Introduction to Vitrinite Reflectance as a Thermal Maturity Indicator*

Brian J. Cardott[1]

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[1]Oklahoma Geological Survey, Norman Oklahoma (bcardott@ou.edu)

Abstract

Thermal maturity is one of the most important parameters used in the evaluation of gas-shale and shale-oil plays. Vitrinite reflectance (VRo) is a commonly used thermal maturity indicator. Many operators use the vitrinite-reflectance value without knowing what it is or how it is derived. Conventional wisdom of the Barnett Shale gas play in the Fort Worth Basin indicates the highest gas rates occur at >1.4% VRo. Knowledge of the oil and condensate windows is essential for liquid hydrocarbon production. This presentation answers the questions: what is vitrinite; what is vitrinite reflectance; how is vitrinite reflectance measured; what are some sources of error; and how does one tell good data from bad data?

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Tulsa Geological Society May 8, 2012

Introduction to Vitrinite Reflectance as a Thermal Maturity Indicator

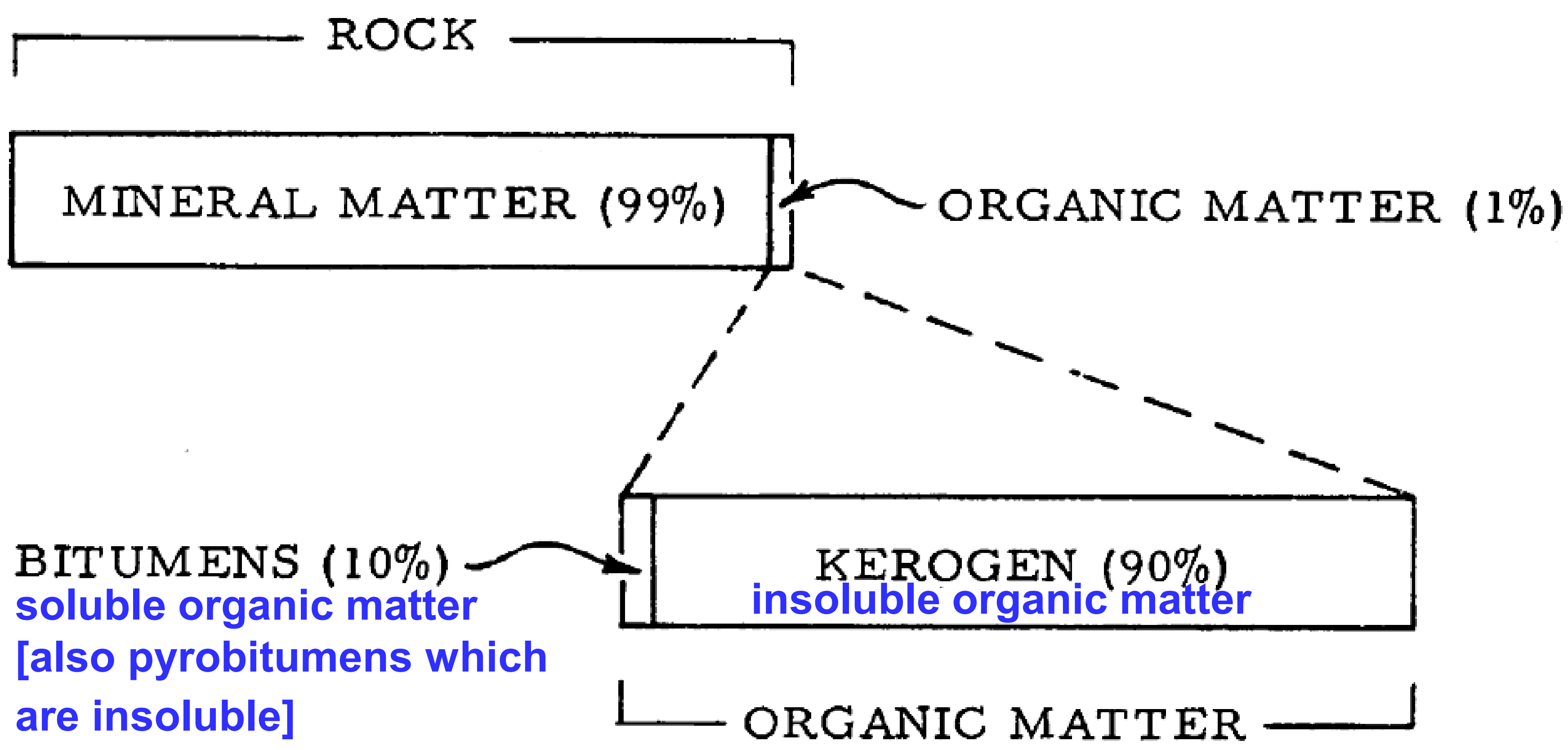
Brian J. Cardott

Oklahoma Geological Survey

Goals of Presentation: Answer the following questions

- What is vitrinite?
- What is vitrinite reflectance?
- How is vitrinite reflectance measured?
- What are some sources of error?
- How do you tell good data from bad data?

Dispersed Organic Matter (DOM) in Shale



Barker, 1979, p. 39

Organic Matter Classifications

MACERAL GROUP (Teichmuller and Ottenjann)	PHYTOCLAST (Bostick)	PHYTOCLAST GROUP (Massoud and Kinghorn)	KEROGEN TYPE (Tissot and Welte; Harwood)	PALYNOLOGICAL KEROGEN (Hunt)
Liptinite (Exinite)	Algal bodies	Keroginite	I	Algal
	Floccules and Groundmass			Amorphous
	Liptinite	Liptinite	II	Herbaceous
Vitrinite	Low-gray Vitrinite	Vitrinite	III	Woody
Inertinite	High-gray Vitrinite and Fusinite	Inertinite	IV	Coaly

Taylor and others, 1998, p. 242-243

MACERAL

(from Latin: "macerare", to soften)

Stopes, 1935

"Macerals are organic substances, or optically homogeneous aggregates of organic substances, possessing distinctive physical and chemical properties, and occurring naturally in the sedimentary, metamorphic, and igneous materials of the earth"

Spackman, 1958

Microscopic Organic Composition (Maceral Classification)

MACERAL GROUP	ORIGIN	REFLECTANCE
VITRINITE	Cell wall material or woody tissue of plants.	Intermediate
LIPTINITE (EXINITE)	Waxy and resinous parts of plants (spores, cuticles, wound resin)	Lowest
INERTINITE	Plant material strongly altered and degraded in peat stage of coal formation.	Highest

Crelling and Dutcher, 1980

Vitrinite occurs in post Silurian- age rocks

Vitrinite Maceral Classification

VITRINITE GROUP (ICCP, 1982)		VITRINITE GROUP (ICCP, 1994)	
Maceral	Submaceral	Maceral	Maceral subgroup
Telinite	Telinite 1 Telinite 2	Telinite	Telovitrinite
Collinite	Telocollinite	Collotelinite	
	Desmocollinite	Collodetrinite	Detrovitrinite
		Vitrodetrinite	
	Corpocollinite	Corpogelinite	Gelovitrinite
	Gelocollinite	Gelinite	
Vitrodetrinite[1]			

[1] Vitrodetrinite is incorporated in the detrovitrinite subgroup (ICCP, 1994)

Potter and others (1998) and ICCP (1998)

International Committee for Coal and Organic Petrology (ICCP) Classification of Dispersed Organic Matter (DOM) (draft) [used in visual kerogen analysis]

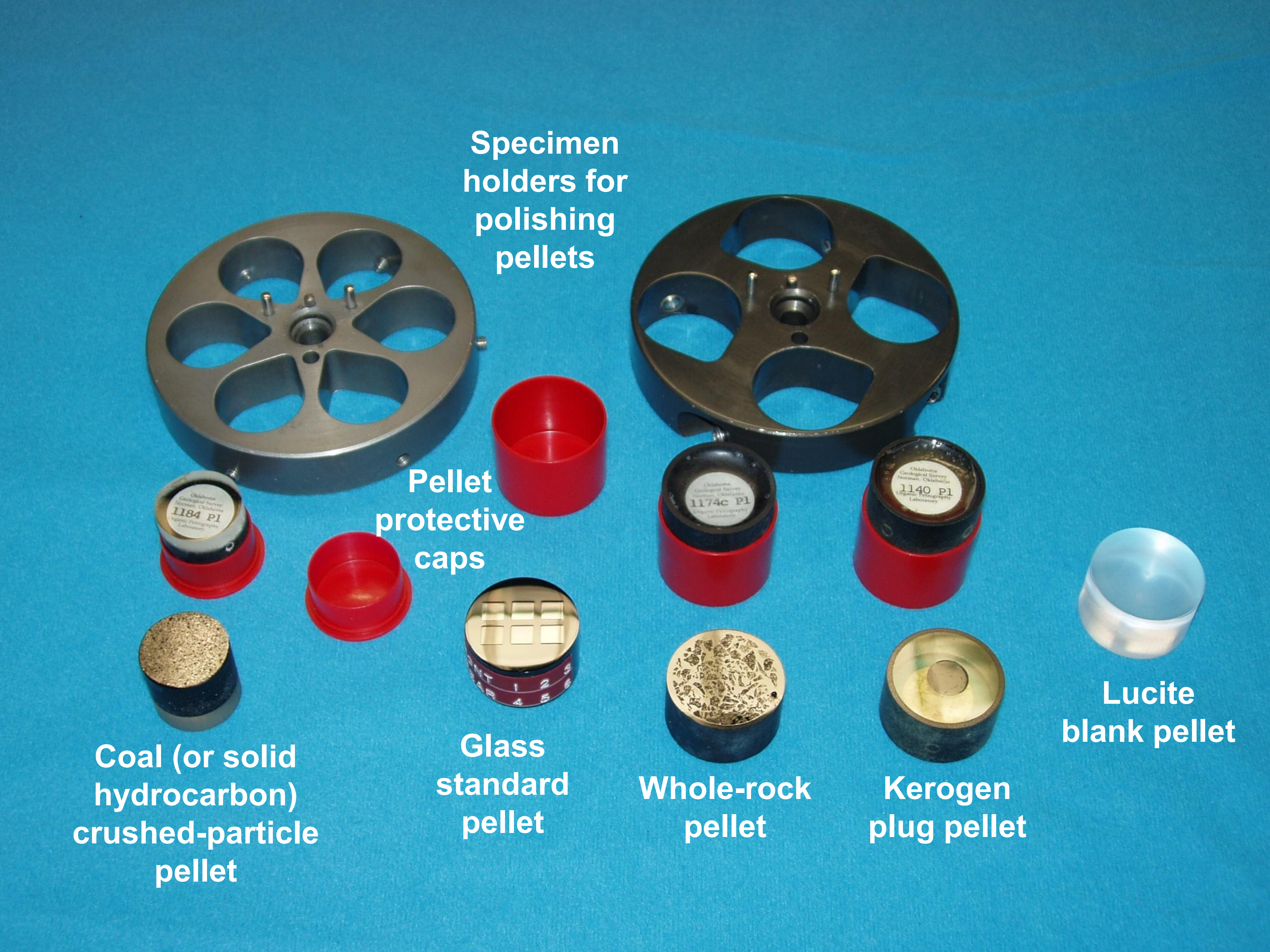
MACERAL GROUP	MACERAL[3]
Vitrinite	Telinite
	Collotelinite
	Vitrodetrinite
	Collodetrinite
	Gelinite
	Corpogelinite
Liptinite	Alginite
	Bituminite/Amorphinite[4]
	Liptodetrinite
	Sporinite
	Cutinite
	Suberinite
	Resinite
	Chlorophyllinite
Inertinite	Fusinite
	Semifusinite
	Funginite
	Macrinite
	Micrinite
	Inertodetrinite
Zooclasts	Scolecodont
	Graptolite
	Chitinozoa
	Foram liners
Secondary Products	Bitumen
	Pyrobitumen
	Oil

The amount of sample needed depends on the sample and how it will be analyzed.

COAL: 30 grams

SHALE (whole rock vs. kerogen concentrate; organic-rich vs. organic lean):

30 to 500 grams

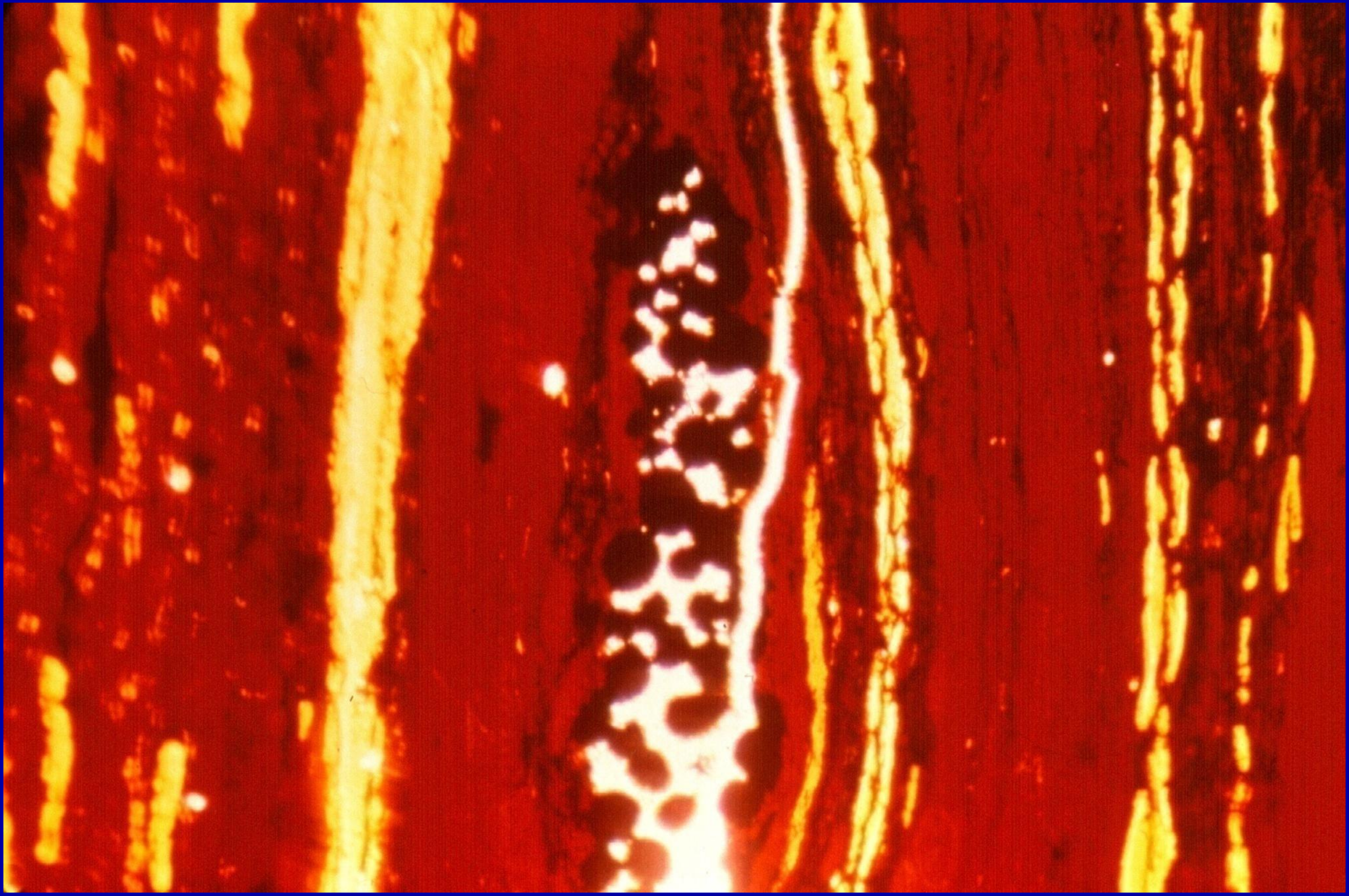

Specimen holders for polishing pellets
Pellet protective caps
Coal (or solid hydrocarbon) crushed-particle pellet
Glass standard pellet
Whole-rock pellet
Kerogen plug pellet
Lucite blank pellet

Whole-rock or kerogen concentrate pellets are prepared using epoxy and allowed to cure over night;

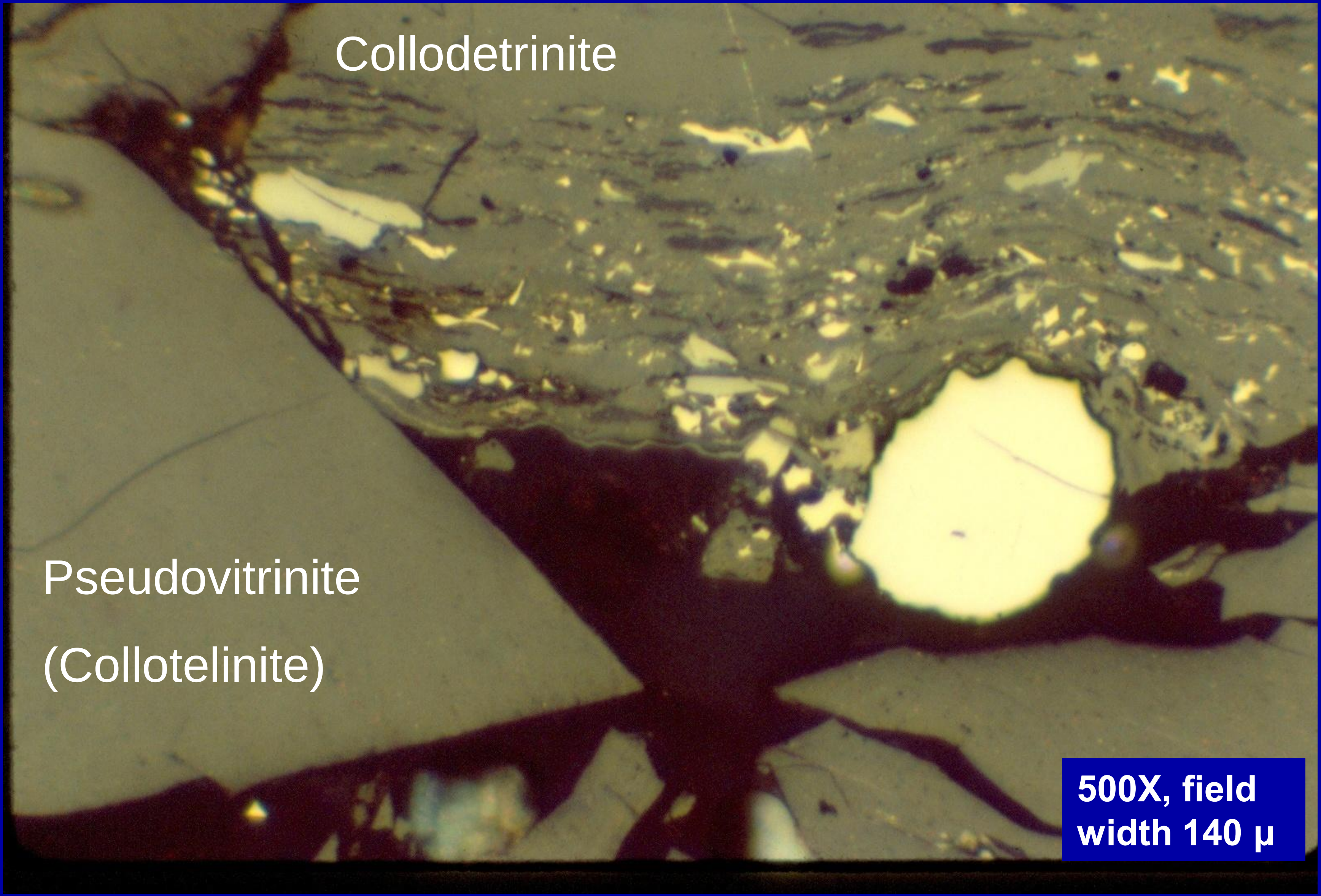
Pellets are polished and placed in a desiccator over night to remove moisture.


Leitz
∞/1×

Coal in Thin Section



Coal in Reflected White Light

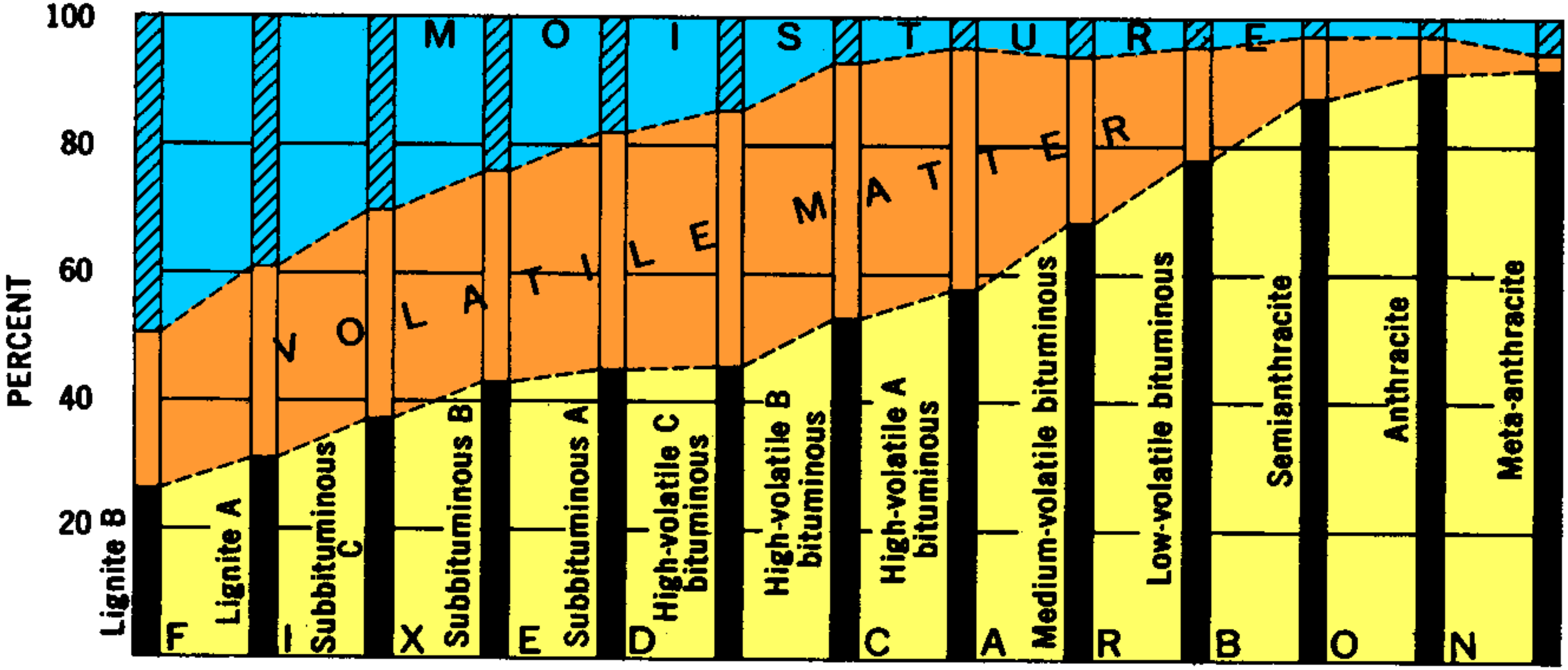


Vitrinite Reflectance is used to determine coal rank and shale thermal maturity.

RANK refers to the physical and chemical changes that occur to organic matter as it is affected by increasing temperature and time.

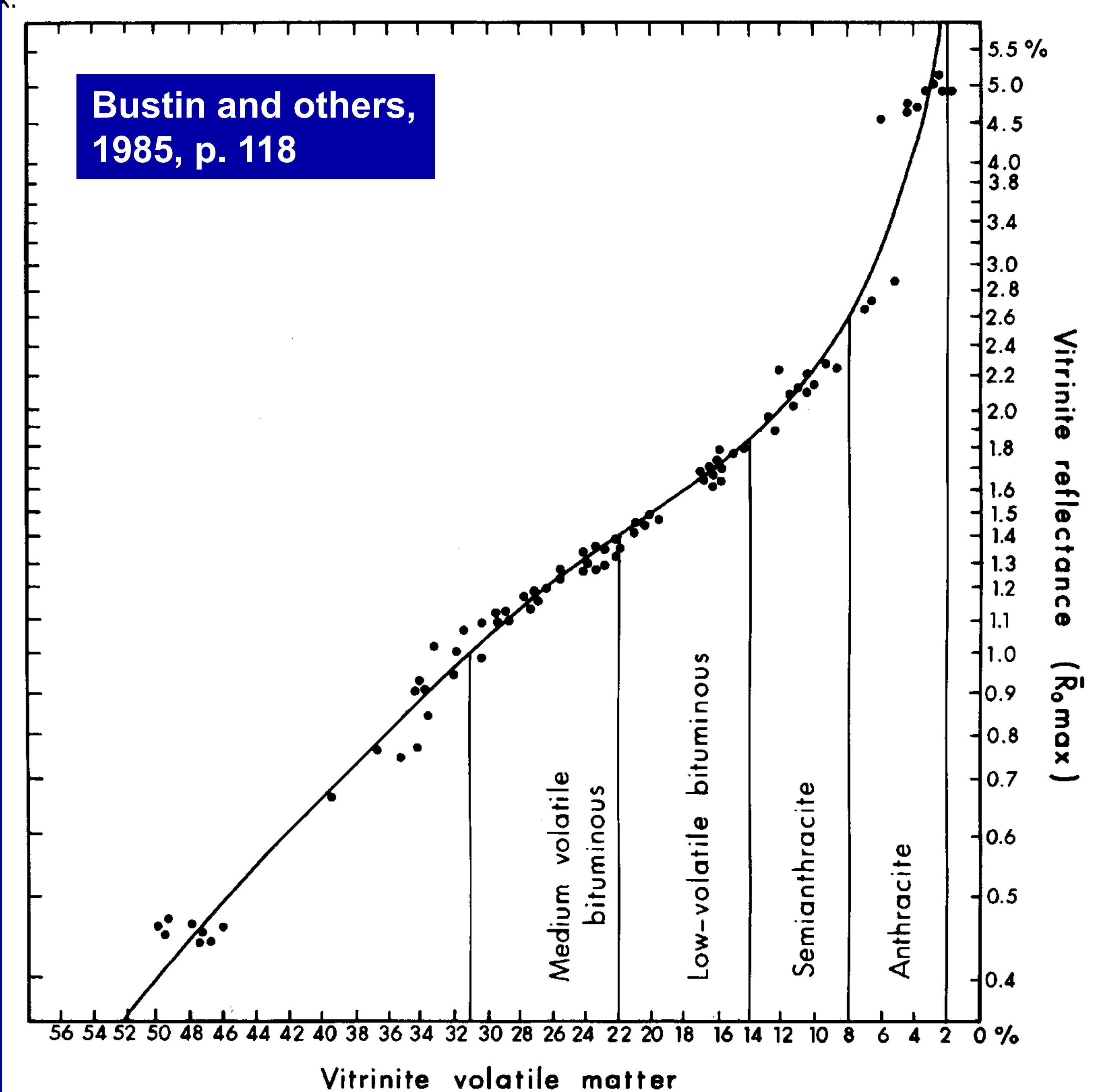
[LIGNITE→SUBBITUMINOUS→ BITUMINOUS (High Volatile; Medium Volatile; Low Volatile)→ ANTHRACITE (Semianthracite; Anthracite; Meta-anthracite)]

COAL RANK FROM PROXIMATE ANALYSIS



Averitt, 1975

Vitrinite Reflectance (%Ro) is a measurement of the percentage of light reflected off the vitrinite maceral at 500X magnification in oil immersion



Bustin and others, 1985, p. 118
Medium volatile bituminous
Low-volatile bituminous
Semianthracite
Anthracite
Vitrinite reflectance ($\bar{R}_o$max)
5.5 %
5.0
4.5
4.0
3.8
3.4
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
56 54 52 50 48 46 44 42 40 38 36 34 32 30 28 26 24 22 20 18 16 14 12 10 8 6 4 2 0 %
Vitrinite volatile matter

American Society for Testing and Materials

Designation: D 2798 – 91

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite in a Polished Specimen of Coal[1]

ASTM, 1994

Designation: D7708 – 11

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the microscopical determination of the reflectance measured in oil of polished surfaces of vitrinite dispersed in sedimentary rocks. This test method can also be used to determine the reflectance of macerals other than vitrinite dispersed in sedimentary rocks.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke

D388 Classification of Coals by Rank

D2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light

D2798 Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal

3. Terminology

3.1 *Definitions*—For definitions of terms, refer to Terminology D121.

3.2 *Abbreviations:*

3.2.1 $R_o$ran—mean random reflectance measured in oil. Other organizations may use other abbreviations for mean random reflectance.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *alginite, n*—a liptinite maceral occurring in structured morphologies, telalginite, and unstructured morphologies, lamalginite.

3.3.2 *bituminite, n*—an amorphous primary liptinite maceral with low reflectance, occasionally characterized by colored internal reflections and weak orange-brown fluorescence, derived from bacterial biomass and the bacterial decomposition of algal material and faunal plankton. Bituminite is equivalent to the amorphous organic matter recognized in strew slides of concentrated kerogen (**1**).[3]

3.3.2.1 *Discussion*—Bituminite may be distinguished from vitrinite by lower reflectance, as well as higher fluorescence intensity if fluorescence is present in vitrinite. Bituminite has poorly-defined wispy boundaries and may be speckled or unevenly colored whereas vitrinite has distinct boundaries and is blockier and evenly colored. The occurrence of bituminite in association with lamalginite and micrinite is common. Rock type, thermal maturity, and geologic occurrence can be used to interpret the potential presence of bituminite; for example, bituminite may be expected to occur in lacustrine or marine settings. It is less commonly present in fluvial or similar proximal depositional environments, where vitrinite may be expected to occur in greater abundance.

3.3.3 *chitinozoan, n*—a group of flask-shaped, sometimes ornamented marine microfossils of presumed metazoan origin which are composed of 'pseudochitin' proteinic material and which occur individually or in chains. Chitinozoan cell walls are thin, opaque to translucent, and range from dark gray to white in reflected white light similar to vitrinite. Chitinozoans are common in Ordovician to Devonian marine shales.

3.3.4 *conodont, n*—the phosphatic, tooth-like remains of marine vertebrate worm-like animals present from the Cambrian through Triassic, composed predominantly of apatite with subordinate amounts of organic matter. Conodont morphology is variable, but often well-defined denticles and blades are preserved. In reflected white light examination conodonts range from pale yellow to light brown to dark brown and to black.

3.3.5 *fusinite, n*—an inertinite maceral distinguished principally by the preservation of some feature(s) of the plant cell wall structure, high relief, and reflectance substantially higher than first cycle vitrinite in the same sample. When less than

[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.28 on Petrographic Analysis of Coal and Coke.

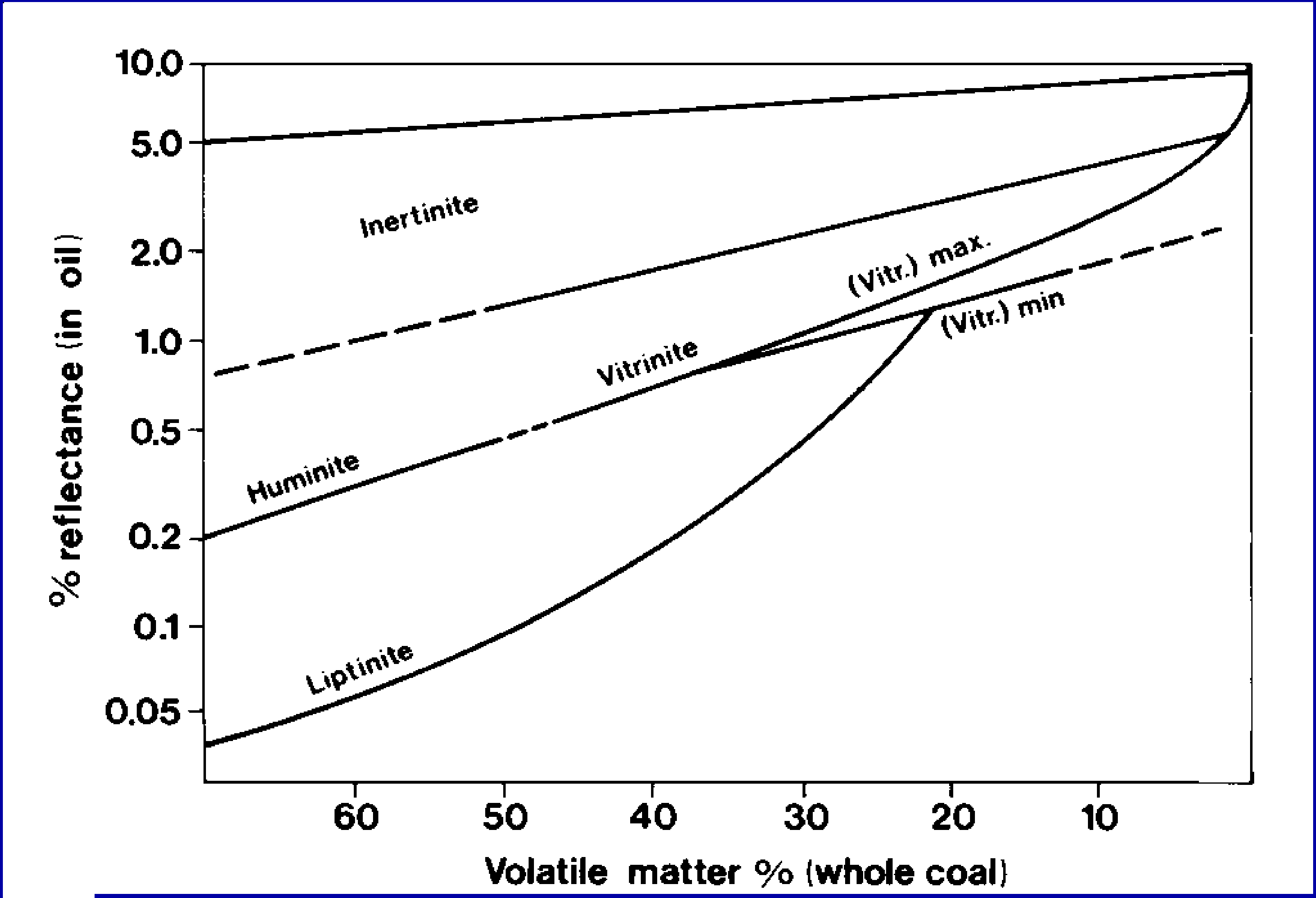
Current edition approved April 1, 2011. Published April 2011. DOI: 10.1520/D7708–11.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

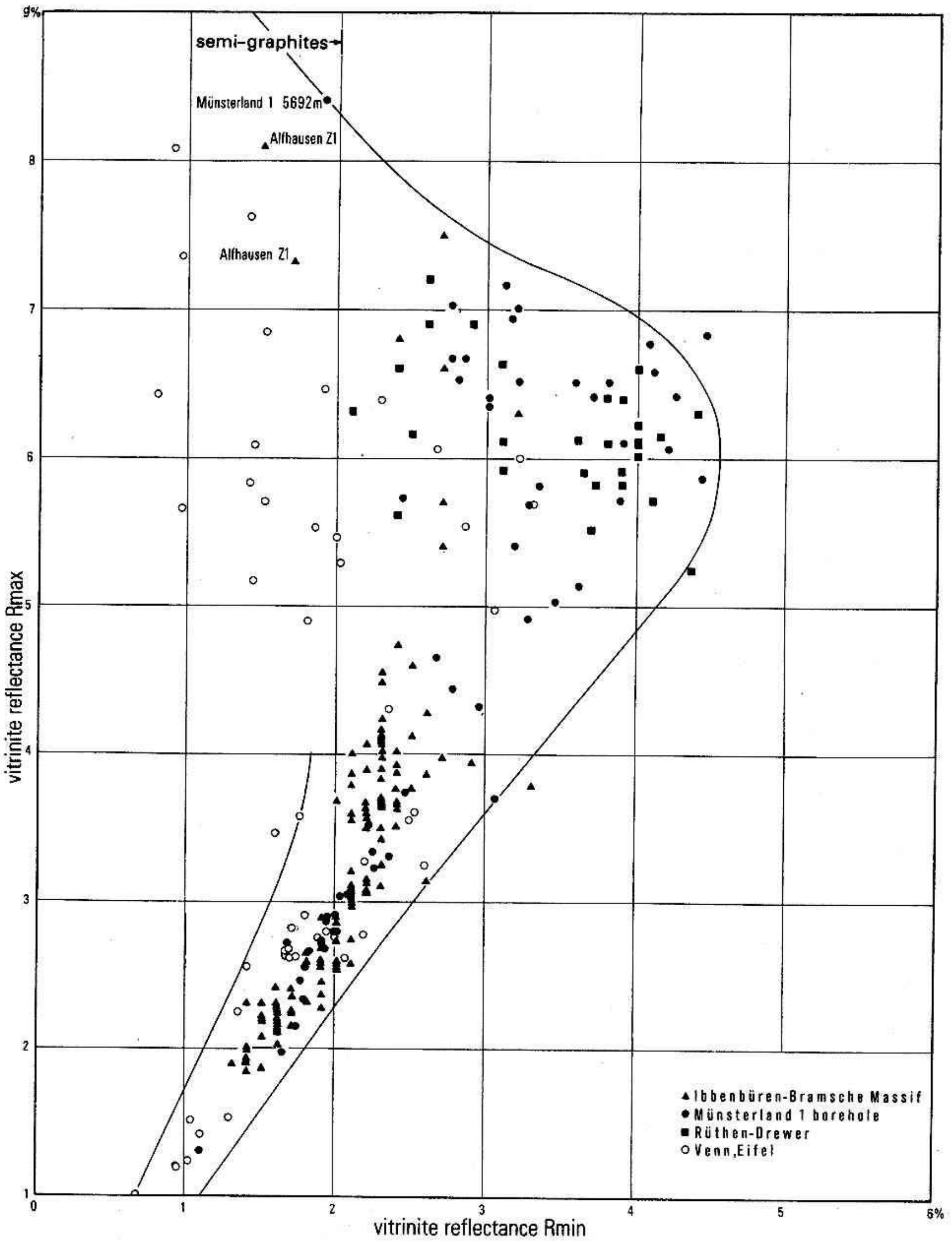
[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

ASTM, 2011

Coalification Curves

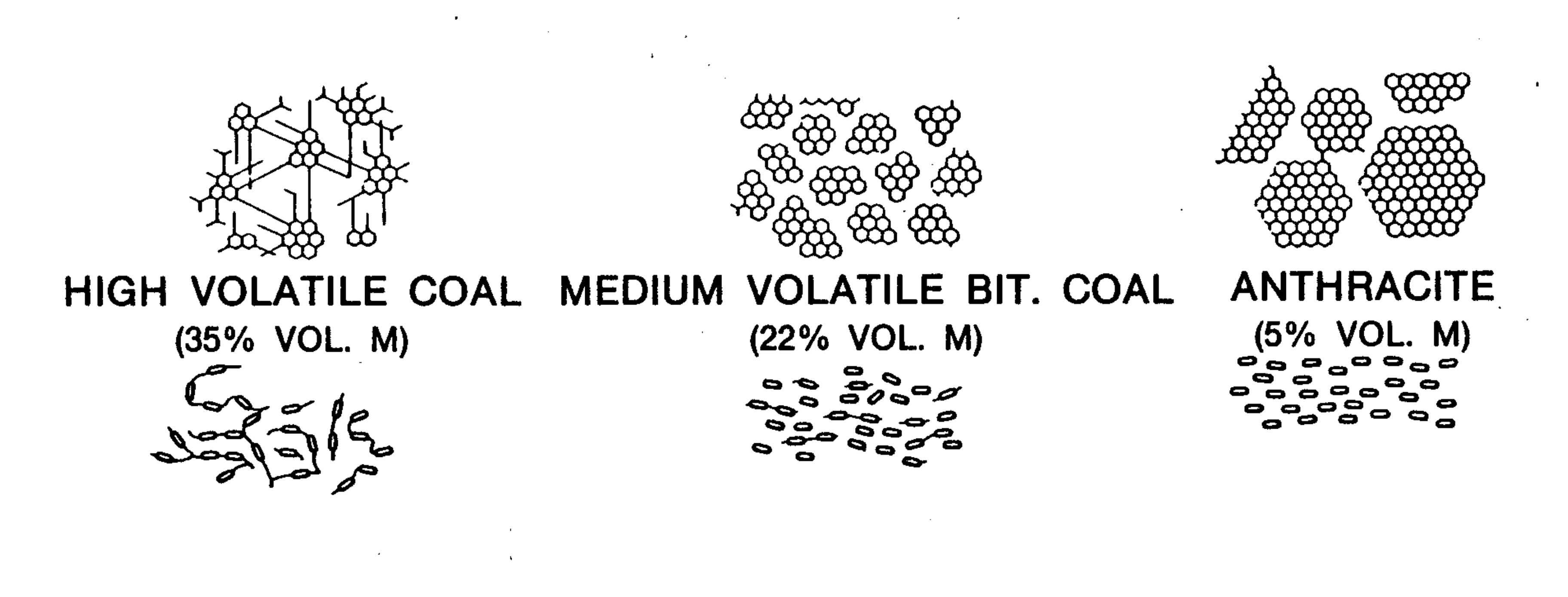


Alpern and Lemos de Sousa, 1970, in Tissot and Welte, 1984, p. 243.



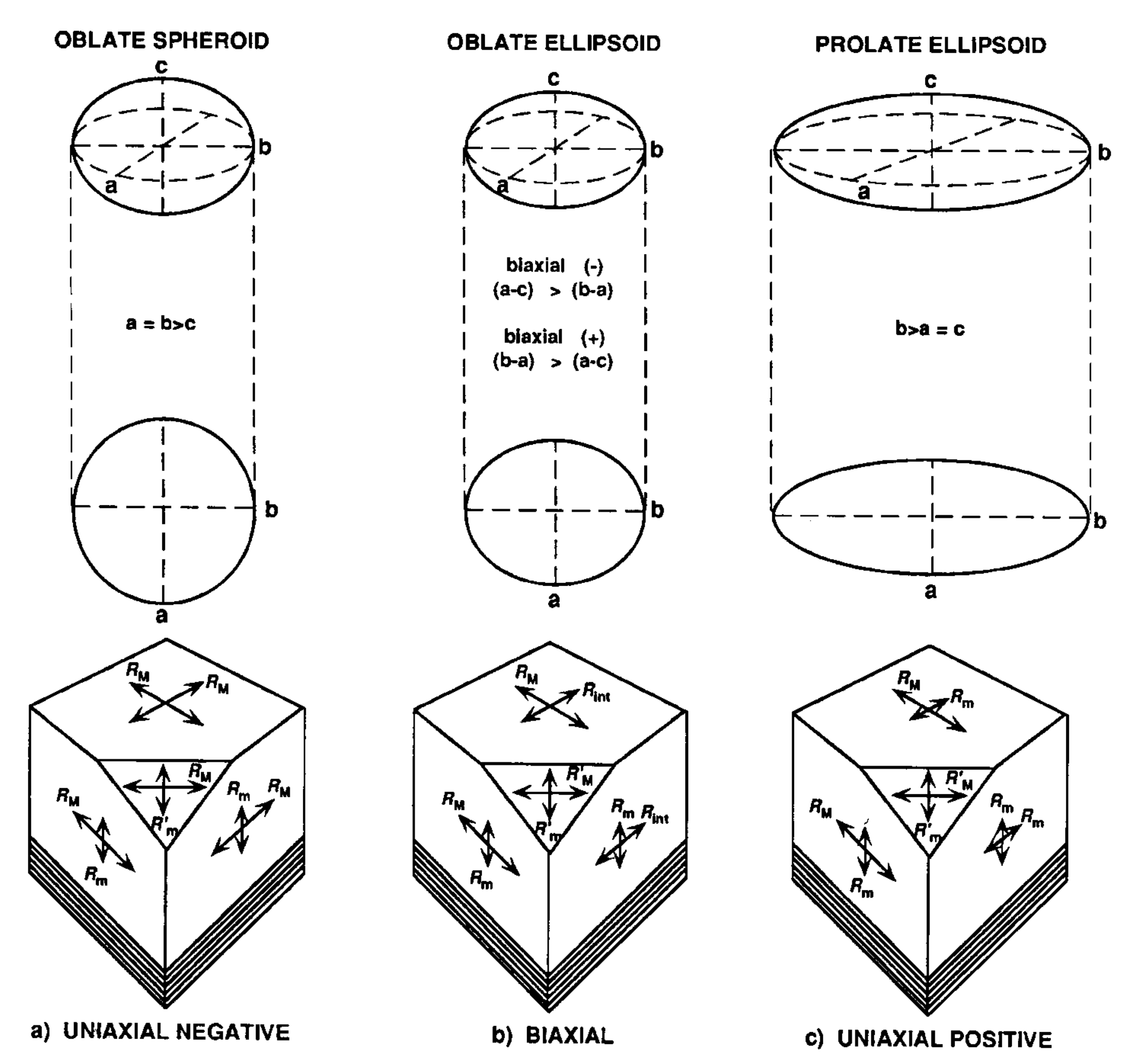
Stach and others, 1982

Physical, Chemical, and Molecular Changes of Vitrinite



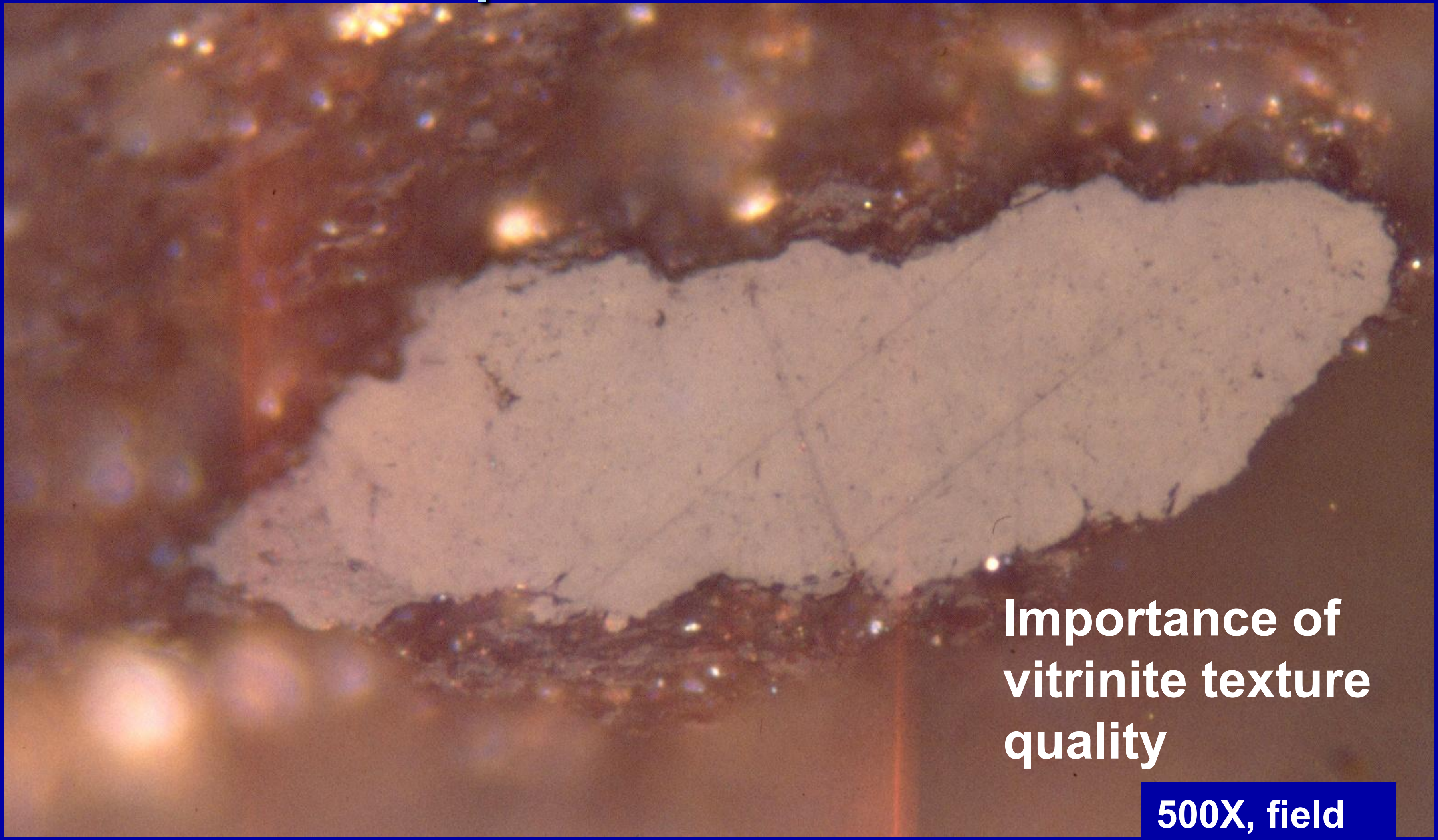
Taylor and others, 1998

Reflectance Indicatrix



Taylor and others, 1998

Dispersed Vitrinite



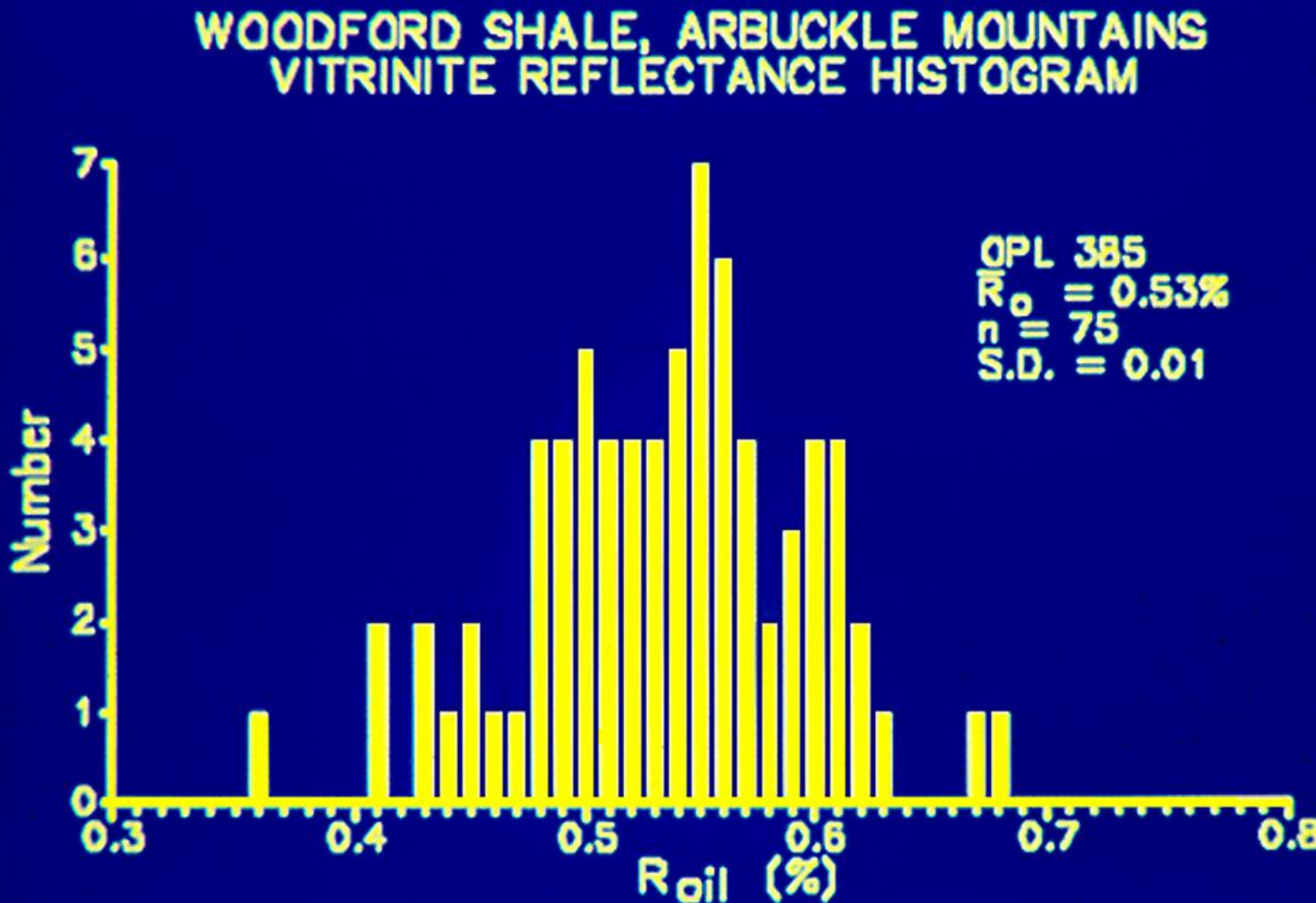
Importance of vitrinite texture quality

500X, field width 140 μ

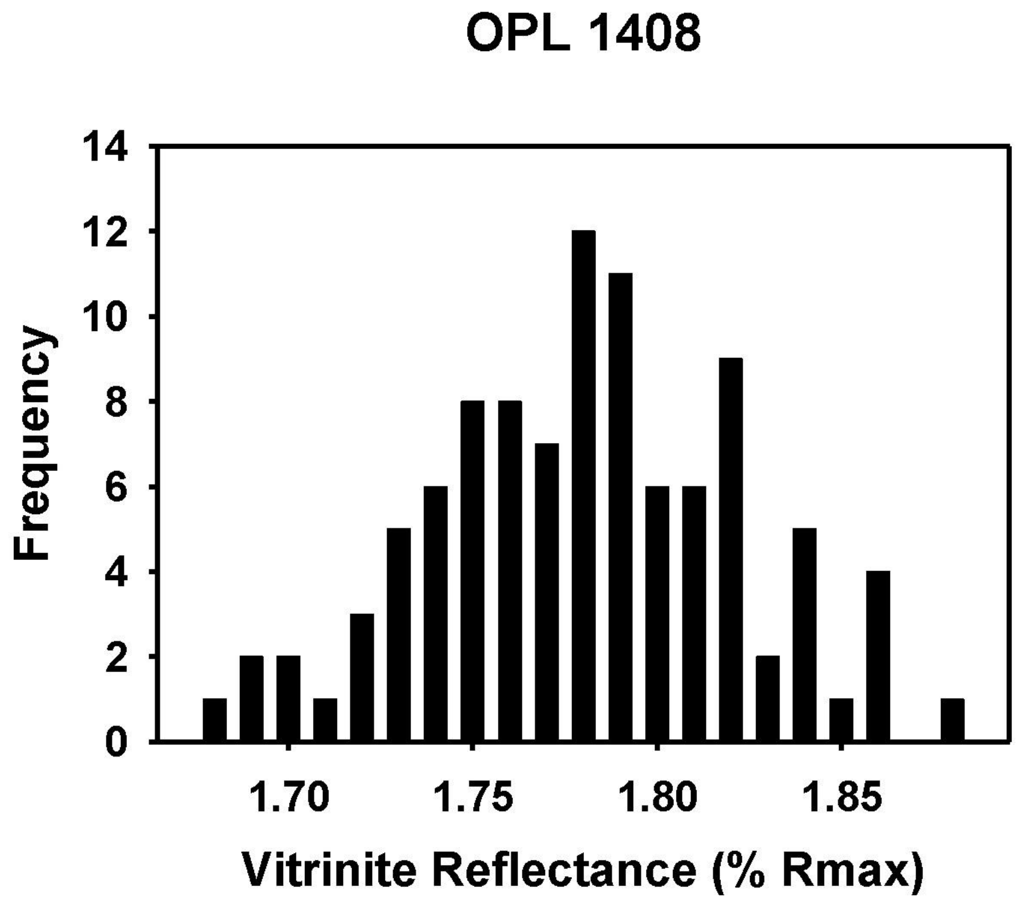
PETROGRAPHER	VRo	SAMPLE IDENTIFICATION
DATE	[mean____________] nonpolarized light	PELLET NUMBER

V		PV	V		PV	V		PV	V		PV	V		PV
	0.30			0.60			0.90			1.20			1.50	
	0.31			0.61			0.91			1.21			1.51	
	0.32			0.62			0.92			1.22			1.52	
	0.33			0.63			0.93			1.23			1.53	
	0.34			0.64			0.94			1.24			1.54	
	0.35			[illegible]			[illegible]			[illegible]			1.55	
	0.36			[illegible]			[illegible]			[illegible]			1.56	
	0.37			[illegible]			[illegible]			[illegible]			1.57	
	0.38			[illegible]			[illegible]			[illegible]			1.58	
	0.39			0.69			0.99			1.29			1.59	
	0.40			0.70			1.00			1.30			1.60	
	0.41			0.71			1.01			1.31			1.61	
	0.42			0.72			1.02			1.32			1.62	
	0.43			0.73			1.03			1.33			1.63	
	0.44			0.74			1.04			1.34			1.64	
	0.45			0.75			1.05			1.35			1.65	
	0.46			0.76			1.06			1.36			1.66	
	0.47			0.77			1.07			1.37			1.67	
	0.48			0.78			1.08			1.38			1.68	
	0.49			0.79			1.09			1.39			1.69	
	0.50			0.80			1.10			1.40			1.70	
	0.51			0.81			1.11			1.41			1.71	
	0.52			0.82			1.12			1.42			1.72	
	0.53			0.83			1.13			1.43			1.73	
	0.54			0.84			1.14			1.44			1.74	
	0.55			0.85			1.15			1.45			1.75	
	0.56			0.86			1.16			1.46			1.76	
	0.57			0.87			1.17			1.47			1.77	

Precision to 0.01%


WOODFORD SHALE, ARBUCKLE MOUNTAINS
VITRINITE REFLECTANCE HISTOGRAM
OPL 385
$\bar{R}_o$ = 0.53%
n = 75
S.D. = 0.01
Number
0
1
2
3
4
5
6
7
0.3
0.4
0.5
0.6
0.7
0.8
R_{oil} (%)

Maximum Vitrinite Reflectance of Coal Stringer from Woodford Shale Core



Rmax = 1.78%
n = 100

PROBLEMS IN OBTAINING TRUE Ro MATURITIES

PROPERLY IDENTIFIED VITRINITE

- Primary
- Recycled
- Caving
- Mud additives
- Subtypes vary Ro (<0.5)

FACTORS AFFECTING ACCURATE Ro MEASUREMENT

- Rough textured vitrinite
  - Weathered
  - Partially dissolved (pitted)
  - Fractured
- Oxidized vitrinite
- Inclusions
  - Pyrite
  - Bitumen
  - Other macerals
- Oily vitrinite
- Natural coking
- Too few readings (<20)

MATERIAL WHICH MAY LOOK LIKE VITRINITE

- Solid bitumen (several types)
- Pseudovitrinite
- Semifusinite

Modified from Dow and O'Connor, 1982

Vitrinite-like organic matter

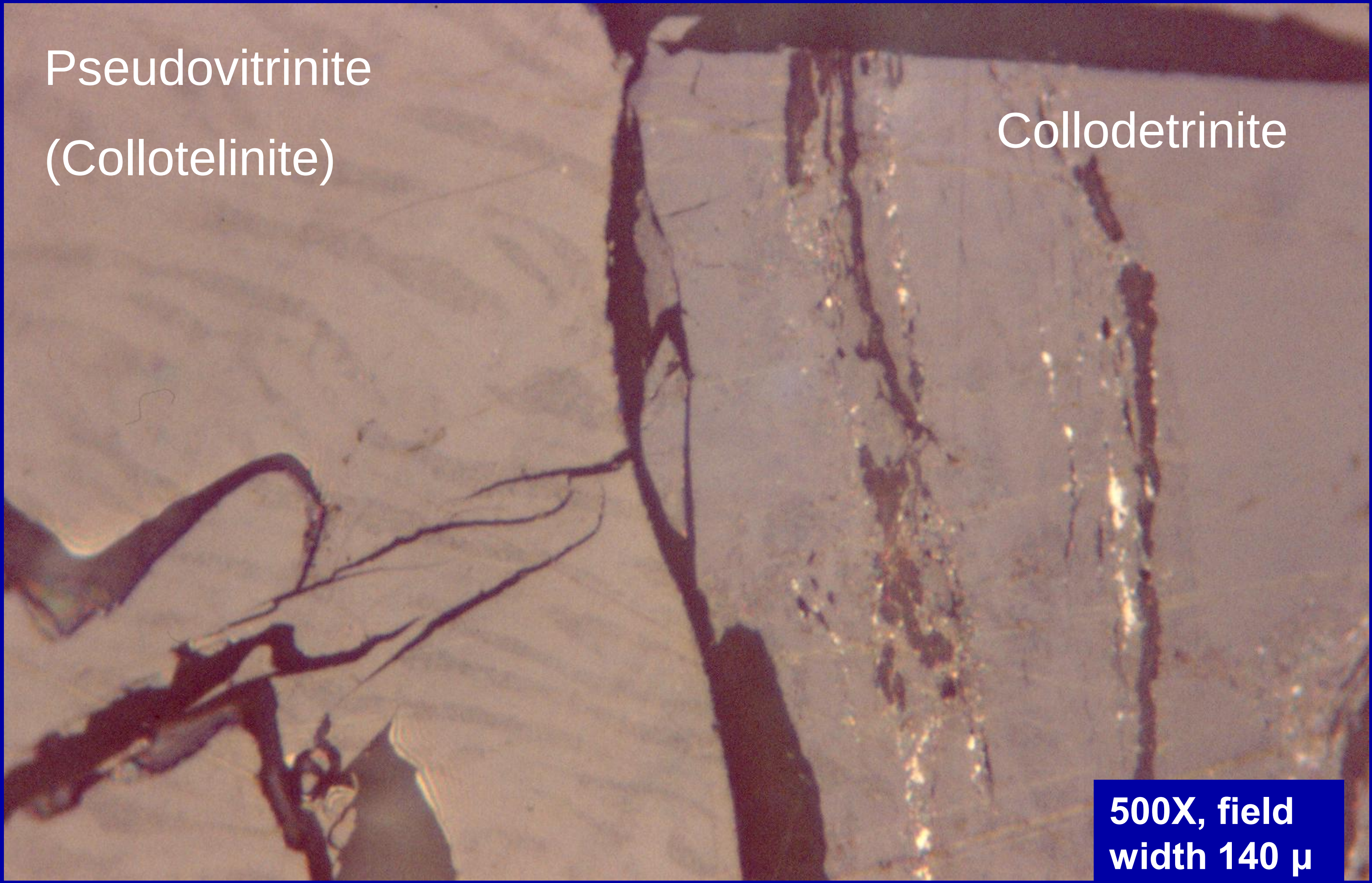
Vitrinite subtypes

Inertinite macerals

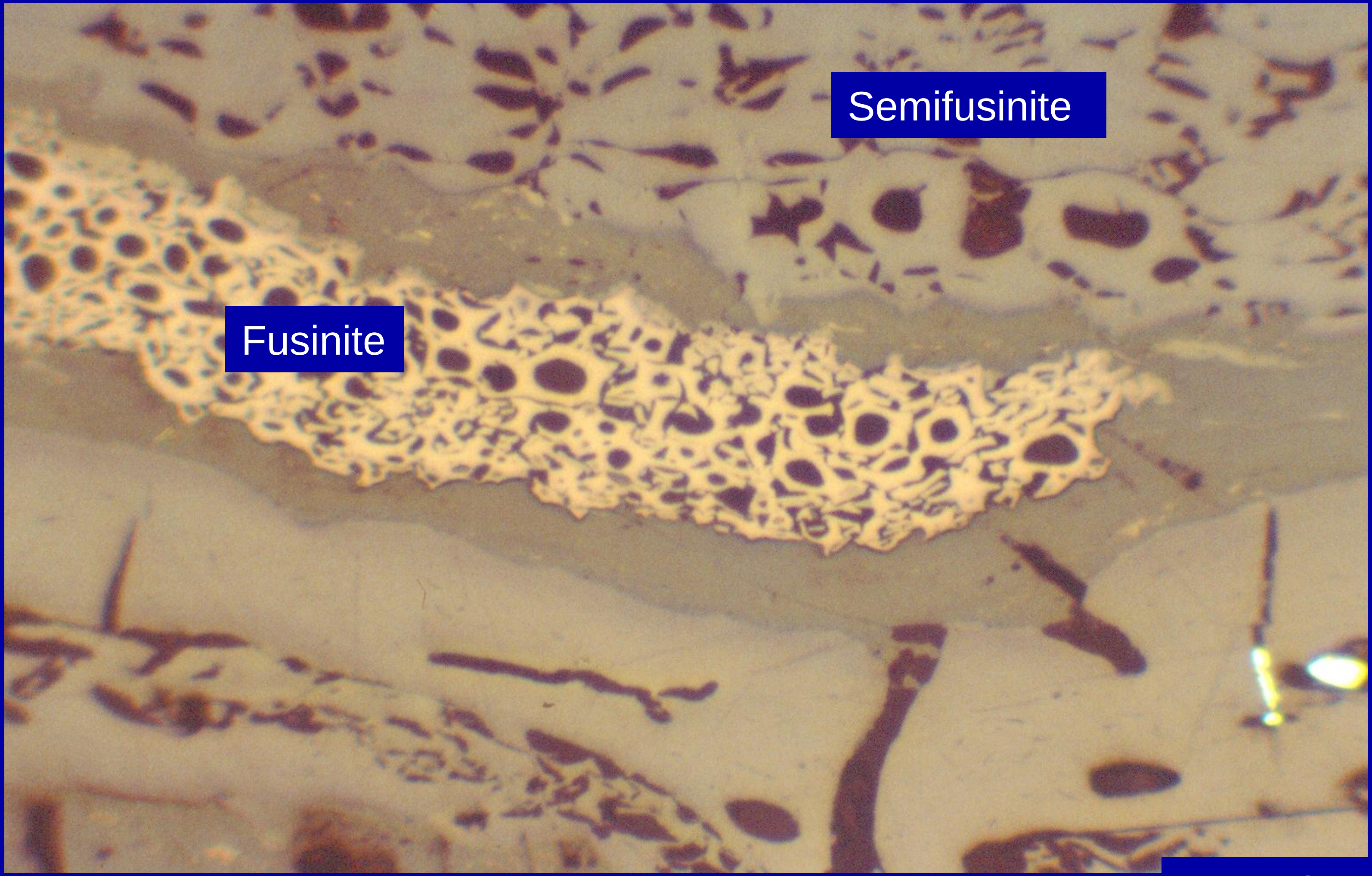
Solid bitumen (several types)

Graptolites

Vitrinite Subtypes



Inertinite Macerals



200X, field width 320 μ

Genetic Bitumen Classification

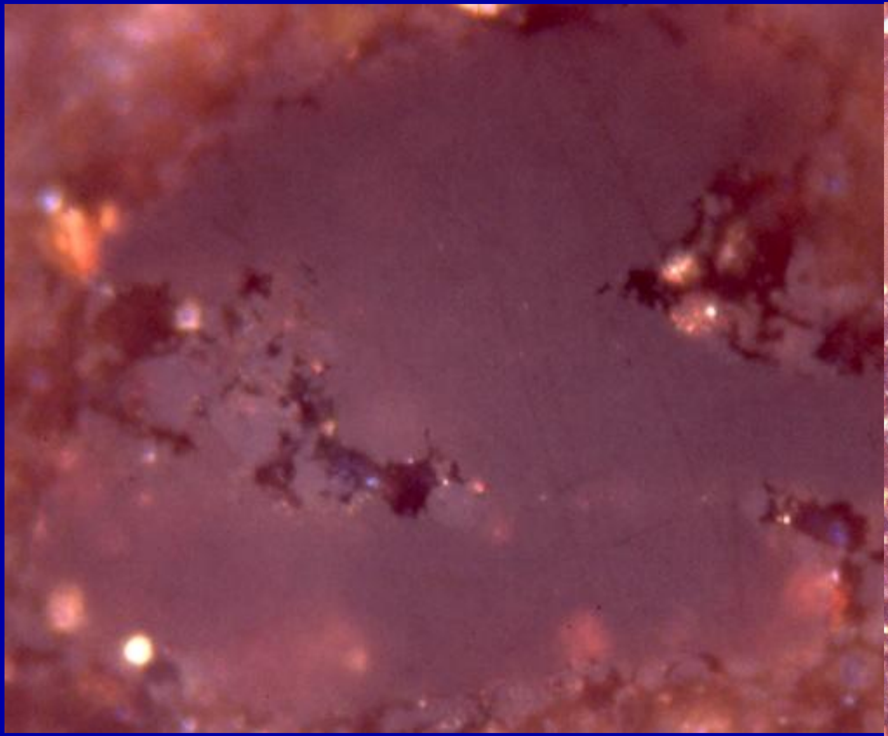
- **Pre-Oil Solid Bitumen**: early-generation products of rich source rocks, probably extruded from their sources as a very viscous fluid, and migrated the minimum distance necessary to reach fractures and voids in the rock. [Kerogen → Bitumen → Oil]
- **Post-Oil Solid Bitumen**: products of the alteration of a once-liquid crude oil, generated and migrated from a conventional oil source rock, and subsequently degraded. [solid residue of primary oil migration]

Curiale (1986)

Two Common Pre-Oil Bitumen Optical Forms Based on Landis and Castaño (1994)

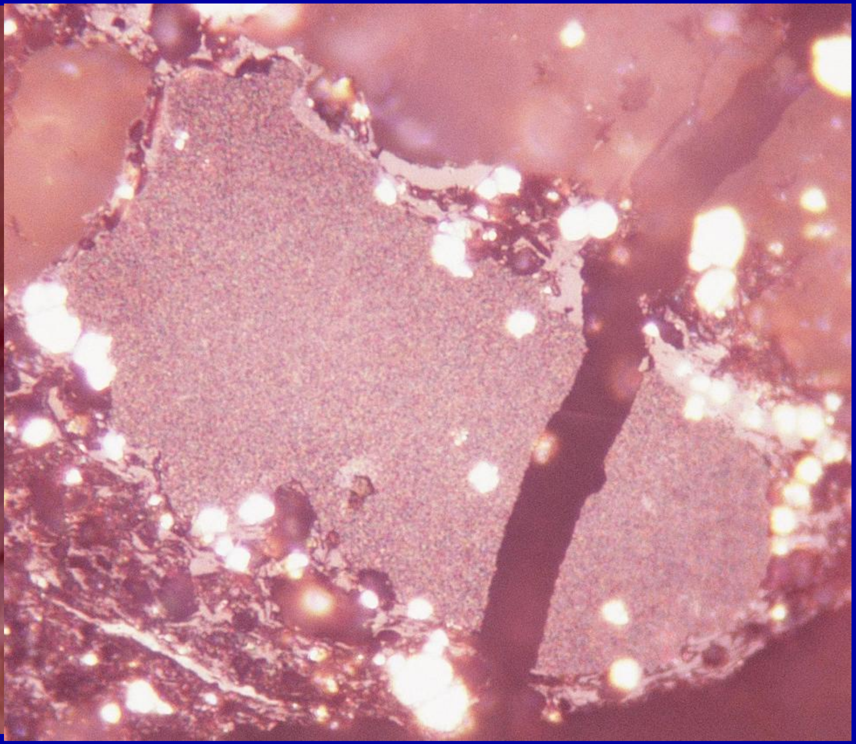
[regression equation is based on homogenous form]

Homogenous form



OPL 1333 500X

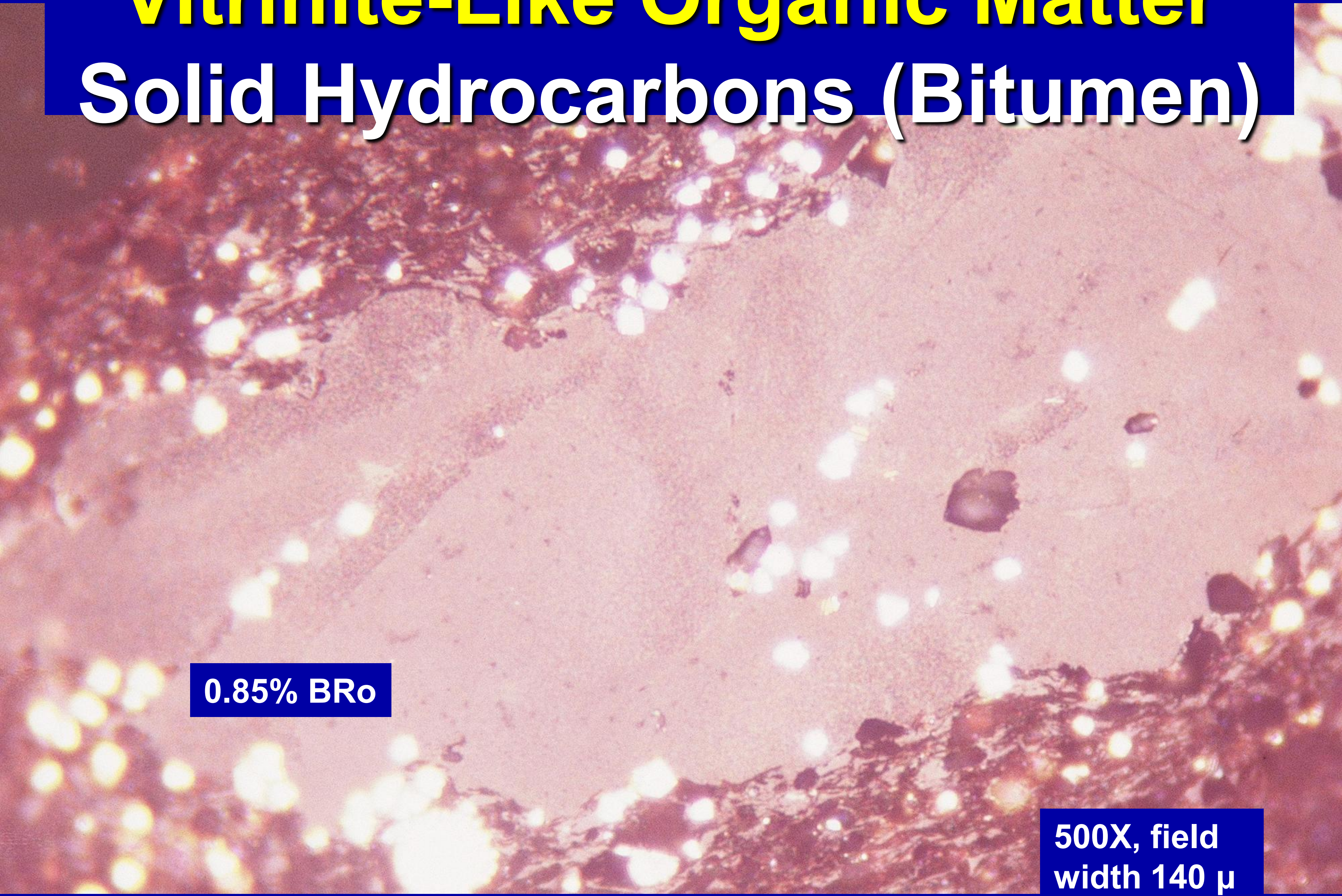
Granular form



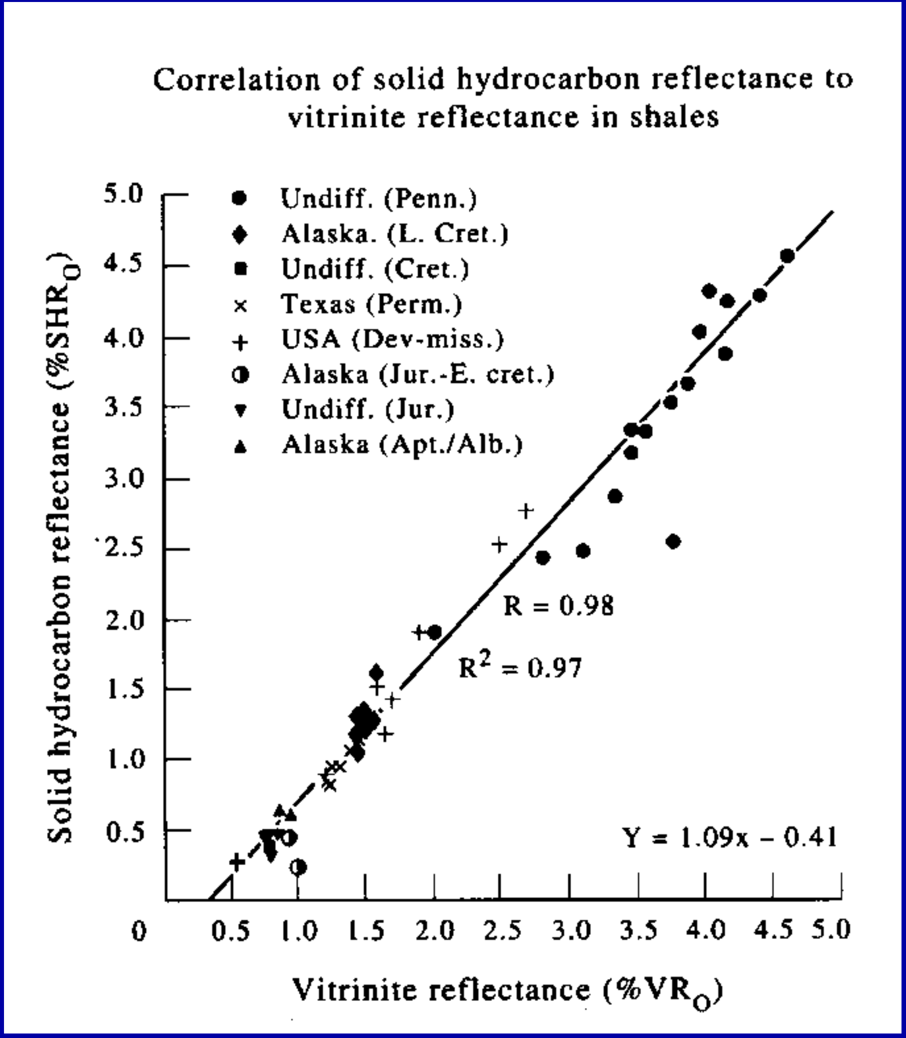
OPL 1076 500X

Vitrinite-Like Organic Matter

Solid Hydrocarbons (Bitumen)



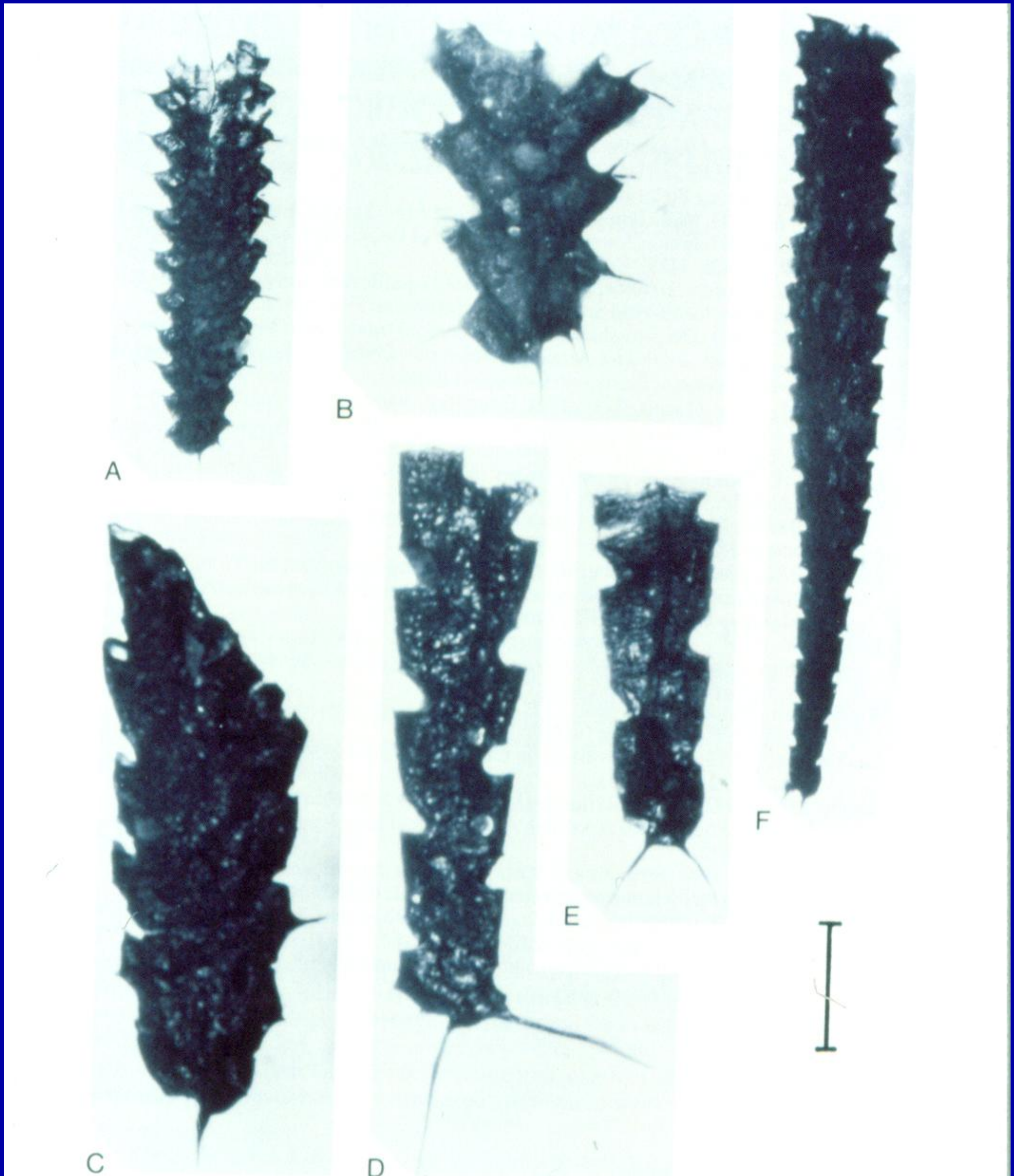
Use of pre-oil solid bitumen as **thermal maturity indicator** following "solid hydrocarbon" reflectance to vitrinite reflectance equivalent regression equation of Landis and Castaño (1994)



$$VRE = (BRo + 0.41)/1.09$$

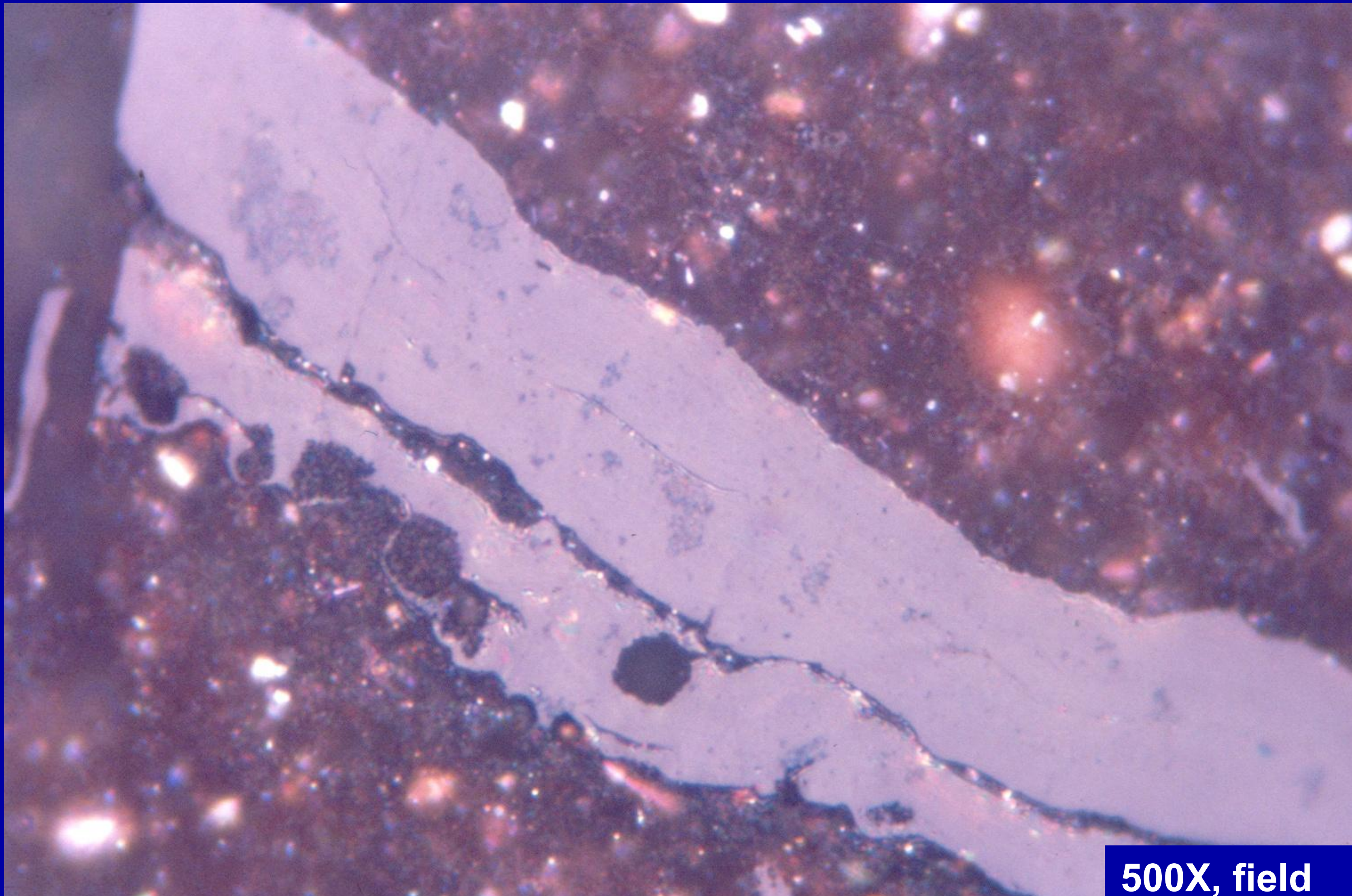
For additional references visit http://www.tsop.org/refs/bitref.htm

Vitrinite-Like Organic Matter



Graptolites

For additional references visit http://www.tsop.org/refs/zooclast.htm



Cardott and Kidwai, 1991

VITRINITE-REFLECTANCE ANALYSIS
SOURCES OF ERROR

- **Samples**
- **Equipment**

Samples are Everything
(Garbage In = Garbage Out)

SAMPLES

- SAMPLE TYPE
- LITHOLOGY
- SAMPLE HANDLING
- ORGANIC MATTER

SAMPLES

- **SAMPLE TYPE** (core, outcrop, well cuttings)
- **LITHOLOGY** (coal, shale, siltstone, sandstone)
- **SAMPLE HANDLING** (oil-based drilling mud, kerogen isolation, oxidation, heating)
- **ORGANIC MATTER** (quantity, quality, size, type, thermal maturity, reflectance suppression/enhancement)

CORE >> **OUTCROP** > **WELL CUTTINGS**

(Weathering) (Caving)

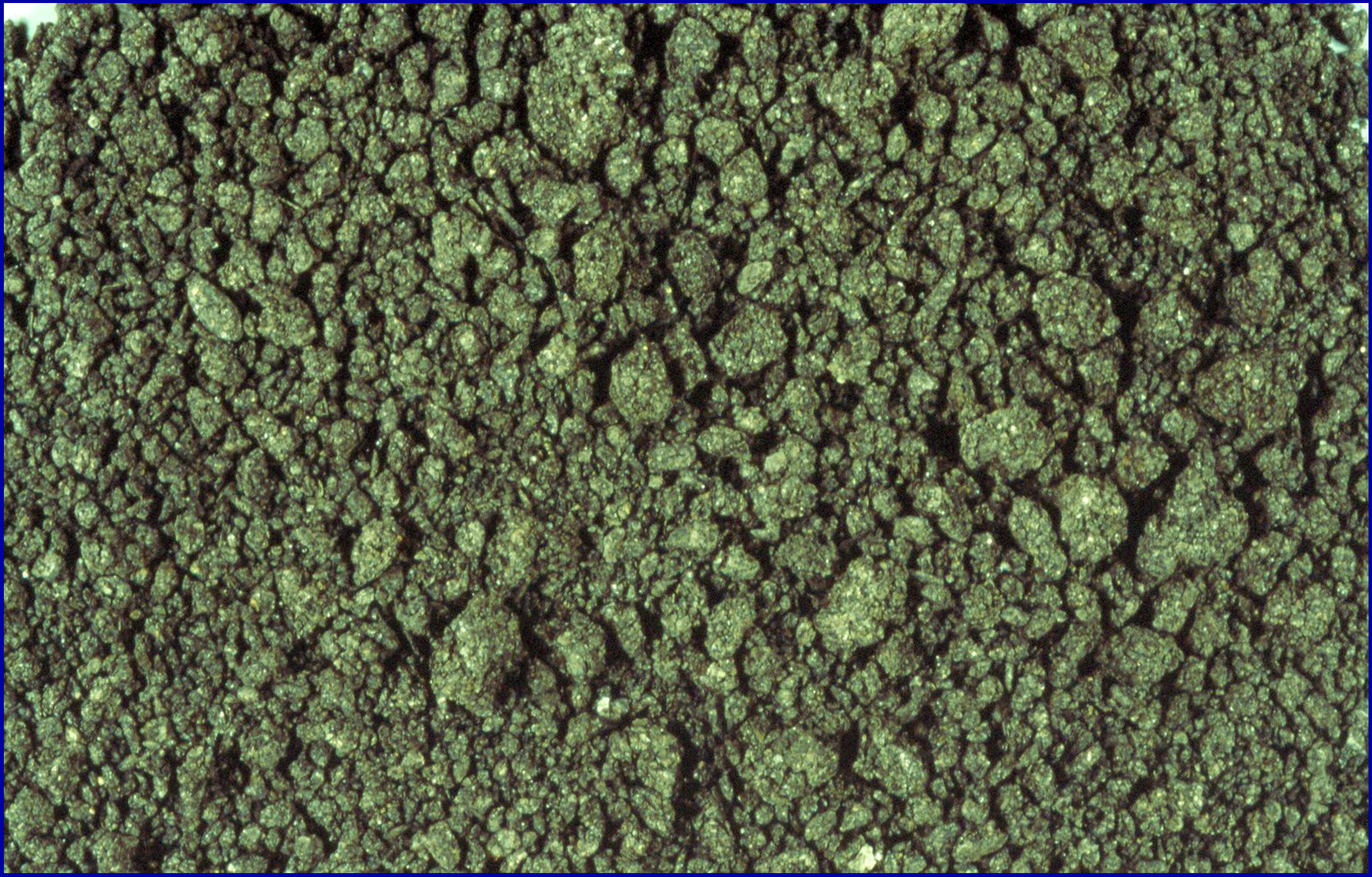
└——— (Recycled Vitrinite) ———┘

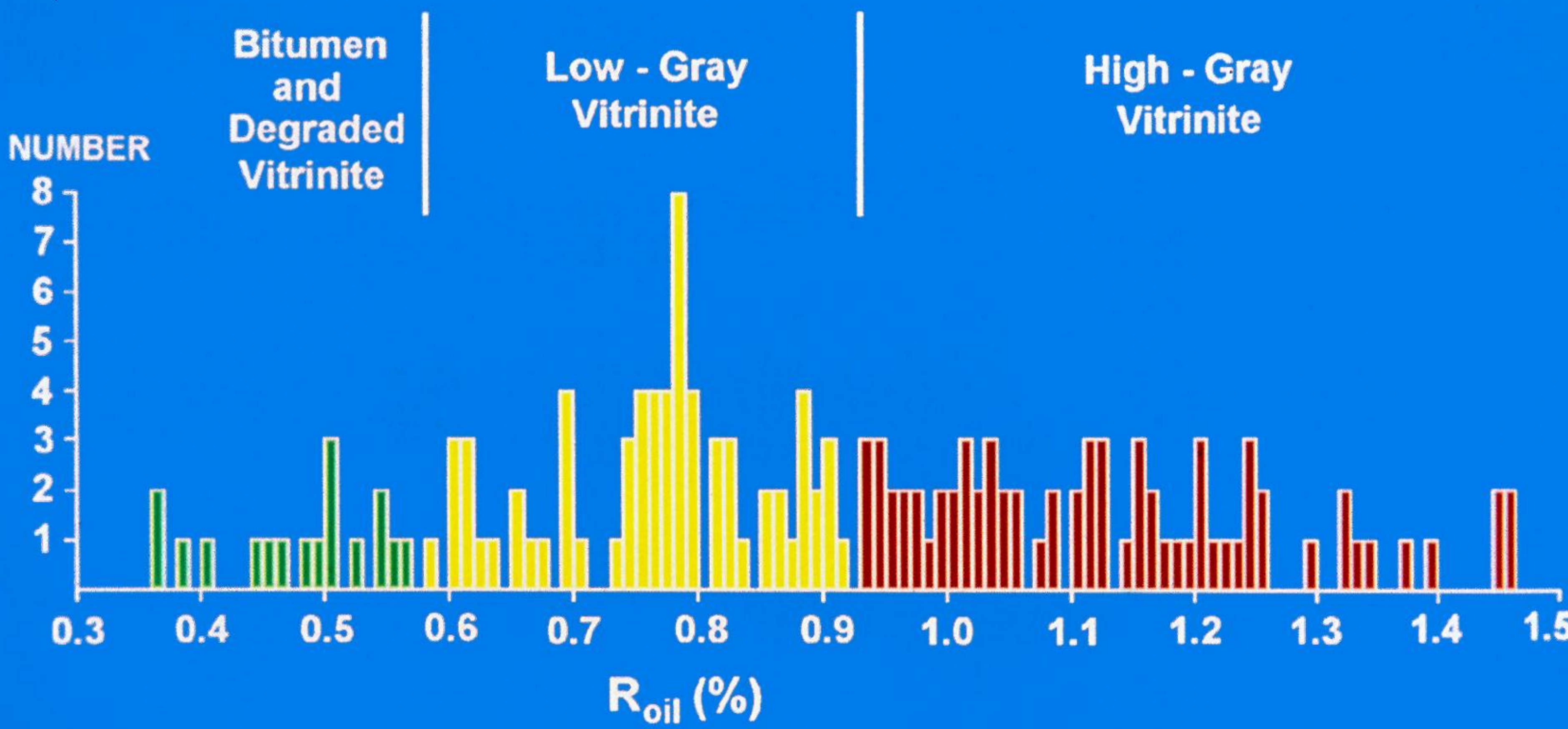
└——— (Vitrinite - Like Organic Matter) ———┘


Drilling Mud
Additive

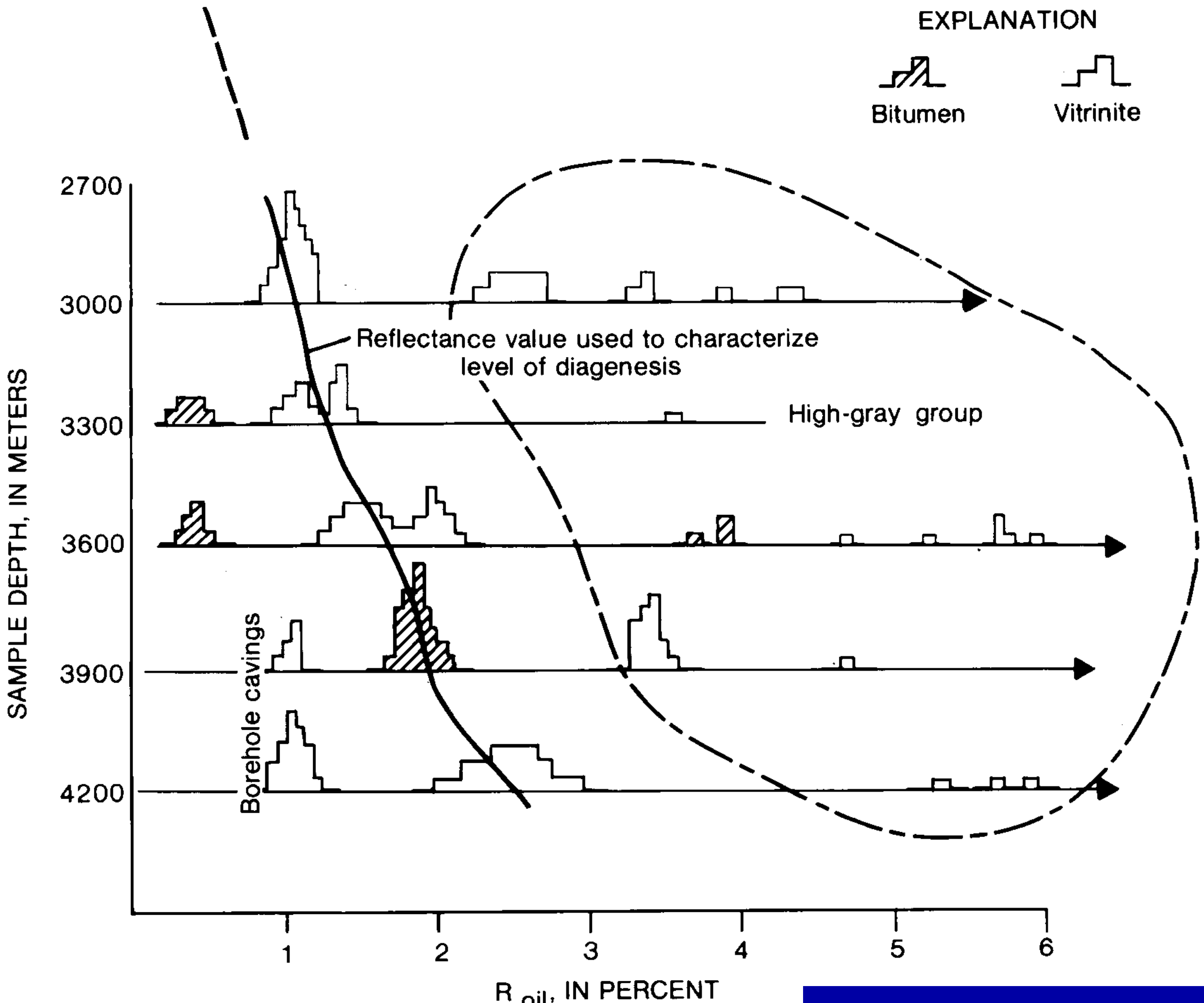
Caving Contamination

Oil-Based Mud





Cardott, 1994



Bostick, 1979

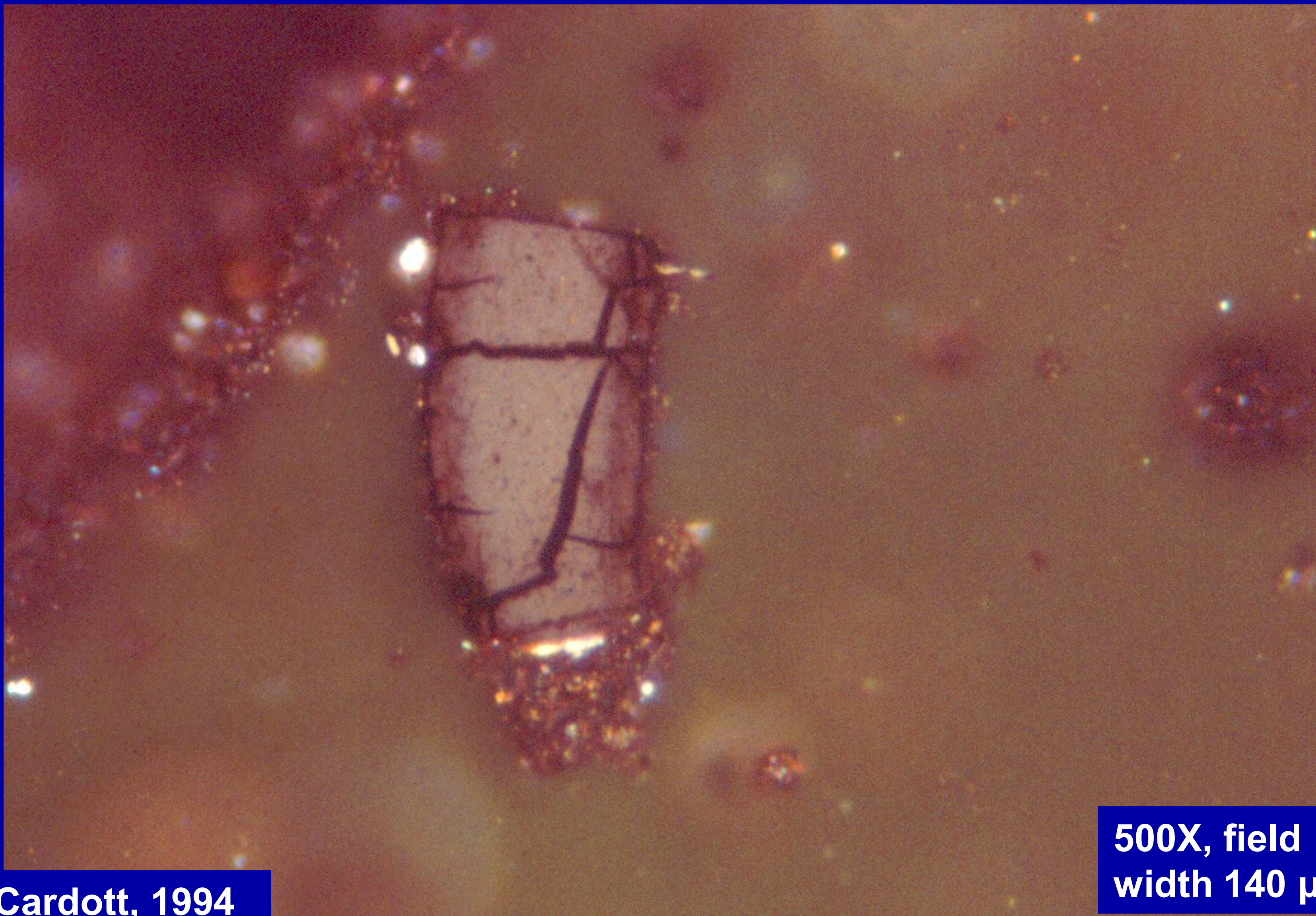
Weathered Coal

For additional references visit http://www.tsop.org/refs/weath.htm

Lo and Cardott, 1994

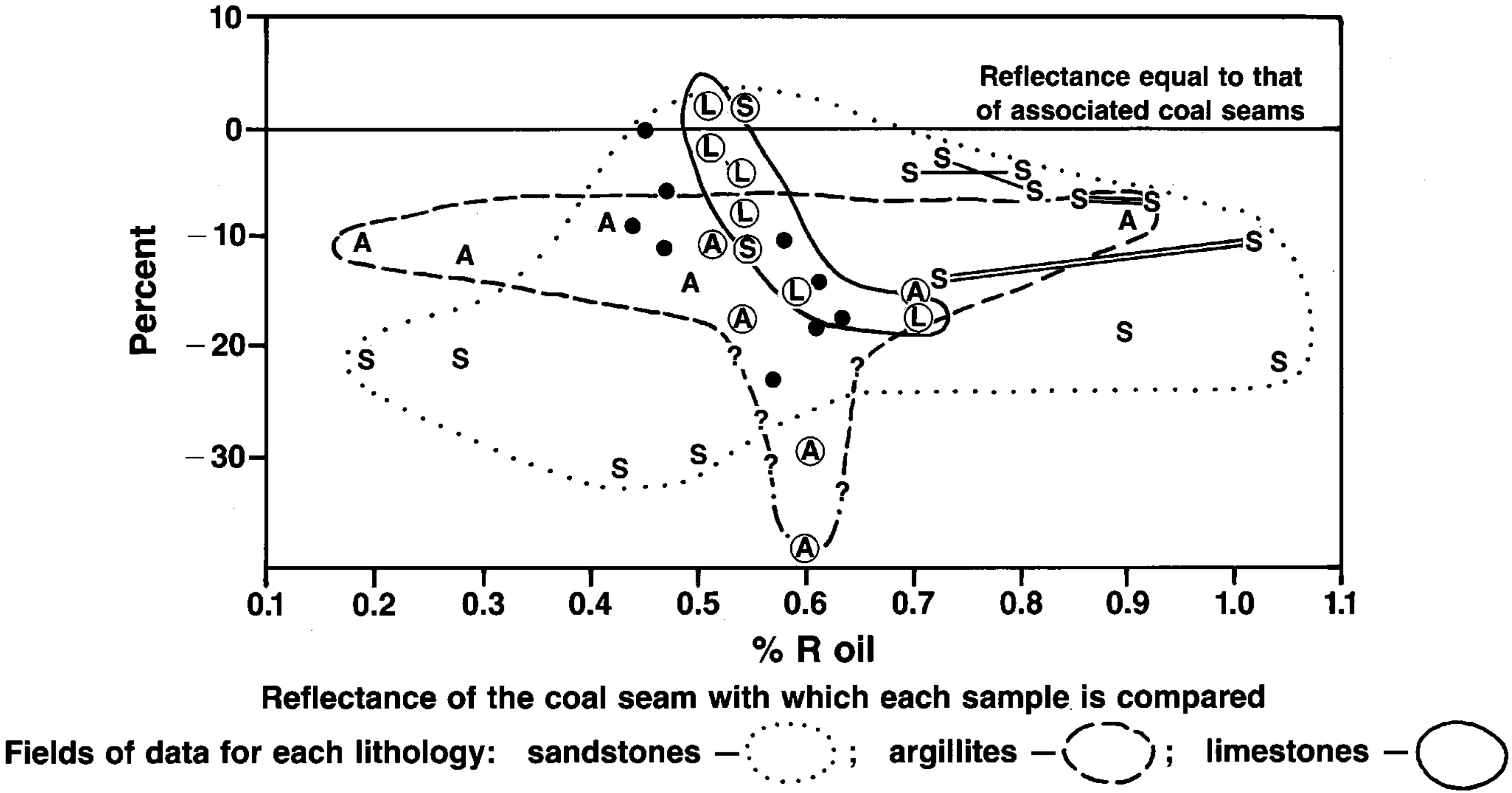
500X, field width 140 μ

Weathered Shale



SAMPLES

- **SAMPLE TYPE** (core, outcrop, well cuttings)
- **LITHOLOGY** (coal, shale, siltstone, sandstone)
- **SAMPLE HANDLING** (oil-based drilling mud, kerogen isolation, oxidation, heating)
- **ORGANIC MATTER** (quantity, quality, size, type, thermal maturity, reflectance suppression/enhancement)



Bostick and Foster, 1975

SAMPLES

- **SAMPLE TYPE** (core, outcrop, well cuttings)
- **LITHOLOGY** (coal, shale, siltstone, sandstone)
- **SAMPLE HANDLING** (oil-based drilling mud, kerogen isolation, oxidation, heating)
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SAMPLES

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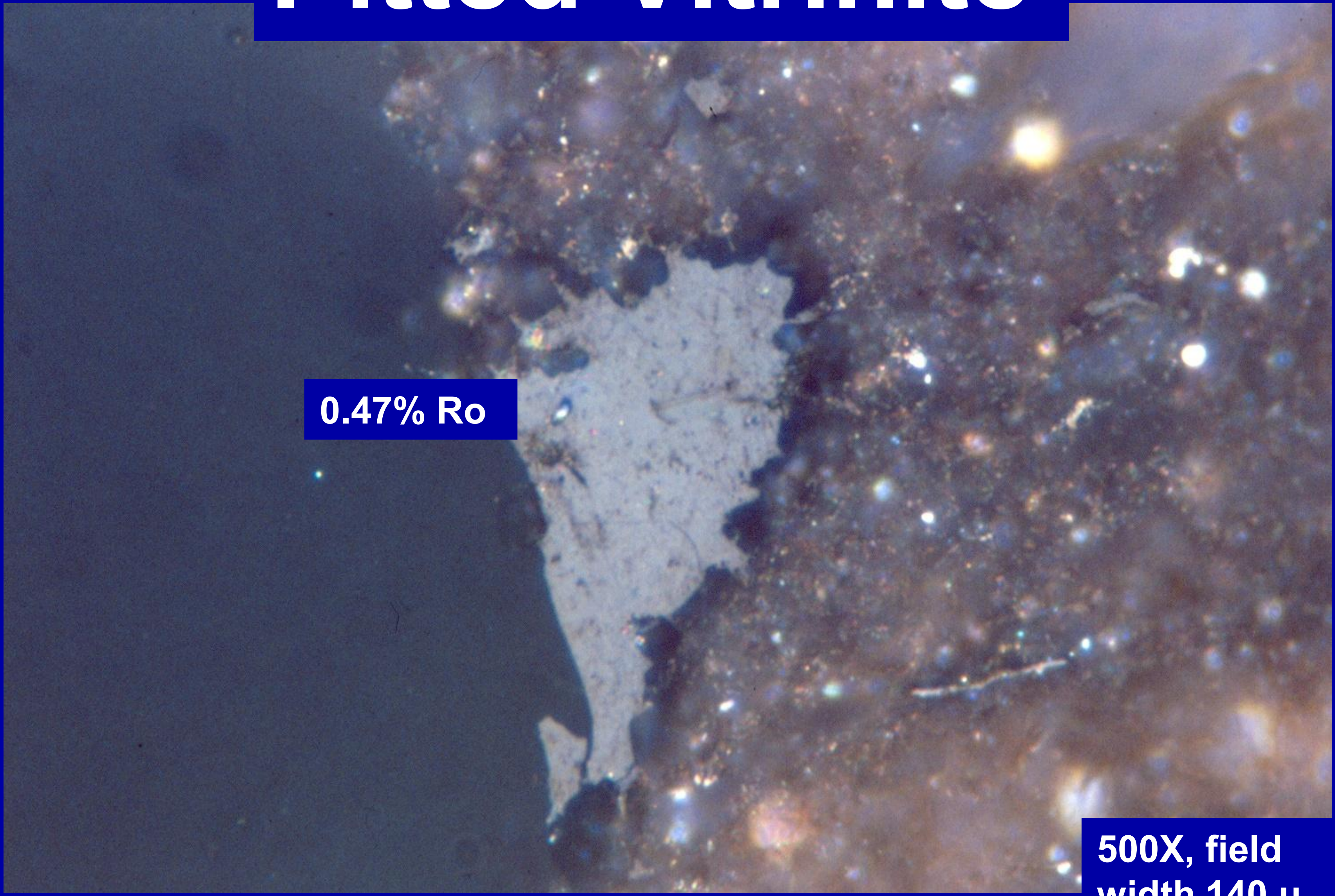
- **SAMPLE HANDLING** (oil-based drilling mud, kerogen isolation, oxidation, heating)
- **ORGANIC MATTER** (quantity, quality, size, type, thermal maturity, reflectance suppression/enhancement)

ORGANIC MATTER ERRORS

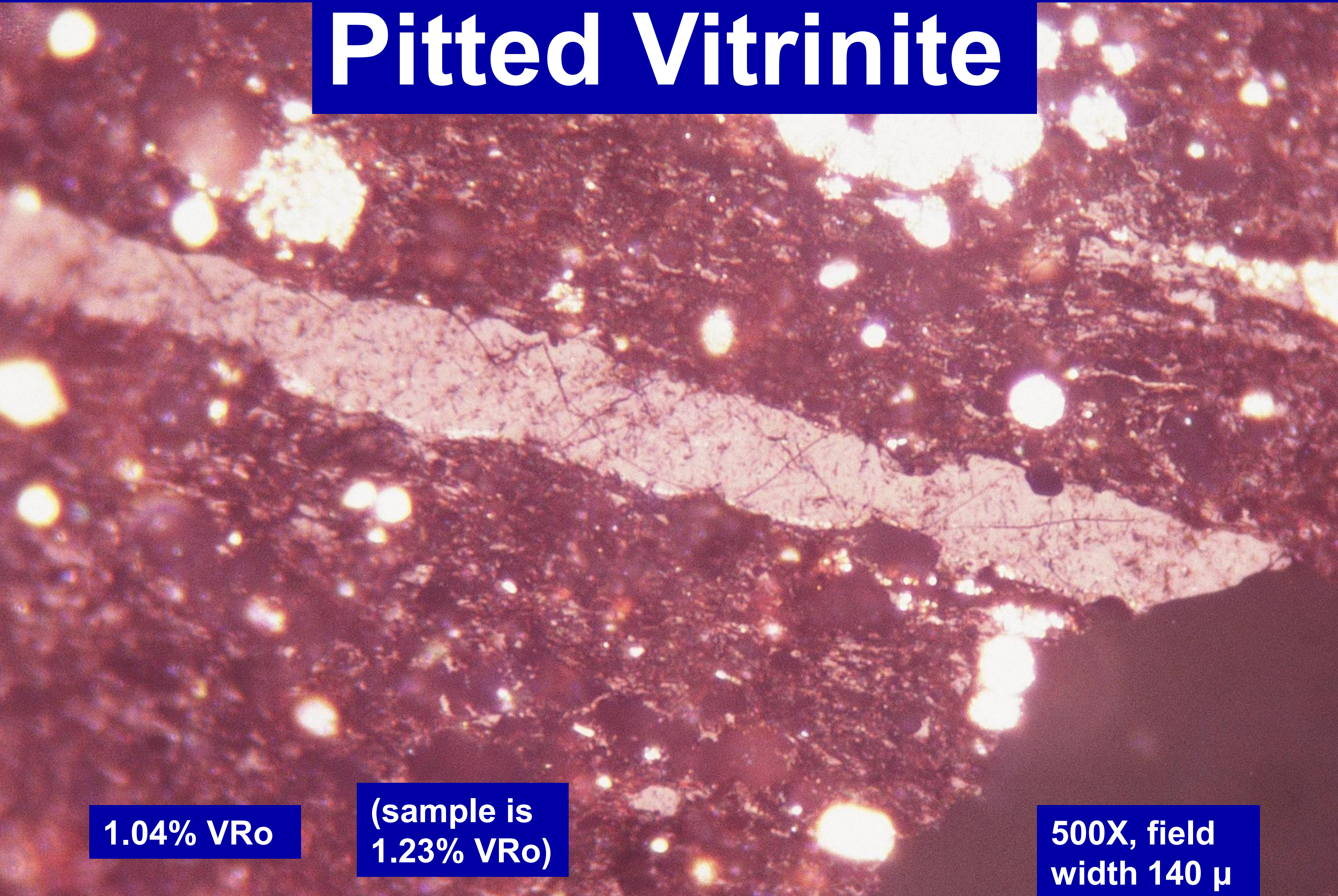
- Quantity (minimum of 20) (Barker and Pawlewicz, 1993)
- Quality (e.g., pitted vitrinite)(ICCP)
- Size (larger than measuring spot, >10 microns)
- Type (vitrinite-like organic matter)
- Thermal maturity (anisotropy, >1% VRo)
- Reflectance suppression/enhancement (e.g., alginite; oxidizing environment)

Pitted Vitrinite



500X, field width 140 μ

Pitted Vitrinite

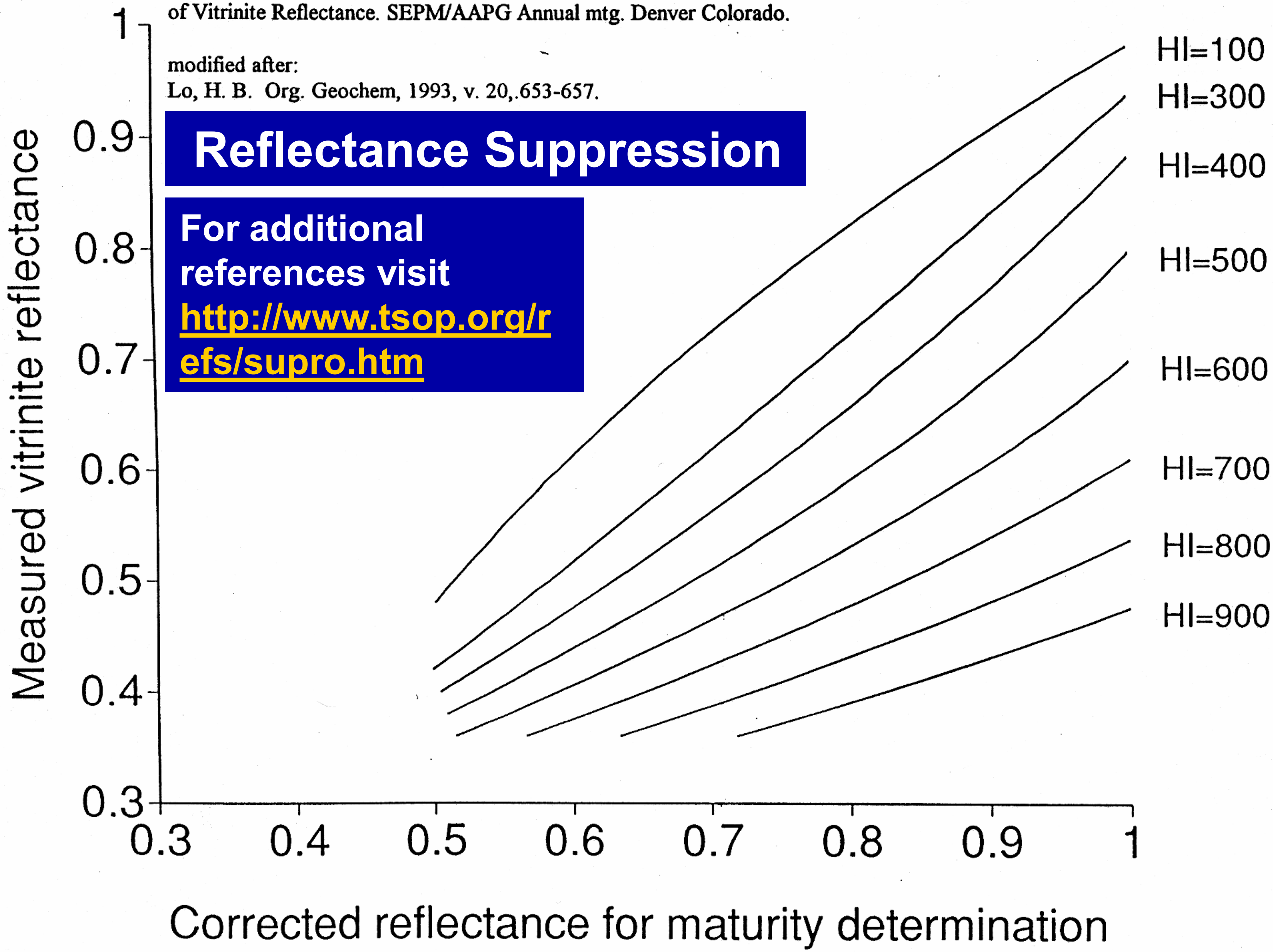


from:
Quick, J. C., Wavrek D. A., 1994, Suppressed reflectance vitrinite:Recognition and correction. , Poster Session on: Thermal Maturity in Sedimentary Bains: Uses and Abuses of Vitrinite Reflectance. SEPM/AAPG Annual mtg. Denver Colorado.

modified after:
Lo, H. B. Org. Geochem, 1993, v. 20,.653-657.

Reflectance Suppression

For additional references visit http://www.tsop.org/refs/supro.htm

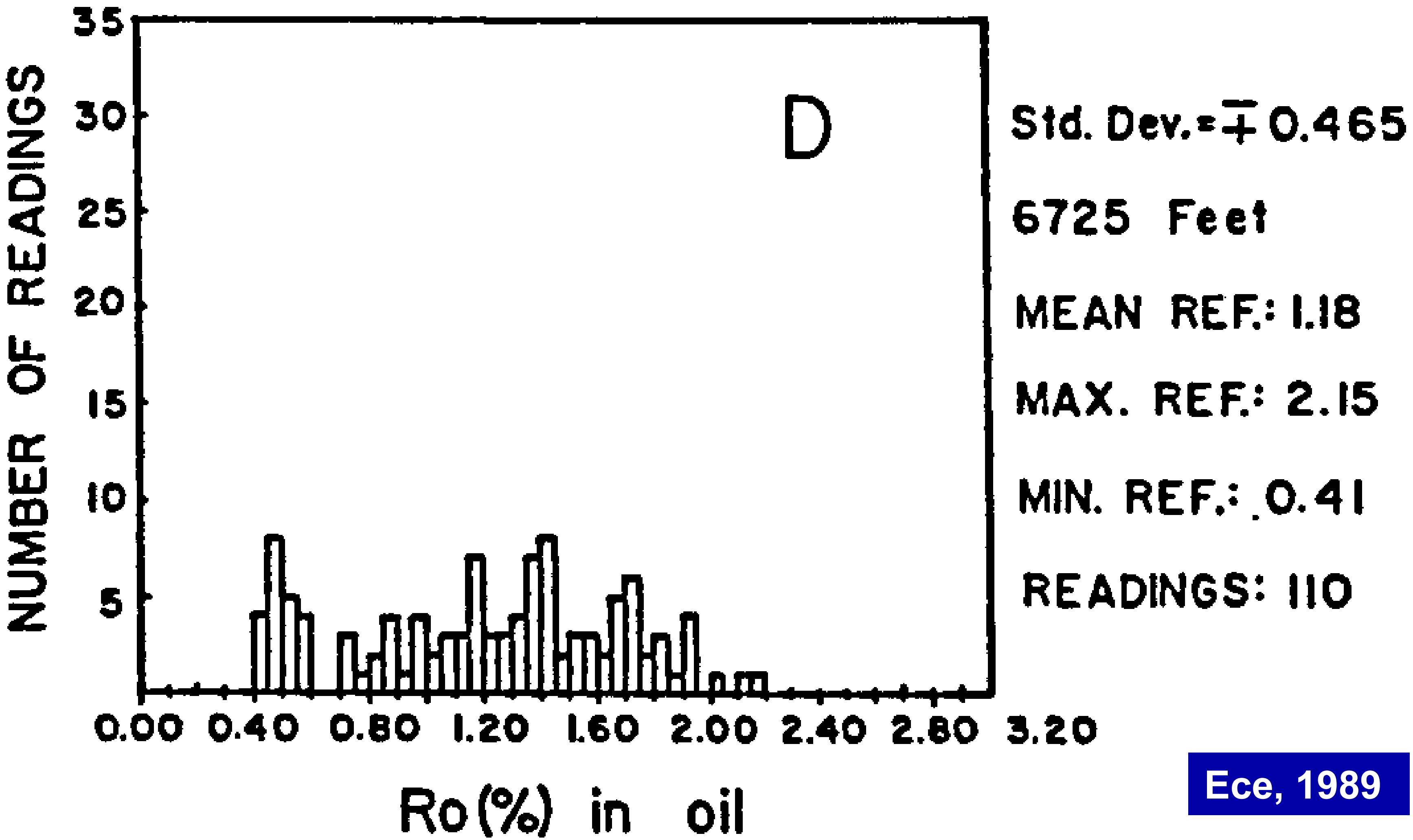


Summary of How to Tell Good Data from Bad Data

- **Number of Measurements** (minimum of 20)
- **Reflectance Histogram**

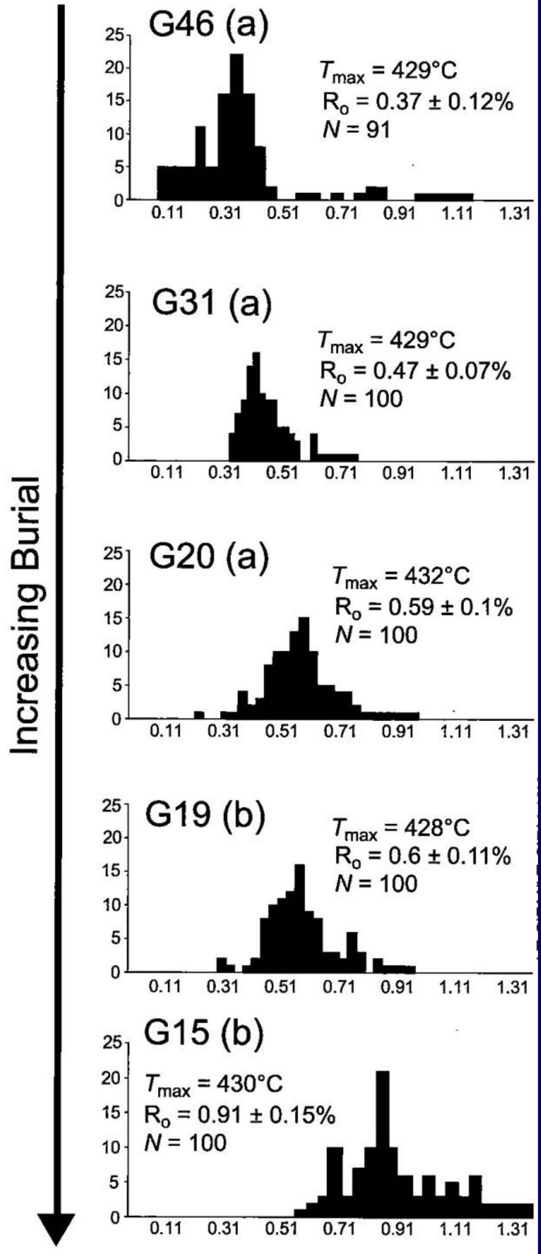
  (shape of distribution and spread of values)
- **Photomicrographs** (quality and size of clasts; surrounding minerals [kerogen concentrate vs. whole-rock]; correct identification of low-gray [primary] vitrinite vs high-gray [recycled] vitrinite or inertinite)

Example of Poor Interpretation from Core Sample



Ece, 1989

Another Example of Poor Interpretation (used to calibrate a new thermal maturity indicator)



(A) Histogram of the Reflectance of Vitrinite

Abdelmalak and others, 2012

EQUIPMENT

- **POLISHING EQUIPMENT** (quality of polish; relief-free, scratch-free surface)
- **GLASS STANDARDS/CALIBRATION**

  (Ro range; immersion-oil contamination; air bubbles)
- **MICROSCOPE/PHOTOMETER** (quality of photometer/optics; stability to 0.01% Ro; frequency of calibration)

Importance of petrographic qualitative thermal maturity indicators to check accuracy of vitrinite-reflectance value:

- **Fluorescence** of liptinite macerals (e.g., algae): fluorescence changes from green, greenish-yellow, yellow, orange with increasing thermal maturity before it is extinguished (0.9-1.0% VRo for Tasmanites)
- Vitrinite Reflectance Equivalent from **bitumen** reflectance values.

SUMMARY

Vitrinite is a coal maceral derived from wood.

Vitrinite reflectance is a measurement of the percentage of light reflected from the vitrinite maceral.

Vitrinite reflectance value is an average of many measurements.

Disadvantages

Vitrinite reflectance cannot tell you whether or not a rock generated oil or gas

Limitations

Post Silurian-age rocks

Dependent on sample quality, size, and contamination

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- Research Committee

TSOP - Beijing

TSOP's ... held in ... th ... CCP will meet for five days and TSOP will meet for two days, with a field trip between.

TSOP - Halifax 2011 Meeting

Our 28th annual meeting was packed with activities and was widely complimented as a complete success. A short course, reception with presentation of the local heritage of Scottish dancing, two full days of technical sessions (the second day having two parallel sessions), keynote addresses, posters, lunches, banquet, and overnight field trip filled five days. Many also toured Nova Scotia on their own before and after the meeting. See the thank-you note on the meeting site, and the image galleries.

TSOP on Facebook

A **Facebook** page for TSOP has been created by Secretary Jackie Holt. All are

TSOP is...

a Society for scientists and engineers involved with coal petrology, kerogen petrology, organic geochemistry and related disciplines. We have over 200 members in 30 countries. If you are part of the organic petrology community, we invite you to join TSOP.

Website includes Members-Only section

TSOP Members! Set up your account in our members-only section (with the user ID which you received in e-mail) to check your membership data, use the new Membership Directory, and more. Members don't need an account to **pay dues online**.

Help develop this new TSOP web site

Volunteers are wanted, for example to work with image galleries, administer sections on links, the calendar, and others. Please contact the webmaster.

Ask An Organic Petrologist

Have a question about organic petrology? See this new feature to ask a TSOP expert for the answer to your question.

TSOP receives 501(c)(3) status

We incorporated as a non-profit in the state of Virginia, USA, in 2008. Following application in June, 2009, the US Internal Revenue Service granted recognition of 501(c)(3) tax-exempt status on February 9, 2010. See the article on page 6 of the March Newsletter.

Other organizations of organic petrologists include ICCP and CSCOP.

Elsevier's International Journal of Coal Geology publishes the proceedings of TSOP Annual Meetings.

TSOP is a Member Society of AGI (publishers of Earth, formerly Geotimes; see evolution statement), and an AAPG Associated Society.

references last updated December 2011

Please submit suggested additions to webmaster@tsop.org .

TSOP: Selected References on Organic Petrology

Introduction

These bibliographic references have been compiled as a TSOP project, and organic petrologists have found the references to be useful in their work. The publications should be available at university or geological research center libraries. They are not available from TSOP, except for those listed on our Publications page, or as part of the TSOP Newsletter.

To view a category of references on this web site, choose from the list of individual topics.

Additional reference citations for inclusion in this project may be submitted to webmaster@tsop.org. Please state which of the categories would include them.

ENDNOTE Library:

Bob Cluff and others at The Discovery Group have formatted all 2200 TSOP references (from the 2007 update) on the topics listed at the left into an EndNote® X1 library. Download it here: TSOP_references_2007.enl

Updates to that file may be downloaded here (for EndNote® X2 and higher):
TSOP_references_2009_update.enl (2008 and 2009)
EndNote users can use its update process to add the contents of the 2009 update to the 2007 file. If comments or additional references have been placed in the 2007 file, this process will preserve them in the resulting file.

Journal styles for EndNote may be downloaded here:
International Journal of Coal Geology style
Please submit requests for other journal style files to webmaster@tsop.org

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