

### ICCP Working Group Identification of Thermal Maturity Relevant Organic Matter 2022 Report

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Presented for ICCP Commission II, September, 2022



# **Outline of this presentation**

- Problem to be solved
- History of the working group
- Updates since 2021:
  - USGS intra-laboratory exercise on images
  - o ASTM D7708 ballot
  - Springer encyclopedia article
- Summary & proposal for new activities







# **Objective of the working group**



• Provide guidelines for identification of thermal maturity relevant organic matter population in dispersed organic matter





### History of the working group



- Proposed by Angeles Borrego 2008 Oviedo
- DOMVR survey 2009 Gramado, ICCP News No. 48
- ASTM standard D7708 in 2011 ASTM Standards







### History of the working group, cont.

- ASTM D7708 interlaboratory study (ILS) in 2012-2013
- Results presented ICCP, Sosnowiec 2013
- Results presented AAPG, Houston, USA, 2014
- Results used to update ASTM D7708
- Results published in J. Marine and Petroleum Geology, 2015 (125+ citations)

Porto 2011 Beijing 2012 Sosnowiec 2013 Kolkata 2014 Potsdam 2015





- Second interlaboratory study, 2015-2016
- Results presented ICCP, Houston 2016
- o Discussions @ ICCP, 2017-2018
- Manuscript published, Marine & Pet. Geol., April 2020 (13 citations, 2.4 FWCI)



### **≊USGS** Summary of working group products





Designation: D7708 – 11

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks<sup>1</sup>

#### Marine and Petroleum Geology 59 (2015) 22–34 Contents lists available at ScienceDirect



Marine and Petroleum Geology

journal homepage: www.elsevier.com/locate/marpetgeo

#### Research paper

Standardization of reflectance measurements in dispersed organic matter: Results of an exercise to improve interlaboratory agreement

CrossMark

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#### Marine and Petroleum Geology 114 (2020) 104172



Research paper



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### **Updates since 2021**

- USGS intra-laboratory image-based study
- ASTM D7708 ballot for renewal
- Springer Encyclopedia of Petroleum Geoscience article: Vitrinite Reflectance Analysis







### **USGS Intra-laboratory Image Study**



- Organic matter identification in PowerPoint images
- Five petrographers Paul, Brett Valentine, Javin Hatcherian, Maggie Sanders, Jennifer Rivera
- Images from 2015-2016 ILS
- Six samples Barnett, Bakken, Eagle Ford, Woodford, Haynesville, Marcellus Shales
- Thermal maturity from ~1.0 to 2.0% BRo



## **USGS Intra-laboratory Image Study**

- Fifty images in PowerPoint, Excel answer key
- Perfect agreement (5 of 5) correlated to TOC
- Lower thermal maturity, better agreement
- Vitrinite 'different' or larger than groundmass OM
- Micrinite <10 µm but non-granular
- Inability to 'focus through' to resolve granularity confused micrinite for inertinite, solid bitumen for micrinite



Woodford

80



### **USGS Intra-laboratory Image Study**

- Inability to scan through sample for comparison
- Differences in white balance
- Provide reflectance measurement of queried maceral
- Provide higher magnification (1,000x) image of queried maceral
- Comment field in answer sheet, guidance that not all macerals present in each image
- o ICCP News 82, p. 6-7







- ASTM standards are revised or withdrawn after 8 years
- D7708 was last revised in 2014, and will be withdrawn Jan 1, 2023, if not revised
- Comments were solicited from ICCP user community, DOMVR-accredited petrographers



Designation: D7708 – 14

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks<sup>1</sup>

This standard is issued under the fixed designation D7708; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (*e*) indicates an editorial change since the last revision or reapproval.





- Many (100s) comments received, used to revise standard
- D7708 balloted (concurrently) online in July 2022

INTERNATIONAL Desig	gnation: D7708 – 14
Date.	April 5, 2022
10:	Subcommittee D05.28/Main Committee D05 members
Tech Contact:	Paul Hackley, phackley@usgs.gov, 301-606-0926 (teleworking)
Work Item #:	WK80505
Ballot Action: Vitrinite Dispers	Revision of D7708 Standard Test Method for Microscopical Determination of the Reflectance of sed in Sedimentary Rocks, sections 1, 2, 3, 4, 5, 6, 7, 9, 10, 11, and 12
Rationale: survey of the us underlined text, balloted; all oth 2023.	This test method is being revised based on comments received by the technical contact from a ser community. Changes to existing text are shown in red/blue font, additions are indicated by red/blue and deletions are indicated by red/blue strikethrough text. Only the actual revisions are being er text present is for context only. The test method needs to be revised or withdrawn by January 1,
Standard Test	Method for cal Determination of the Reflectance of Vitrinite Dispersed in Sedimentary



Rocks<sup>1</sup>



#### • Example of a revision

#### 4. Summary of Test Method

4.1 In this test method, the reflectance of light reflected from a polished surface of the vitrinite maceral or other maceralsorganic matter types is covered by immersion oilis measured is and determined by illuminating the polished surface of a sedimentary rock or thin section, or a particulate preparation of rock core, drill cuttings, or kerogen concentrate covered by immersion oil using a microscope-photometer or other detection system that measures the amount of light reflected from the surface. The reflected light is recorded in percent reflectance after calibration of the photometric or other detection equipment. The calibration of the detection equipment is accomplished by measuring the reflected light reflected from standards of known reflectance as calculated from their refractive indices (see 6.13, Calibration Standards), and measured against reference standards. All measurements are made on surfaces covered by an immersion oil.

4.1.1 Color photomicrographs of vitrinite and other organic materials dispersed in sedimentary rocks are available from various publications and websites.

4.1.2 The scholarly organization International Committee for Coal and Organic Petrology hosts a biennial accreditation program for organic petrographers using D7708 to measure reflectance of vitrinite dispersed in sedimentary rocks.



- ~300 members of ASTM D05, committee for coal and coke
- no DOMVR petrographers
- Two negative voters
- Negotiating for withdrawal of negative votes
- Technical changes will be reballoted later in 2022 or 2023

MyASTM / Membership / MyCommittees									
МуС	omn	MyTools							
Commi	ittee D05	Negative & Comments 3							
Ballots	Rosters	Meetings & Symposia	Minutes & Agendas	Committee Documents	Standar Trackin	rds g	MyNext Meetings		
ITEM	SUB	ACTION							
012	28	REVISION OF D7708-2014							
TECHNICAL CONTACT:									
Paul C H	ackley				Main	Sub			
WORK ITEM: WK80505				Affirmative	33	11			
				Negative	0	1			
				Abstain	56	10			
				%Affirmative	100.00	91.66			
NEGATIVE VOTERS: (all ASTM member negatives must be considered)									
Thomas W Vetter Editorial Withdraw 09/19/2022 Processed									
*	* <u>Xianai Huang</u> <u>Enter Disposition</u>								
NON-OFFICIAL VOTING MEMBER: # INDICATES SUB; * INDICATES MAIN									
COMMENTS:									
Section	State	ement							
3.2.2	Rom rotati meas	Romax measurement should not be limited to "using a fixed microscope stage and a rotating polarizer" since "a rotary stage and fixed polarizer" should also be included for measuring the Romax.							









#### Dear Xianai,

This message is in regard to your negative vote on ASTM D7708 regarding the language of rotating polarizer and fixed stage vs fixed polarizer and rotating stage.

Unlike coal vitrinite analysis, it is generally not possible to measure Romax of dispersed organic matter using a rotating stage, because the small size (5-10 um) of dispersed organic matter particles prevents their centration and continual location under the measuring aperture during stage rotation. This is discussed in the two attachments.

To my knowledge, there are no petrographic laboratories worldwide which currently report the Romax of dispersed organic matter, or, if approached to do so, would perform this analysis via the use of a rotating stage.

With that information in consideration, I ask if you would consider withdrawing your negative? If you're still of the opinion that fixed polarizer and rotating stage should be included, what language would you suggest to use? I hope then you would also withdraw the negative, with the understanding that I will register a new work item and re-ballot to include your revisions once the current item has been reapproved, allowing it to remain active.

Thanks and best regards,

Paul



#### [EXTERNAL] RE: ASTM D7708 ballot 🛛 🗍 4 🗸 🖽



Vetter, Thomas W. (Fed) <thomas.vetter@nist.go</th>...To: Hackley, Paul CFri 7/8/2022 6:42 PM

Paul: Everything I commented on is likely to be considered editorial, with the exception of my comments on Equation 1 (define 100 and 1.5180), the suggestion in 11.1.2 that "concentration" be replaced with "mass fraction, and the comments for Table 1 stating that "weight" and "wt." be replaced by "mass fraction". ASTM editorial will consider all of those to be technical so they need to be balloted.

Tom

....

 $\leftarrow$  Reply  $\rightarrow$  Forward



WITHDRAWN (Entire negative vote was withdrawn without editorial changes)

WITHDRAWN WITH EDITORIAL CHANGES (Entire negative vote was withdrawn with editorial changes) Clearly distinguish editorial changes from the ballot item using "track changes" or provide in a separate list format (attach below). Please do not provide a clean copy of the document. Submission may be made in separate document.

Choose File | No file chosen

NOT PERSUASIVE (Entire negative vote was found not persuasive or there is a combination of not persuasive and not related or withdrawn dispositions)

NOT RELATED (Entire negative vote was found not related or there is a combination of not related and withdrawn dispositions)



# **Springer article**

- o Article "Vitrinite Reflectance Analysis"
- Published December 2021
  - Definition-Introduction
  - Origin of Vitrinite
  - Diagenesis
  - o Catagensis
  - Distinguishing feature
  - Analysis
  - Applications



**Springer Reference** 

Encyclopedia of Petroleum





### **Proposal for New Activities 2022 onwards**

- Photograph round robin to identify vitrinite vs solid bitumen
- A Hilgers Fossil Student round robin on calibrated images
- USGS funding in question ....
- Maintenance of D7708
- Spectral fluorescence standardization
- Broad ion beam sample preparation standardization





## Thanks!

