The Biomarker Guide

Second Edition
Volume 1

The second edition of *The Biomarker Guide* is a fully updated and expanded version of this essential reference. Now in two volumes, it provides a comprehensive account of the role that biomarker technology plays both in petroleum exploration and in understanding Earth history and processes.

Biomarkers and Isotopes in the Environment and Human History details the origins of biomarkers and introduces basic chemical principles relevant to their study. It discusses analytical techniques, and applications of biomarkers to environmental and archeological problems.

The Biomarker Guide is an invaluable resource for geologists, petroleum geochemists, biogeochemists, environmental scientists, and archeologists.

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I. Biomarkers and Isotopes in the Environment and Human History

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CAMBRIDGE
UNIVERSITY PRESS

PUBLISHED BY THE PRESS SYNDICATE OF THE UNIVERSITY OF CAMBRIDGE
The Pitt Building, Trumpington Street, Cambridge, United Kingdom

CAMBRIDGE UNIVERSITY PRESS
The Edinburgh Building, Cambridge CB2 2RU, UK
40 West 20th Street, New York, NY 10011-4211, USA
477 Williamstown Road, Port Melbourne, VIC 3207, Australia
Ruiz de Alarcón 13, 28014 Madrid, Spain
Dock House, The Waterfront, Cape Town 8001, South Africa

http://www.cambridge.org

First published 1993 by Prentice Hall, Inc.
Second edition published 2005 by Cambridge University Press

Printed in the United Kingdom at the University Press, Cambridge

Typeface Ehrhardt 9.5/12 pt *System* LaTeX 2ε [TB]

A catalog record for this book is available from the British Library

Library of Congress Cataloging in Publication data

Peters, Kenneth E.
The biomarker guide. – 2nd ed. / K. E. Peters, C. C. Walters, and J. M. Moldowan.
p. cm.
Includes bibliographical references and index.
Contents: 1. Biomarkers in the environment and human history – 2. Biomarkers in petroleum systems and Earth history.
ISBN 0 521 78158 2
1. Petroleum – Prospecting. 2. Biogeochemical prospecting. 3. Biochemical markers. I. Walters, C. C. (Clifford C.)
II. Moldowan, J. M. (J. Michael), 1946– III. Title.
TN271.P4P463 2004
622′.1828–dc22 2003065416

ISBN 0 521 78158 2 hardback vol. 1
ISBN 0 521 83762 6 hardback vol. 2
ISBN 0 521 83763 4 set of two vols.

Cambridge University Press
0521781582 - The Biomarker Guide: Biomarkers and Isotopes in the Environment and Human History, Second Edition
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Dedicated to

Vanessa, Brent, and Miwok

Johnet

Mary

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About the authors

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Clifford C. Walters received Bachelor degrees in chemistry and biology from Boston University in 1976. He attended the University of Maryland, where he worked on the chemistry of Martian soil and conducted field and laboratory research on metasediments from Isua, Greenland, the oldest sedimentary rocks on Earth. After receiving a Ph.D. in geochemistry in 1981, Cliff continued with postdoctoral research on the organic geochemistry of Precambrian sediments and meteorites. He joined Gulf Research and Development in 1982, where he implemented a program in biological marker compounds. In 1984, he moved to Sun Exploration & Production Company, where he was responsible for technical service and establishing biomarker geochemistry and thermal modeling as routine exploration tools. Mobil's Dallas Research Lab hired Cliff in 1988, where he became Supervisor of the Geochemical Laboratories in 1991. He is now Senior Research Associate with ExxonMobil Research and Engineering Company, where he conducts work on the modeling of oil generation and reservoir transformations, geomicrobiology, and processes of solids formation. Cliff published numerous papers, served as Editor of the ACS Geochemistry Division from 1990 to 1992, and is a current Associate Editor of *Organic Geochemistry*.

J. Michael Moldowan attained a B.S. in chemistry from Wayne State University, Detroit, Michigan, and a Ph.D. in chemistry from the University of Michigan in 1972. Following a postdoctoral fellowship in marine natural products with Professor Carl Djerassi at Stanford University, he joined Chevron's Biomarker Group in 1974. The Chevron biomarker team, led by the late Dr. Wolfgang K. Seifert in the mid 1970s to early 1980s, is largely credited with pioneering the application of biological marker technology to petroleum exploration. Mike joined the Department of Geological and Environmental Sciences of Stanford University as Professor (Research) in 1993. In 1986, he served as Chair of the Division of Geochemistry of the American Chemical Society, and he has twice been awarded the Organic Geochemistry Division of the Geochemical Society Best Paper Award for publications he co-authored in 1978 and 1989.

Preface

Biological markers (biomarkers) are complex molecular fossils derived from biochemicals, particularly lipids, in once-living organisms. Because biological markers can be measured in both crude oils and extracts of petroleum source rocks, they provide a method to relate the two (correlation) and can be used by geologists to interpret the characteristics of petroleum source rocks when only oil samples are available. Biomarkers are also useful because they can provide information on the organic matter in the source rock (source), environmental conditions during its deposition and burial (diagenesis), the thermal maturity experienced by rock or oil (catagenesis), the degree of biodegradation, some aspects of source rock mineralogy (lithology), and age. Because of their general resistance to weathering, biodegradation, evaporation, and other processes, biomarkers are commonly retained as indicators of petroleum contamination in the environment. They also occur with certain human artifacts, such as bitumen sealant for ancient boats, hafting material on spears and arrows, burial preservatives, and as coatings for medieval paintings.

Biomarker and non-biomarker geochemical parameters are best used together to provide the most reliable geologic interpretations to help solve exploration, development, production, and environmental or archeological problems. Prior to biomarker work, oil and rock samples are typically screened using non-biomarker analyses. The strength of biomarker parameters is that they provide more detailed information needed to answer questions about the source-rock depositional environment, thermal maturity, and the biodegradation of oils than non-biomarker analyses alone.

Distributions of biomarkers can be used to correlate oils and extracts. For example, C_{27}–C_{28}–C_{29} steranes or monoaromatic steroids distinguish oil-source families with high precision. Cutting-edge analytical techniques, such as linked-scan gas chromatography/mass spectrometry/mass spectrometry (GCMS/MS) provide sensitive measurements for correlation of light oils and condensates, where biomarkers are typically in low concentrations. Because biomarkers typically contain more than ~20 carbon atoms, they are useful for interpreting the origin of the liquid fraction of crude oil, but they do not necessarily indicate the origin of associated gases or condensates.

Different depositional environments are characterized by different assemblages of organisms and biomarkers. Commonly recognized classes of organisms include bacteria, algae, and higher plants. For example, some rocks and related oils contain botryococcane, a biomarker produced by the lacustrine, colonial alga *Botryococcus braunii*. *Botryococcus* is an organism that thrives only in lacustrine environments. Marine, terrigenous, deltaic, and hypersaline environments also show characteristic differences in biomarker composition.

The distribution, quantity, and quality of organic matter (organic facies) are factors that help to determine the hydrocarbon potential of a petroleum source rock. Optimal preservation of organic matter during and after sedimentation occurs in oxygen-depleted (anoxic) depositional environments, which commonly lead to organic-rich, oil-prone petroleum source rocks. Various biomarker parameters, such as the C_{35} homohopane index, can indicate the degree of oxicity under which marine sediments were deposited.

Biomarker parameters are an effective means to rank the relative maturity of petroleum throughout the entire oil-generative window. The rank of petroleum can be correlated with regions within the oil window (e.g. early, peak, or late generation). This information can provide a clue to the quantity and quality of the oil that may have been generated and, coupled with quantitative petroleum conversion measurements (e.g. thermal modeling programs), can help evaluate the timing of petroleum expulsion.

Biomarkers can be used to determine source and maturity, even for biodegraded oils. Ranking systems are based on the relative loss of *n*-alkanes, acyclic isoprenoids, steranes, terpanes, and aromatic steroids during biodegradation.

Biomarkers in oils provide information on the lithology of the source rock. For example, the absence of rearranged steranes can be used to indicate petroleum derived from clay-poor (usually carbonate) source rocks. Abundant gammacerane in some petroleum appears to be linked to a stratified water column (e.g. salinity stratification) during deposition of the source rock.

Biomarkers provide information on the age of the source rock for petroleum. Oleanane is a biomarker characteristic of angiosperms (flowering plants) found only in Tertiary and Upper Cretaceous rocks and oils. C_{26} norcholestanes originate from diatoms and can be used to distinguish Tertiary from Cretaceous and Cretaceous from older oils. Dinosterane is a marker for marine dinoflagellates, possibly distinguishing Mesozoic and Tertiary from Paleozoic source input. Unusual distributions of *n*-alkanes and cyclohexylalkanes are characteristic of *Gloeocapsomorpha prisca* found in early Paleozoic samples. 24-*n*-Propylcholestane is a marker for marine algae extending from at least the Devonian to the present.

Continued growth in the geologic, environmental, and archeological applications of biomarker technology is anticipated, particularly in the areas of age-specific biomarkers, the use of biomarkers to indicate source organic matter input and sedimentologic conditions, correlation of oils and rocks, and understanding the global cycle of carbon. New developments in analytical methods and instrumentation and the use of biomarkers to understand petroleum migration and kinetics are likely. Finally, early work suggests that biomarkers will continue to grow as tools to understand production, environmental, and archeological problems.

HOW TO USE THIS GUIDE

The Biomarker Guide is divided into two volumes. The first volume introduces some basic chemical principles and analytical techniques, concentrating on the study of biomarkers and isotopes in the environment and human history. The second volume expands on the uses of biomarkers and isotopes in the petroluem industry, and investigates their occurrence throughout Earth history.

The Biomarker Guide was written for a diverse audience, which might include the following:

- students of geology, environmental science, and archeology who wish to gain general knowledge of what biomarkers can do;
- practicing geologists and geochemical coordinators in the petroleum industry with both specific and general questions about which biomarker and/or non-biomarker parameters might best answer regional exploration, development, or production problems;
- experienced geochemists who require detailed information on specific parameters or methodology;
- managers or research directors who require a concise explanation for terms and methodology;
- refinery process chemists requiring a more detailed knowledge of petroleum; and
- archeologists and environmental scientists interested in a technology useful for characterizing petroleum in the environment.

The text in each chapter is supplemented by many references to related sections in the book and to the literature. Various parts of the guide, such as notes, highlight detailed discussions that supplement the text.

The following is a brief overview of each chapter in the two volumes.

PART I BIOMARKERS AND ISOTOPES IN THE ENVIRONMENT AND HUMAN HISTORY

1 Origin and preservation of organic matter

This chapter introduces biomarkers, the domains of life, primary productivity, and the carbon cycle on Earth. Morphological and biochemical differences among different life forms help to determine their environmental habitats and the character of the biomarkers that they contribute to sediments, source rocks, and petroleum. The discussion summarizes processes affecting the distribution, preservation, and alteration of biomarkers in sedimentary rocks. Various factors, such as type of organic matter input, redox potential during sedimentation, bioturbation, sediment grain size, and sedimentation rate, influence the quantity and quality of organic matter preserved in rocks during Earth's history.

2 Organic chemistry

A brief overview of organic chemistry includes explanations of structural nomenclature and stereochemistry necessary to understand biomarker parameters. The discussion includes an overview of compound classes in petroleum and concludes with examples of the structures and nomenclature for several biomarkers, their precursors in living organisms, and their geologic alteration products.

3 Biochemistry of biomarkers

This chapter provides an overview of the biochemical origins of the major biomarkers, including discussions of the function, biosynthesis, and occurrence of their precursors in living organisms. Some topics include lipid membranes and their chemical compositions, the biosynthesis of isoprenoids and cyclization of squalene, and examples of hopanoids, sterols, and porphyrins in the biosphere and geosphere.

4 Geochemical screening

This chapter describes how to select sediment, rock, and crude oil samples for advanced geochemical analyses by using rapid, inexpensive geochemical tools, such as Rock–Eval pyrolysis, total organic carbon, vitrinite reflectance, scanning fluorescence, gas chromatography, and stable isotope analyses. The discussion covers sample quality, selection, storage, and geochemical rock and oil standards. Other topics include how to test rock samples for indigenous bitumen, surface geochemical exploration using piston cores, geochemical logs and their interpretation, chromatographic fingerprinting for reservoir continuity, and how to deconvolute mixtures of oils from different production zones. Mass balance equations show how to calculate the extent of fractional conversion of kerogen to petroleum, source-rock expulsion efficiency, and the original richness of highly mature source rocks.

5 Refinery oil assays

Many refinery oil assays differ substantially from geochemical analyses conducted by petroleum or environmental geochemists, although interdisciplinary use of these tools is becoming more common. Some basic oil assays include API (American Petroleum Institute) gravity, pour point, cloud point, viscosity, trace metals, total acid number, refractive index, and wax content. More advanced oil assays include chemical group-type fractionation and field ionization mass spectrometry. A brief overview of refinery processes includes the fate of biomarkers in straight-run and processed refinery products with tips on how to distinguish refined from natural petroleum products in environmental or geological samples.

6 Stable isotope ratios

This chapter describes stable isotopes and their use to characterize petroleum, including gases, crude oils, sediment and source-rock extracts, and kerogen, with emphasis on stable carbon isotope ratios. The discussion includes isotopic standards and notation, principles of isotopic fractionation, and the use of various isotopic tools, such as stable carbon isotope-type curves, for correlation or quantification of petroleum mixtures. The chapter concludes with new developments in compound-specific isotope analysis, including its application to better understand the origin of carboxylic acids and the process of thermochemical sulfate reduction in petroleum reservoirs.

7 Ancillary geochemical methods

Ancillary geochemical tools (e.g. diamondoids, C_7 hydrocarbons, compound-specific isotopes, and fluid inclusions) can be used to evaluate the origin, thermal maturity, and extent of biodegradation or mixing of petroleum, even when the geological samples lack or have few biomarkers. Molecular modeling can be used to rationalize or predict the geochemical behavior of biomarkers and other compounds in the geosphere.

8 Biomarker separation and analysis

This chapter describes the organization of a biomarker laboratory and the methods used to prepare and separate crude oils and sediment or source-rock extracts into fractions prior to mass spectrometric analysis. The concept of mass spectrometry is explained. Many of these fundamentals, such as the difference between a mass chromatogram and a mass spectrum, or between selected ion and linked-scan modes of analysis, are critical to understanding later discussions of biomarker parameters. Several key topics, including analytical procedures, internal standards, and examples of gas chromatography/mass spectrometry (GCMS) data problems, help the reader to evaluate the quality of biomarker data and interpretations.

9 Origin of petroleum

This chapter describes evidence against the deep-earth gas hypothesis, which invokes an abiogenic origin for petroleum by polymerization of methane deep in the Earth's mantle. The deep-earth gas hypothesis has little scientific support but, if correct, could have major implications for petroleum exploration and the application of biomarkers to environmental science and archeology. The discussion covers experimental, geological, and geochemical evidence supporting the thermogenic origin of petroleum.

10 Biomarkers in the environment

This chapter explains how analyses of biomarkers and other environmental markers, such as polycyclic aromatic hydrocarbons, are used to characterize, identify, and assess the environmental impact of oil spills. The discussion covers processes affecting the composition of spilled oil, such as emulsification, oxidation, and biodegradation, as well as oil-spill mitigation and modeling. Field and laboratory procedures for sampling and analyzing spills are discussed, including program design, chemical fingerprinting, and data quality control. The chapter includes sections on smoke, natural gas, and gasoline and other light fuels as pollutants, and a detailed discussion of the controversial *Exxon Valdez* oil spill.

11 Biomarkers in archeology

This chapter provides examples of the growing use of biomarker and isotopic analyses to evaluate organic materials in archeology. Some of the topics include bitumens in Egyptian mummies, such as Cleopatra, archeological gums and resins, and biomarkers in art and ancient shipwrecks. The discussion covers the use of biomarkers and isotopes in studies of paleodiet and agricultural practices, including studies of ancient wine and beeswax. Other topics include archeological DNA, proteins, and evidence for ancient narcotics.

PART II BIOMARKERS AND ISOTOPES IN PETROLEUM AND EARTH HISTORY

12 Geochemical correlations and chemometrics

Geochemical correlation can be used to establish petroleum systems to improve exploration success, define reservoir compartments to enhance production, or identify the origin of petroleum contaminating the environment. This chapter explains how chemometrics simplifies genetic oil–oil and oil–source rock correlations and other interpretations of complex multivariate data sets.

13 Source- and age-related biomarker parameters

This chapter explains how biomarker analyses are used for oil–oil and oil–source rock correlation and how they help to identify characteristics of the source rock (e.g. lithology, geologic age, type or organic matter, redox conditions), even when samples of rock are not available. Biomarker parameters are arranged by groups of related compounds in the order: (1) alkanes and acyclic isoprenoids, (2) steranes and diasteranes, (3) terpanes and similar compounds, (4) aromatic steroids, hopanoids, and similar compounds, and (5) porphyrins. Critical information on specificity and the means for measurement are highlighted above the discussion for each parameter.

14 Maturity-related biomarker parameters

This chapter explains how biomarker analyses are used to assess thermal maturity. The parameters are arranged by groups of related compounds in the order (1) terpanes, (2) polycadinenes and related products, (3) steranes, (4) aromatic steroids, (5) aromatic hopanoids, and (6) porphyrins. Critical information on specificity and the means for measurement are highlighted in bold print above the discussion for each parameter.

15 Non-biomarker maturity parameters

This chapter explains how certain non-biomarker parameters, such as ratios involving *n*-alkanes and aromatic hydrocarbons, are used to assess thermal maturity. Critical information on specificity and the means for measurement are highlighted above the discussion for each parameter.

16 Biodegradation parameters

This chapter explains how biomarker and non-biomarker analyses are used to monitor the extent of biodegradation. Compound classes and parameters are discussed in the approximate order of increasing resistance to biodegradation. The discussion covers recent advances in our understanding of the controls and mechanisms of petroleum biodegradation and the relative significance of aerobic versus anaerobic degradation in both surface and subsurface environments. Examples show how to predict the original physical properties of crude oils prior to biodegradation.

17 Tectonic and biotic history of the Earth

The evolution of life is closely tied to biomarkers in petroleum. This chapter provides a brief tectonic history of the Earth in relation to the evolution of major life forms. Mass extinctions and their possible causes are discussed. The end of the section for each time period includes examples of source rocks and related crude oils with emphasis on the geochemistry of the oils. These examples are linked to more detailed discussion of petroleum systems in Chapter 18.

18 Petroleum systems through time

This chapter defines petroleum systems and provides examples of the geology, stratigraphy, and geochemistry of source rocks and crude oils through geologic time. Gas chromatograms, sterane and terpane mass chromatograms, stable isotope compositions, and other geochemical data are provided for representative crude oils generated from many worldwide source rocks.

19 Problem areas and further work

This chapter describes areas requiring further research, including the application of biomarkers to migration, the kinetics of petroleum generation, geochemical correlation and age assessment, and the search for extraterrestrial life.

Purpose

The Biomarker Guide provides a comprehensive discussion of the basic principles of biomarkers, their relationships with other parameters, and their applications to studies of maturation, correlation, source input, depositional environment, and biodegradation of the organic matter in petroleum source rocks, reservoirs, and the environment. It builds upon previous books by Tissot and Welte (1984), Waples and Machihara (1991), Bordenave (1993), Peters and Moldowan (1993), Hunt (1996), and Welte *et al*. (1997). The volumes were prepared for a broad audience, including students, company exploration geologists, geochemists, and environmental scientists for several reasons:

(1) Biomarker geochemistry is a rapidly growing discipline with important worldwide applications to petroleum exploration and production and environmental monitoring.
(2) Biomarker parameters are becoming increasingly prominent in exploration, production, and environmental reports.
(3) Different parameters are used within the industry, academia, service laboratories, and the literature.
(4) The quality of biomarker data and interpretation can vary considerably, depending on their source.

The objective of this guide is to provide a single, concise source of information on the various biomarker parameters and to create general guidelines for the use of selected parameters. An important aim is to clarify the relationships between biomarker and other geochemical parameters and to show how they can be used together to solve problems. It is not intended to teach interpretation of raw biomarker data. This is a job for a biomarker specialist with years of training in instrumentation and organic chemistry. A crash-course or cookbook approach cannot provide such training without the consequence of serious interpretive errors and a tarnished view of the applicability of biomarkers in general.

A final objective of the guide is to impart in each reader a feeling for the excitement and vigor of the new field of biomarker geochemistry. Expanding research efforts at geochemical laboratories worldwide have increased the rate of change and growth in our geochemical concepts. Applications of many of the biomarker parameters presented here will undoubtedly improve with time and further research. We anticipate that more than a few readers will be directly involved in making these improvements possible.

Acknowledgments

The authors gratefully acknowledge the support of Chevron management and technical personnel (now ChevronTexaco) during preparation of the precursor to this book, which was called *The Biomarker Guide*. In particular, the authors thank G. J. Demaison, C. Y. Lee, F. Fago, R. M. K. Carlson, P. Sundararaman, J. E. Dahl, M. Schoell, E. J. Gallegos, P. C. Henshaw, S. R. Jacobson, R. J. Hwang, D. K. Baskin, and M. A. McCaffrey for discussions, technical assistance, and helpful review comments.

We acknowledge the support of Mobil and ExxonMobil management and technical personnel during preparation of much of *The Biomarker Guide* Second Edition. In particular, the authors thank Ted Bence, Paul Mankiewicz, John Guthrie, Jim Gormly, and Roger Prince for their input. We also thank Bill Clendenen, Larry Baker, Gary Isaksen, Jim Stinnett (Mobil, retired), Al Young (Exxon, retired), and Steve Koch.

We acknowledge the support of management at the US Geological Survey during preparation of the book. Special thanks are due to Les Magoon, Bob Eganhouse, Mike Lewan, Keith Kvenvolden, Fran Hostettler, Tom Lorenson, and Ron Hill.

Special thanks are due to Steve Brown and John Zumberge of GeoMark Research, Inc. for allowing us to use various oil Information sheets from their Oil Information Library System (OILS) and several cross-plots of biomarker ratios. We also thank David Zinniker of Stanford University for input on terrigenous biomarkers.

Finally, we thank the many reviewers of various drafts of the Second Edition, who are listed in the following table. Their dedication to the sometimes thankless job of peer review is to be commended.

Chapter	Title	Reviewer	Affiliation
1	Origin and preservation of organic matter	Kirsten Laarkamp	ExxonMobil Upstream Research
		Phil Meyers	University of Michigan
2	Organic chemistry	Kirsten Laarkamp	ExxonMobil Upstream Research
3	Biochemistry of biomarkers	Robert Carlson	ChevronTexaco
4	Geochemical screening	Dave Baskin	OilTracers, L. L. C.
		George Claypool	Mobil (retired)
		Jim Gormly	ExxonMobil Upstream Research
		Tom Lorenson	US Geological Survey
5	Refinery oil assays	Owen BeMent	Shell Oil Company
		Paul Mankiewicz	ExxonMobil Upstream Research
		Robert McNeil	Shell Oil Company
6	Stable isotope ratios	Mike Engel	University of Oklahoma
		Martin Schoell	ChevronTexaco (retired)
		Zhengzheng Chen	Stanford University
		John Guthrie	ExxonMobil Upstream Research
		Jeffrey Sewald	Woods Hole Oceanographic Institution

(*cont.*)

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Chapter	Title	Reviewer	Affiliation
7	Ancillary geochemical methods	Ron Hill	US Geological Survey
		Dan Jarvie	Humble Geochemical Services, Inc.
		Yitian Xiao	ExxonMobil Upstream Research
8	Biomarker separation and analysis	John Guthrie	ExxonMobil Upstream Research
		Robert Carlson	ChevronTexaco
9	Origin of petroleum	Kevin Bohacs	ExxonMobil Upstream Research
		Barbara Sherwood Lollar	University of Toronto
10	Biomarkers in the environment	Ted Bence, Rochelle Jozwiak, Mike Smith, Bill Burns (retired)	ExxonMobil Upstream Research
		Roger Prince	ExxonMobil Strategic Research
		Bob Eganhouse, Keith Kvenvolden, Fran Hostettler	US Geological Survey
		Ian Kaplan	UCLA (retired)
11	Biomarkers in archeology	Roger Prince	ExxonMobil Strategic Research
		Max Vityk	ExxonMobil Upstream Research
12	Geochemical correlations and chemometrics	Jaap Sinninghe Damsté	Netherlands Institute for Sea Research
		Paul Mankiewicz	ExxonMobil Upstream Research
		Scott Ramos, Brian Rohrback	Infometrix, Inc.
13	Source- and age-related biomarker parameters	Jaap Sinninghe Damsté	Netherlands Institute for Sea Research
		Leroy Ellis	Terra Nova Technologies
		Kliti Grice	University of Western Australia
		Paul Mankiewicz	ExxonMobil Upstream Research
		Roger Summons	Massachusetts Institute of Technology
		David Zinniker	Stanford University
14	Maturity-related biomarker parameters	Gary Isaksen	ExxonMobil Upstream Research
		Ron Noble	BHP Billiton
15	Non-biomarker maturity parameters	Gary Isaksen	ExxonMobil Upstream Research
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16	Biodegradation parameters	Dave Converse	ExxonMobil Upstream Research
		Roger Prince	ExxonMobil Strategic Research
17	Tectonic and biotic history of the Earth	Kevin Bohacs	ExxonMobil Upstream Research
		Keith Kvenvolden	US Geological Survey
18	Petroleum systems through time	Steve Creaney	ExxonMobil Exploration Company
		Les Magoon	US Geological Survey
19	Problem areas and further work	John Guthrie	ExxonMobil Upstream Research
		Mike Lewan	US Geological Survey
–	References	Jan Heagy, Marsha Harris	ExxonMobil Upstream Research
		Susie Bravos, Page Mosier, Emily Shen-Torbik	US Geological Survey