide is used to accelerate the combustion or thermal decomposition reactions.

3. EQUIPMENT AND SUPPLIES

3.1. Shimadzu Total Organic Carbon analyzer TOC-V CPH or Shimadzu Total Organic Carbon analyzer TOC-L CPH equipped with a SSM-5000A solid sample module (SSM 1, SSM 2, or SSM3)

3.2. Shimadzu Ceramic Sample Boats

3.3. Spatula

3.4. Analytical Balance

3.5. Ceramic Fiber

3.6. Muffle Furnace (Capable of reaching 900 °C)

3.7. Oven (capable of reaching 105 °C for sample drying)

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4. REAGENTS AND STANDARDS

Note: Prior to using the chemicals in this analysis, please read their MSDS and the safety notes found in the safety/hazardous waste section of this SOP.

Note: All reagent and standard preparations must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard and reagent are generated in the module and documented in the Reagent and Standard Logsheet (NU-RGTSTD-1.0_S-TOC) located on the network at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\TOC\. This excel sheet needs to be updated and submitted as part of the analysis report folder. If available, a Certificate of Analysis for each purchased standard or reagent is also maintained in the REAGENT_PREP\TOC folder. All containers holding standards or reagents must be labeled with their unique ID and expiration date.

Note: The volumes prepared for the reagents and standards may be altered by the analyst as necessary. The final concentrations of the constituents of a standard or reagent must not be changed.

4.1. Anhydrous Dextrose ($C_6H_{12}O_6$) (Ground)

4.2. Anhydrous Sodium Carbonate (Na_2CO_3)

4.3. Tungsten Oxide (WO_3) (Used only as an accelerant if necessary)

4.4. Concentrated Phosphoric Acid (H_3PO_4)

4.5. 2M Hydrochloric Acid (HCl) (Used only to clean the sample boats)

4.6. Certified Soil Standard (Available from Environmental Resource Associates)

4.7. Ottawa Sand

4.8. UPC Grade Oxygen (99.9 % O_2)

5. SAMPLE HANDLING, PRESERVATION AND STORAGE

5.1. Upon receipt the samples are stored refrigerated in the custody room C301. All samples must be analyzed within 28 days of the collection date to avoid Q qualification of results.

5.2. The samples are prepared for TOC analysis in the metals section of the laboratory according to DEP SOP MT-064. The data for this process is archived in the following locations: \\FLDEP1\DEAR\labs\lab5\Chemistry\InorganicPrep\METALS\% SOLID LOG BOOK\ in subfolders called PS60% & Grinding YYYY or PS105% YYYY. After prep, samples are turned from W to V status using the LIMS prep worksheet "SOP MT-064". Record the filename(s) and/or the prep worksheet ID(s) associated with the samples analyzed on the S-TOC analysis worksheet. After preparation, the dried and pulverized sample aliquots are stored in centrifuge tubes in the nutrients laboratory (B338). Consult the supervisor if other sample aliquots need to be located.

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5.3 All samples are dried again at 105 °C at least overnight before they are analyzed. The inorganic carbon part of the analysis is performed on a wetted sample aliquot. This aliquot usually stems from the same sample aliquot used for TC analysis, which is wetted by addition of water prior to IC analysis. If the sample is difficult to wet, a freeze dried aliquot, the original sample, or a ground aliquot prepared for sediment total phosphorus/total Kjeldahl nitrogen analysis (see DEP SOP NU-048) can also be used (see section 7.5. below). Use the % solids data to aid in the determination of the most appropriate aliquot.

6. INSTRUMENT SETUP

6.1. Turn on all instrument components and computer, and open valves on oxygen and air carrier gas.

6.2. Open software by clicking on 'TOC-Control-V' icon for the TOC-V instrument or "TOC-Control-L" for the TOC-L instrument.

6.3. Select the 'Sample Table Editor' icon.

6.4. On the window that appears: Select 'ok'.

6.5. Select *File → New*. Select 'Sample Table'. The general information window appears. **Depending on the instrument, select 'TOCV-S' or 'TOCL-S'. By default it shows TOC-W.**

6.6. Connect instrument by clicking on the CONNECT icon at the top of the screen or by selecting *Instrument → Connect*.

6.7. Select Instrument. → *H/W Settings* and click on the SSM Tab. Make sure the radial buttons indicating that the TC and IC furnaces are on are selected and click OK. **The furnaces must be given at least one hour to come to temperature and achieve a stable baseline.**

6.8. To format the results table go to *Options →Display settings → Sample Table/Schedule File*. Then go to → Display items → Select the following - 1. Type, 2. Analysis, 3. Sample, 4. Sample ID, 5. Result, 6. Status, and 7. Date/Time.

6.9. Highlight row one and select *Insert → Calibration Curve*.

6.10. Choose calibration for TC component "Calibration_stc.cal" and click OK. (The TC component must be run before the IC component for the QC Manager driver to perform TOC calculations correctly.)

6.11. Highlight the second row and select *Insert → Calibration Curve*.

6.12. Choose calibration for IC component "Calibration_sic.cal" and click OK.

6.13. Highlight the third row and select *Insert → Multiple Samples* and activate the "Calibration Curve" radial button. Select "Calibration_stc.cal" from the list → Open and click "Next" to continue.

6.14. Enter the number of cups to be analyzed for TC, enter the run number (i.e., the

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filename: YYMMDDS#, where #1 indicates SSM 1, #2 indicates SSM 2, and #3 indicates SSM3) where it asks for sample name, and leave the sample ID box empty. → *'Finish'*.

6.15. Select *Insert* → *Multiple Sample* and activate the "Calibration Curve" radial button. Select "Calibration_sic.cal" from the list → Open and click "Next" to continue.

6.16. Enter the number of cups to be analyzed for IC, enter the run number (YYMMDDS#) where it asks for sample name, and leave the sample ID box empty. →*"Finish"*.

6.17. Click Save icon at the top → enter file name YYMMDDS# → Save

6.18. After the run template is completed, enter the IDs of all samples and QC.

7. PROCEDURE/SAMPLE ANALYSIS/QUANTITATION/CALIBRATION AND STANDARDIZATION (SEE DEP SOP NU-033)

7.1. Screen all samples for their inorganic carbon content - Take a small amount of each sample and add about 2-3 drops of conc. phosphoric acid. If you notice bubbles evolving from the wetted sample, it contains a significant amount of inorganic carbon. For these samples, weigh no more than 100 mg as your first weight for the IC analysis. These samples can potentially boil over in the reaction tube and damage the instrument.

7.2. Two sets of standards are required due to separate analysis of IC (Na_2CO_3) and TC (Dextrose). Four sample boats are needed for each calibration.

TC calibration: Load three sample boats with 45 mg, 20 mg, and 3.75 mg Dextrose, respectively. The last sample boat is loaded with 500 mg Ottawa sand.

IC calibration: Load three sample boats with 160 mg, 80 mg, and 13.2 mg Na_2CO_3, respectively. The last sample boat is loaded with 500 mg Ottawa sand. Before an IC standard or sample is inserted into the sample port, add deionized (DI) water, usually about 300 uL, to wet the sample entirely.

7.3. Click on the Start icon at the top of the screen to begin analysis. TC and IC calibrations are run first. After both calibrations are completed, all TC samples are analyzed before the IC samples are run.

7.4. The instrument will begin calibration by showing information corresponding to the first standard. Insert the correct sample boat into the sample chamber and seal. The instrument baseline will fluctuate when the sample chamber is opened due to atmospheric CO_2. Wait for 2 min to allow the baseline to correct and show ready, then click OK on the pop up window. Follow on screen directions concerning sample position. Repeat for each calibration standard.

7.5. After the instrument is calibrated, it will automatically move on to the samples to be analyzed. Load samples into sample boats. The first analysis should be performed using 200 mg of sample (unless the sample contains a significant amount of inorganic carbon, or its appearance points to a particularly high or low

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TC content). A maximum weight of 500 mg is used per sample boat. Sample weight can be adjusted after the initial analysis to assure results are between the PQL and the upper calibration limit (see 7.7. below). If the sample does not wet entirely during IC analysis, make a note on the bench sheet and reanalyze the sample using an aliquot that is not 100 % solid and wet it additionally, if possible, before analysis (see section 5.3. above).

7.6. At the beginning of the analysis, a box will appear to enter the weight (in mg) of the sample. If a wet (< 100 % solid) sample was used for IC analysis make sure to enter the corresponding dry weight of the sample aliquot in the box. After entering the weight, click "Start" and follow on screen directions until analysis is complete.

7.7. After analysis is complete, the TOC Measurement box will appear. This will allow the analysis to be stopped by clicking "Stop". Continue to the next sample by clicking "Next" or repeat the sample analysis by clicking "Repeat". Repeat the analysis at a different weight if results are not between the PQL and upper calibration limit. Before selecting "Repeat", the first result should be excluded. Highlight the sample number in the box and click "Exclude". An 'E' will appear as a remark code for the first analysis. Now click on "Repeat" to continue.

7.8. Following completion of the final sample in the run, the instrument will go into standby mode. The SSM furnaces should be powered down. Go to *Instrument→ H/W Settings*. Click on the SSM Tab. Uncheck the selection boxes for the furnaces. Open the sample port cover (TC and IC side), then turn the oxygen and air carrier gases off after the temperature goes down to about 100 °C.

7.9. Post-run procedures:

7.9.1. Results are exported as an ASCII file and converted into a raw data in the QC Manager for performing calculations and uploading to the Laboratory Information Management System (LIMS).

7.9.1.1. Check/Set the Export Options. Go to *'File'* → *'Export Options'* → *'Data'*. Make sure the box *'Export Data'* is checked. Four items need to be selected: 1. Sample ID, 2. Date/Time, 3. Result, 4. Weight.

7.9.1.2. Export the file. Go to *File* → *ASCII Export* →*Detail (include injection data).*

7.9.1.3. Export file location - C:\TOC3201\Data for TOC-V and C:\TOC-L\Data for TOC-L. There are three files created (.txt, .pkt, .t32 for TOC-V and .txt, .pkt, .tlx for TOC-L) that need to be transferred to the nutrients drive.

7.9.1.4. Open Windows Explorer and copy the files described above to the S-TOC folder on the nutrients drive at \\FLDEP1\DEAR\labs\lab3\Chem_Data\Inorganic\Nutrients\S-Toc\. Open .txt file→Edit→Replace→type % in Find what→ % (one space before %) in Replace with→ Replace all→save.

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7.9.2. Print a copy of the final report and data tables containing injection peaks by selecting: *File → Print → Sample Report – All Report* then *File → Print → Table*. Place into a manila folder along with the bench sheet and standard sheet.

7.9.3. Quantitation/Calculations:

7.9.3.1. The instrument will give the analysis results for the IC and TC components of the sample in % Carbon. The QC Manager calculates the TOC content of each sample, which is also reported as % Carbon.

7.9.3.2. The QC Manager calculates recoveries for spikes and QC standards as well as relative percent differences (RPD) for duplicate samples and spikes. For general calculations including RPDs and spike recoveries, see Section 5.9 of the FDEP Quality Manual.

7.9.3.3. The calibration standards for the IC and TC calibration are prepared according to section 7.2. The correlation coefficients for the linear calibrations are calculated by the instrument software.

7.9.3.4. If a sample amount less than the maximum (500 mg) was used for IC or TC analysis enter the ratio of the maximum amount to the amount used in the 'Dilution X' column in the QC Manager sample table before calculation. Make sure the 'Apply Dil?' column is set to 'N'. This will update the MDL and PQL associated to the analysis.

8. MAINTENANCE/TROUBLESHOOTING

8.1. Maintenance:

Note: Document all maintenance performed in the instrument's maintenance log.

8.1.1. Before beginning sample analysis, conduct a general inspection of the instrument to ensure that everything is in order.

8.1.2. The humidifier vessel should be filled with DI water to the fill line. Once a month, this vessel should be drained and refilled with fresh DI water. This prevents contamination.

8.1.3. Clean sample ports using cotton swabs after each day's run to prevent build-up of sample ash and phosphoric acid from analysis.

8.1.4. Remove tubing connecting acid dispenser to the sample port, to prevent siphoning into sample port while not in use. Flush out tube after disconnecting to prevent formation of solid particles that may clog tubing.

8.1.5. The power to the main instrument should never be turned off while SSM furnaces are hot; this will turn off power to the cooling fan and will

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damage the SSM.

8.1.6. Check gas flow rate and pressure prior to analysis – both tanks should have at least 1000 psi before starting analysis.

8.1.7. Cleaning of sample boats - Shimadzu SSM-5000A User's Manual p.15.

8.2. Troubleshooting - See Shimadzu TOC-V Series SSM-5000A User's Manual Section 4.2.

9. QUALITY CONTROL/METHOD PERFORMANCE/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOPS NU-038 AND NU-043)

9.1. For every 20 TC and IC samples, analyze a method blank (MBLK), duplicate sample or duplicate spike, laboratory fortified blank (LFB), and an appropriately spiked sample.

9.1.1. MBLK and blank (BLK)- Prepare a sample boat containing 500 mg of Ottawa sand for the IC and TC part of the analysis. Acceptance limits for the MBLK are > - 0.12 % C and < 0.12 % C.

9.1.2. Matrix Spike - The TC sample is spiked with an amount of dextrose to yield an appropriate carbon spike (contact your supervisor if you have problems selecting the sample spike). The IC sample selected is spiked with an amount of Na_2CO_3 to yield a 2.26 % carbon spike. Calculate of spike amounts as follows:

TC Spike → Sample Wt. (mg) x (Spike % C as fraction) = Carbon Needed (mg)

(Carbon Needed (mg))/(0.40) = Dextrose for Spike (mg)

IC Spike → Choose a 500mg sample with less than 1 % carbon and spike with 100 mg Na_2CO_3.

Note: When the weight of spiked samples is entered into the instrument during analysis, only the sample weight is entered.

9.1.3. The LFB and check standards (CHCK) are prepared by weighing out a 20 mg (TC)/80 mg (IC) aliquot of the TC/IC calibration standards. For LFB (TC/IC) only, add 500 mg Ottawa sand after weighing out 20 mg (TC)/80 mg (IC) of the TC/IC calibration standards. The TC LFB (Dextrose) result acceptance limits are 34-46 %. The IC LFB (Na_2CO_3) acceptance limits are 9.7-12.9 %.

9.2. Analyze a check standard and a blank every 20 TC and IC samples. If the analysis is performed over multiple days, a check standard and a blank should be also analyzed at the beginning and at the end of each day.

9.3. A PQCK spike (0.30 % C) should be analyzed at the beginning of the run and at the beginning of each day, if analysis continues over multiple days. PQCK standard is prepared by weighing out 3.75 mg of Dextrose + 500 mg of Ottawa Sand for TC analysis (PQCK3) or 13.2 mg of Na_2CO_3 + 500 mg of Ottawa Sand

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for IC analysis (PQCK4). PQCK4 is analyzed only for IC. PQCK3 is analyzed for both TC and IC and is used for assessing method performance for Total Carbon and Organic Carbon. PQCK spikes serve two purposes: to assess instrument performance at the low concentration range and for ongoing MDL/PQL verification.

9.4. Analyze a certified soil sample at the beginning of the run. (An ERA certified soil sample is currently used.) Dry the certified soil sample at 105 °C overnight before the run. For the concentration, acceptance limits, and expiration date of the certified standard, please see the manufacturer's certificate.

9.5. Acceptance limits for recovery of spikes and the check standard are 85-115 %. Acceptance limits for recovery for the PQL (PQCK spike) are 70-130 %.

9.6. Relative percent difference (RPD) for replicate samples must be less than 20 %.

9.7. If there are any anomalous results (e.g., differing results for multiple analyses of same sample) or any unusual events (spilled sample, cannot locate sample, etc.) see supervisor for further instruction.

9.8. For method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data, refer to DEP SOP NU-038.

9.9. Linear Calibration Range: See FDEP QA Manual, Appendix B, Section 7.0, 'Technical Requirements'. The correlation coefficient of the linear calibration must be equal to or larger than 0.995.

The run sequence is identical for the Total Carbon and Inorganic Carbon runs:

An example includes:

Calibration:

TC Calibration (4 cups)
IC Calibration (4 cups)

Total Carbon/ Inorganic Carbon samples and QC:

Initial QC:
TC/IC ALTQC-sample (certified soil sample) analyzed at the beginning of each analytical run

TC/IC CHCK (CCV) - analyzed at the beginning and end of each run, and at beginning and end of each batch, and at the beginning and end of each day of a multi-day analysis

TC/IC PQCK - analyzed at the beginning of each analytical run and at the beginning of each day of a multi-day analysis; analyze PQCK3 for TC, analyze both PQCK3 and PQCK4 for IC

TC/IC Blank (CCB)- analyzed at the beginning and end of each run, beginning and end of each batch, and at the beginning and end of each day of a multi-day analysis

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Samples and batch QC:

TC/IC MBLK (Can be used as the initial QC blank to shorten the run. Analyze minimum of one MBLK per batch of 20 samples)

TC/IC LFB (min one LFB per batch of 20 samples)

TC/IC Matrix Spike (min one MS per batch of 20 samples)

TC/IC Matrix Spike or sample duplicate (min one duplicate per batch of 20 samples)

TC/IC samples (up to 20 samples per batch) *
*if more than one batch analyzed, each batch must be bracketed by a CHCK and a BLK standards

End of the run or end of the batch QC:

TC/IC blank (BLK)

TC/IC CHCK – end of the run

Note: Additional QC for multi-day runs: if the TC analysis runs over multiple days, end each day with CHCK and BLK, then start next day with CHCK, BLK, PQCK

10. DATA ARCHIVAL

10.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test (S-TOC), filename/s (YYMMDDS#, where #1 indicates SSM 1, #2 indicates SSM 2, and #3 indicates SSM 3), and initials of the analyst.

10.2. Each report folder should contain the following items - run report including raw data and calibration curves, results report, bench sheet, Reagent and Standard Logsheet, a copy of the ERA certificate, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

10.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

10.4. TOC instrument raw data files (*.pkt and *.t32/*.tlx) and data transfer files for upload to QC manager are stored in electronic format in the current year's subfolder located at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\S-Toc\.

10.5. If available, a Certificate of Analysis for each purchased standard or reagent is maintained in the \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\TOC\Certificate of Analysis\ S-TOC certificates\ folder.

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11. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

Warning: This analysis is performed at high temperatures. Allow all sample boats plenty of time to cool after removing from furnace before they are handled! Hot sample boats must be placed on a heat resistant surface.

11.1. Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/system/files/Division-of-Environmental-Assessment-and-Restoration/governing-documents/Health%20%20Safety%20Plan_0.docx).

11.2. Proper safety equipment such as lab coats, gloves, and safety glasses should be worn at all times during analysis.

11.3. Read the Materials Safety Data Sheet (MSDS) information for safe reagent and sample handling.

11.4. After all events are authorized the prepared sample aliquots are discarded in the regular lab trash. If a sample contains a known hazard consult the safety officer for proper disposal instructions.

12. REFERENCES/DEFINITIONS/VALIDATION DATA

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use. Method validation data is on file with the supervisor.

12.1. EPA Method 415.1.

12.2. Shimadzu TOC-V/L Manual.

12.3. Shimadzu SSM-5000A Manual.

12.4. Quality Manual, Bureau of Laboratories, FDEP.s

12.5. Laboratory Health and Safety Plan.

12.6. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

12.7. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

12.8. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

12.9. DEP SOP MT-064, Homogenization and Pulverization of Solid Samples.

12.10. DEP SOP NU-048, Preparation of Sediments for Total Kjeldahl Nitrogen and Total Phosphorus Analyses.

12.11 DEP SOP LB-006, Standard operating procedure for records maintenance and storage.

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Appendix of Significant Changes

09/21/2010 Added second TOC instrument and SSM to Section 3.

09/28/2012 Sections 6, 7 and 10 edited to reflect new instrument software which is installed on the TOC-L-CPH instrument.

09/15/2014 Section 5 and 7 are revised to address sample analysis in cases where the sample cannot be appropriately wetted during IC analysis. A reference to definitions and validation data is added to section 12.

12/10/2014 Clarified instrument used for analysis in section 3.

12/10/2015 Data archival information was clarified in Section 10.

02/25/2016 Section 7 was edited to clarify details of the QC Manager file write up.

03/09/2018 Table of context added. Example run sequence added to section 9.

09/18/2018 Added references to TOC-L and SSM-3 use. Highest amount of carbon measurable reduced to 18 mg, PQL lowered to 0.36 % C, and MDL determined to be 0.12 % C. Modified standard amounts used for calibration (see section 7.2), PQCKs (0.30 % C), and check standards (20 mg Dextrose for TC, 80 mg Na_2CO_3 for IC). Introduced use of PQCKs for ongoing MDL/PQL verification.

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