STUDIES ON THE TEXTS OF THE DESERT OF JUDAH [87]

Holistic Qumran

Trans-Disciplinary Research of Qumran and the Dead Sea Scrolls

PROCEEDINGS OF THE NIAS-LORENTZ CENTER QUMRAN WORKSHOP, 21-25 APRIL 2008

EDITED BY
JAN GUNNEWEG,
ANNEMIE ADRIAENS,
JORIS DIK

BRILL

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Studies on the Texts of the Desert of Judah

Edited by

Florentino García Martínez

Associate Editors

Peter W. Flint
Eibert J.C. Tigchelaar

VOLUME 87

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LEIDEN • BOSTON
2010

This book is printed on acid-free paper.

Library of Congress Cataloging-in-Publication Data

Holistic Qumran : trans-disciplinary research of Qumran and the Dead Sea Scrolls /
edited by Jan Gunneweg, Annemie Adriaens, Joris Dik.
p. cm. — (Studies on the texts of the Desert of Judah ; v. 87)
Includes index.
ISBN 978-90-04-18152-6 (hardback : alk. paper)
1. Qumran community. 2. Dead Sea scrolls. I. Gunneweg, Jan. II. Adriaens, A. (Annemie)
III. Dik, Joris. IV. Title. V. Series.

BM175.Q6H65 2010
296.8'15—dc22

2009051494

ISSN 0169-9962
ISBN 978 90 04 18152 6

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PRINTED IN THE NETHERLANDS

CONTENTS

Preface ix

Acknowledgements xi

List of Contributors xiii

Map 1. Schematic map of Middle East, Israel & Qumran xvi

Map 2. Schematic map of Qumran Site, Caves and Cemetery with indication of recent excavations xvii

Map 3. Excavation Plan of the Qumran settlement. xviii

Introduction
JAN GUNNEWEG 1

1. The coin beneath the crust: A pilot study of coins from the Mediterranean coast of Israel
ANNEMIE ADRIAENS, MARK DOWSETT, EBERHARD LEHMANN, YOAV FARHI, JAN GUNNEWEG & L. BOUCHENOIRE 11

2. A short note on the application of synchrotron-based micro-tomography on the Dead Sea scrolls
JORIS DIK, LUKAS HELFEN, PETER REISCHIG, JORIK BLAAS AND JAN GUNNEWEG 21

3. Gender and Qumran
KATHARINA GALOR. 29

4. Was the Qumran settlement a mere pottery production center? What Instrumental Neutron Activation revealed
JAN GUNNEWEG AND MARTA BALLA. 39

5. Introduction to the "Buried Bones"
JAN GUNNEWEG 63

6. Animal remains from Khirbet Qumran: A case study of two bones (QUM 392 and 393) from two bone burials
GILA KAHILA BAR-GAL, TZVIKI ROSENBERG AND CHARLES GREENBLATT 67

7. Degradation of parchment and ink of the Dead Sea scrolls investigated using Synchrotron-based X-ray and Infrared Microscopy
BRIDGET MURPHY, MARINE COTTE, MARTIN MUELLER, MARTA BALLA AND JAN GUNNEWEG 77

8. Radiocarbon dating and Qumran
JOHANNES VAN DER PLICHT AND KAARE L. RASMUSSEN 99

9. Characterization of the writing media of the Dead Sea Scrolls
IRA RABIN, OLIVER HAHN, T. WOLFF, E. KINDZORRA, A. MASIC, U. SCHADE AND G. WEINBERG 123

10. On the age of Jar-35
KAARE L. RASMUSSEN, JAN GUNNEWEG, JOHANNES VAN DER PLICHT AND MARTA BALLA 135

11. Analyzes of a sample of "masse de fer" from Qumran locus 104 excavated by R. de Vaux
KAARE L. RASMUSSEN, ANDREW BOND, GREGORY DOUDNA AND JAN GUNNEWEG 145

12. The sciences and the reconstruction of the ancient scrolls: possibilities and impossibilities (summary)
EMANUEL TOV 155

13. Introduction to "Soap at Qumran"
JAN GUNNEWEG 163

14. Making soap as the Qumranites did
SASJA VAN DER VAART, ALEXANDRA VAN DEN BROEK, LAURA KLERKX AND NANDA DE VREE 171

15. The Dead Sea, the nearest neighbor of Qumran and the Dead Sea manuscripts. What SEM, XRD and Instrumental Neutron Activation may show about Dead Sea mud
JAN GUNNEWEG 175

Epilogue 183

Publications 185

Index 189

Color plates follow page 192

PREFACE

A handshake is usually a *hapax* gesture when people either meet or leave or instead of a signature under a contract, as in the diamant trade. A handshake between Humanities and Science can also be just that: A *hapax* collaboration to solve a specific problem in art, archaeology, restoration, conservation, chemistry and geology. After the problem is resolved, each member of the aforementioned domains goes his own way and he seldom meets a second time his collaborators. The latter can also be compared to the use of the word 'Taskforce' that usually is meant to solve too only one specific problem or question, such as for example the identification of a real Rembrandt vis-à-vis a fake.

When one wants to describe a long term or permanent collaboration within the various domains of Humanities, Social Sciences and the so-called Hard Science, then one uses the word inter-disciplinary, a study involving various techniques in different domains. In the present book, I will use the word trans-disciplinary because the topics treated and the techniques applied to solve pending questions vis-à-vis Qumran and the Dead Sea scrolls involve all possible cutting edge techniques available today. These techniques may start with the rather subjective way of making use of paleography to obtain a date for a manuscript or telling apart different writers basing oneself on the way certain letters of the alphabet were written by the scribes on the one hand, to analytical techniques as, for example, sophisticated neutron-tomography on the other whereby the human eye is less or not involved at all. In the latter, objective data are assembled and evaluated by highly developed software and statistics instead.

There are two ways of getting information on a material cultural relic left behind by our forefathers. 1. One may try a progressive accumulation of data using a one-track technique, such as microscopy, scanning and/or transmitting electron microscopy and synchrotron radiation, all in the domain of optics, or 2. One may also try to accumulate data by the use of a series of techniques in different nuclear domains, such as Neutron Activation, Radiocarbon and Thermoluminescence dating and Neutron Tomography. One may add to this epigraphy and paleography and DNA when it concerns manuscripts,

as well as exegesis and history when it concerns the translation of a text and its context.

Often any one of the optical techniques will offer new insight into the essence of an artifact, how it was made and of what it was made. But in a project that treats Qumran and the Dead Sea manuscripts, we have opted for all the studies that can provide information of what went on in Qumran more than 2000 years ago. This approach we have dubbed "Holistic Qumran", or "a Trans-disciplinary research of Qumran", both encompassing the same goal to get as much as possible information of the historic importance from monotheistic Jewish writings that is the basis of the entire western civilization of today. Just bear in mind that there are on earth 15 million Jews, 2.1 billion Christians and 1.9 billion Muslims, which are more than 4 billion monotheists of the six billion people that inhabit the globe today. The manuscripts of Qumran are the oldest and most complete set of writings that are the basis of monotheism we have access to since the late 1940s. That is the importance of Qumran.

ACKNOWLEDGEMENTS

The organizer of the workshop, Jan Gunneweg is deeply thankful for the fruitful collaboration of his two co-organizers and co-editors of the Qumran Workshop and Proceedings, Annemie Adriaens of Gent University (previously the chair of COST G8) and Joris Dik of the Technical University of Delft

Moreover, my deepest appreciation to NIAS rector, Wim Blockmans and Jos Hooghuis and his staff who invited me for a yearlong research at the Netherlands Institute of Advanced Studies in Wassenaar and the opening of the lecture series at the Institute with a contribution of an overview of ten years Qumran research.

Furthermore, sincere thanks to Wim van Saarloos of the Lorentz Center at Leiden University who with Martje Kruk, Henriette Jensenius and the workshop coordinator, Gerda Filippo made the workshop into a real trans-disciplinary encounter.

Further thanks are due to all the collaborators in the 'Holistic Qumran' workshop, who came from the Netherlands and from five foreign countries in Europe, the USA and Asia and who also contributed to the present Proceedings of the workshop, in spite of the short time at their disposal due to each one's own academic duties.

Thanks to the Dean of Physics at Leiden University who invited me to present the Lorentz Center Highlights in 'This week Discoveries Lunch colloquium' entitled "Qumran, a scientific jigsaw puzzle; The Dead Sea scrolls and Archaeometry".

I am in debt to Joris Dik of Material Sciences and the entire team of the Chemistry Department at the Technical University of Delft who were so willing to hear me out regarding the preparation of making soap at Qumran. The cooperation of Ger Koper is highly appreciated. Special thanks are due to a group of youngsters of the Honour Class of future archaeometrists who will eventually replace the old guard in applying science to archaeology and geology.

Special thanks to Florentino García Martínez who gave us the opportunity to publish in the STDJ Series. We thank also Machiel Kleemans of Brill Publishing House in Leiden for the opportunity to spread the newly obtained scientific data concerning the archaeology of Qumran and the Dead Sea manuscripts.

Furthermore, thanks to the European Community COST Actions G8 and D-42 in which framework five of the present authors (Adriaens, Dowsett, Gunneweg, Rasmussen and Rabin) performed their research. Finally, my sincere thanks to all the archaeologists who provided me with the bio- and material relics of Qumran and the discussions we had prior to analysis. Thanks to the Israel Antiquities Authority (IAA) with Pnina Shor, Elena Libman at the Dead Sea scroll restoration lab and Hava Katz and Allegre Savariego at the Rockefeller Museum in Jerusalem. And last but not least, sympathy to my wife Hanna and friends who as usually followed the Qumran endeavor with great enthusiasm.

LIST OF CONTRIBUTORS

Johannes van der Plicht, Center for Isotope Research, University of Groningen, Groningen and Faculty of Archaeology, University of Leiden, Leiden, the Netherlands
j.vander.Plicht@rug.nl

Kaare L. Rasmussen, Institute of Physics and Chemistry, University of Southern Denmark, Odense, Denmark
klr@chem.sdu.dk

Ira Rabin, Federal Institute for Materials Research and Testing, 12200 Berlin, Germany
ira.rabin@bam.de

Oliver Hahn, Federal Institute for Materials Research and Testing, 12200 Berlin, Germany

Bridget Murphy, Kiel University, Germany
Murphy@physik.uni-kiel.de

Martin Mueller, GKSS Research Centre Geesthacht,Max-Planck-Straße 1,21502 Geesthacht Geesthacht; also Kiel University, Germany
Martin.Mueller@gkss.de

Marine Cotte, Louvre, Paris
marine.cotte@culture.gouv.fr

Timo Wolff, Federal Institute for Materials Research and Testing, 12200 Berlin, Germany; & the Technical University of Berlin, 10623 Berlin, Germany

Emanuel Kindzorra, Federal Institute for Materials Research and Testing, 12200 Berlin, Germany; and Technical University of Berlin, 10623 Berlin, Germany

Admir Masic, Max Planck Institute of Colloids and Interfaces, 14424 Potsdam, Germany

Ulrich Schade, BESSY, Berlin, Germany

Gisela Weinberg, Fritz-Haber-Institut of the MPG, 14195 Berlin, Germany

Emanuel Tov, Hebrew University of Jerusalem, Mount Scopus, 91905 Jerusalem, Israel
tov@mscc.huji.ac.il

Sasja van der Vaart, University of Leiden
sasjavdv@gmail.com

Alexandra van den Broek, University of Leiden
amlb@planet.nl

Laura Klerkx, University of Leiden
l.klerkx@umail.leidenuniv.nl

Nanda de Vree, University of Leiden
nandadevree@hotmail.com

Marta Balla, Nuclear Reactor at the Technical University of Budapest, 1520 Budapest, Hungary
balla@reak.bme.hu

Gila Kahila Bar-Gal, School of Veterinary Medicine, The Hebrew University, 76100 Rehovot
bargal@agri.huji.ac.il

Jan Gunneweg, Archaeometry, Institute of Archaeology/ Archaeometry, The Hebrew University of Jerusalem, 91905 Jerusalem, Israel
jan.gunneweg@huji.ac.il

Annemie Adriaens, Department of Analytical Chemistry, Ghent University, Belgium
Annemie.Adriaens@ugent.be

Mark Dowsett, Department of Physics, Warwick University, UK
m.g.dowsett@warwick.ac.uk

Eberhard Lehmann, Paul Scherrer Institute, Switzerland
eberhard.lehmann@psi.ch

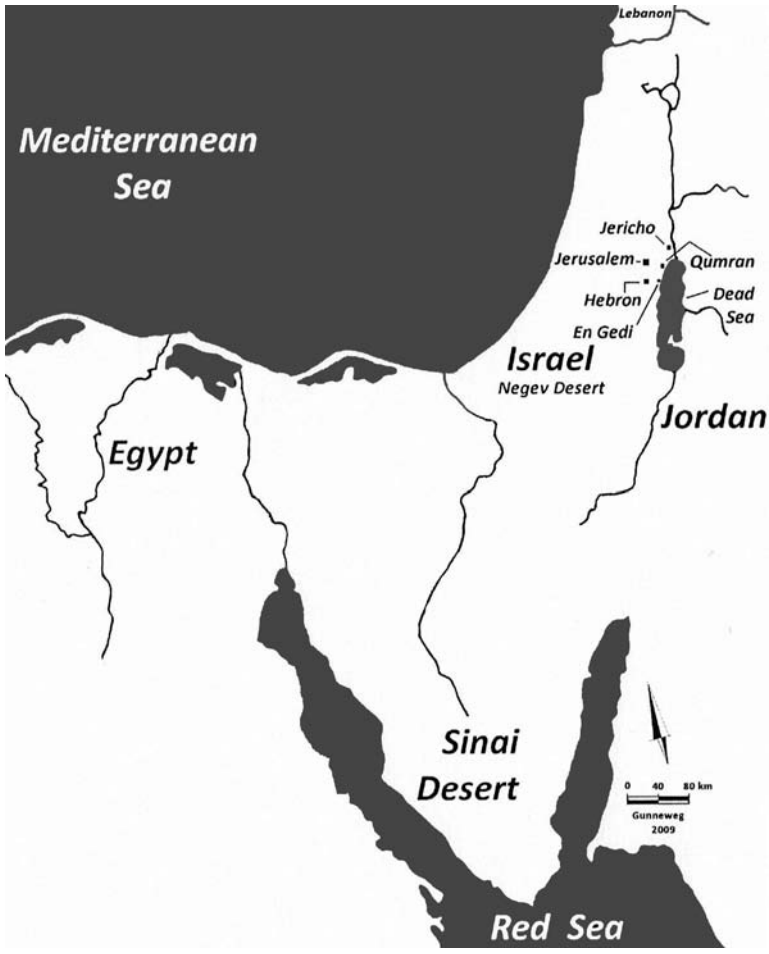
Yoav Farhi, The Hebrew University, Jerusalem
vavy15@gmail.com

L. Bouchenoire, XmaS beamline, ESRF, Grenoble, France
bouchenoire@esrf.fr

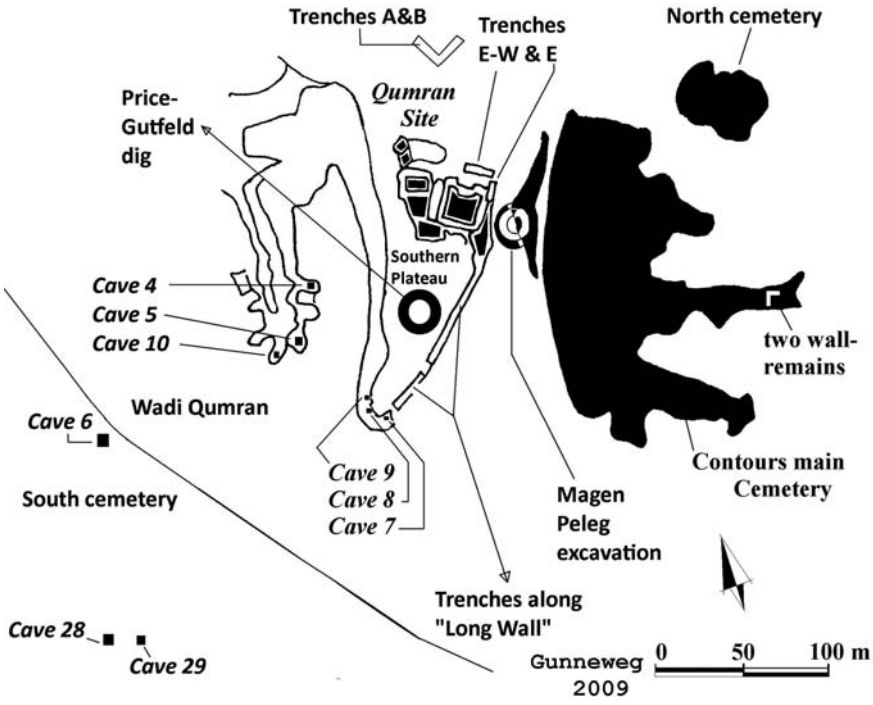
Katharina Galor, Brown University, USA
katharina_galor@brown.edu

Charles Greenblatt, Faculty of Medicine, Kuvin Center, Hebrew University of Jerusalem, Israel
greenbl@cc.huji.ac.il

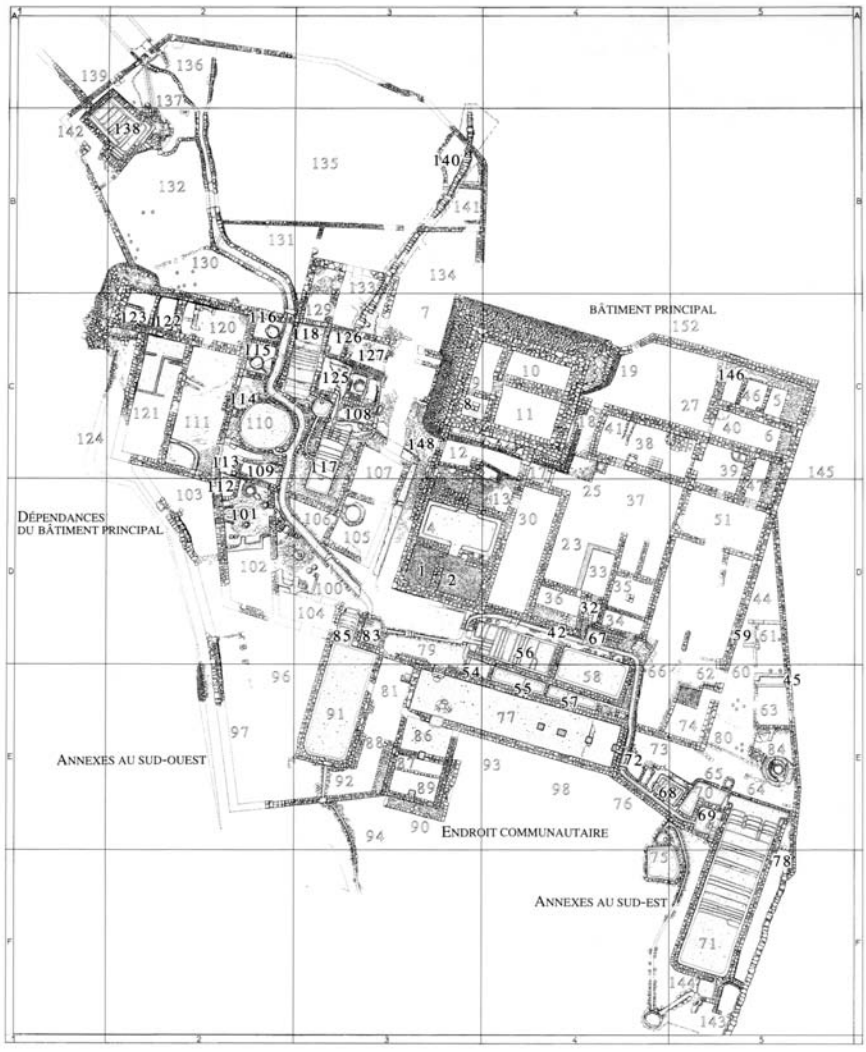
Joris Dik, Material Sciences, Technical University of Delft
j.dik@tudelft.nl



Map 1. Schematic map of Middle East, Israel & Qumran



Map 2. Schematic map of Qumran Site in the center, the Caves and Cemetery are at the right, the caves at the left. New Excavations of Magen/Peleg and Price/Gutfeld encircled. (See color plates Figures XI and XII)



Map 3. Excavation Plan of the Qumran settlement (credit to Ecole Biblique of Jerusalem, Humbert & Gunneweg 2003)

INTRODUCTION

Jan GUNNEWEG
Archaeometry-Archaeology, The Hebrew University of Jerusalem

In 2006, Israel (Robert) Auman of the Hebrew University of Jerusalem received the Nobel Prize for his idea of the "Law of repeated games" in economics. The model is based on the principle that one keeps contact in practical finances by applying the concept of wheeling and dealing, just as in the bazaars of the Middle East. By applying the theory to the present research of Qumran, the more repeated efforts that will take place, the smoother will be the collaboration between the parties involved. Too high and/or one-sided ventures will perhaps succeed, but continuous one-sided ventures will result in failure. However, searching for collaboration and giving in at times and keeping the momentum alive, will sooner or later pay back and turn into a firm basis for collaboration of long duration. It is the latter that is needed in scientific research at present.

Having been appointed to be a NIAS fellow (the Netherlands Institute of Advanced Studies) with obligations to the Lorentz Center of Leiden University for holding a workshop, it was expected that I would continue my work of ten years of Qumran research that had started at the Hebrew University in collaboration with Marta Balla at the nuclear reactor of the Technical University of Budapest and with Charles Greenblatt of the Kuvin Center for Tropical Diseases of the Hebrew University.

To invite scholars that are of the same opinion as you is, of course, good for one's own image. Therefore, one of the most important issues for organizing a layout of a Qumran meeting was to invite people who were known to be against Roland de Vaux, the excavator of Qumran, with his classical theory, which says that Qumran was a sectarian community. Norman Golb of Chicago who sees Qumran as a garrison is almost everywhere banned where Qumran is mentioned and as such he is interviewed on every TV station and daily newspaper. So was it in the Qumran meeting at Brown University in 2001 whose press gave the impression that the most important issue was that, again, N. Golb

was not invited. Thus, in order to follow my rules for collaboration, I wrote N. Golb twice inviting him to come to the Lorentz Center and to lecture, whereas the Congress would cover his trip and lodging. He answered me that it was not convenient to be in Holland during the days of 21-25 April 2008.

The second scholar, who was invited, also being on the black list of those who do not believe in the classical theory of De Vaux, was Katharina Galor of Brown University whom I asked whether she could lecture on 'gender at Qumran' that usually is neglected, primarily because the present theory is that the population at Qumran was strictly male. Galor accepted and her paper can be found in Chapter 3.

In June 2007, in preparation for the Qumran workshop planned for April 21-25, 2008, new interesting relics of material- and bio cultures had to be collected to be treated in a trans-disciplinary workshop at the Lorentz Center to continue the trend of collaboration within the Mathematics' and Physics' Institute at Leiden University of which the Lorentz Center is an integral part.

The following topics were on the agenda for the workshop, for which artifacts had to be gathered, scholars be convinced to collaborate and people invited to deliver their newly obtained analytical data in the Qumran workshop. With some research going on already, the following questions were formulated:

1. Is the Qumran marl/clay as found by Yitzhak Magen and Yuval Peleg at their excavation of 1993-2004 —and heralded in the media as the final blow to Qumran sectarian Jews with their library of holy scriptures—indeed the clay that was used at Qumran for making pottery in their potter's workshop, the only reason why these people lived at Qumran?
2. What is the final setup for Qumran by ignoring the existence of the Dead Sea scrolls as written by and for the Qumranites?
3. What is the date of the only jar (Jar-35) found so far that originated in Jerusalem, since no other pottery has been traced to the capital of Israel in those days? Is the jar of the 8th century AD as Thermoluminescence suggested, in which case some different population lived at Qumran? Was this jar originally filled with wine, as Salvado Buti et al. at Barcelona University have stated?
3. Do the animal bone burial piles found within the Building Complex, but especially on the southern plateau outside the buildings by Randall Price and Oren Gutfeld really belong to the same period

as the material content found within the Qumran building complex, meaning that they all belong to the same period?
4. What is the composition of the ink used for the writing of the Dead Sea scrolls and what is the composition of the paint used for the writing of the about 80 Qumran ostraca? The latter is important if restorers in the museums have found that the writing on ostraca is flaking and looses its grip on the ceramic.
5. What is the reason that the parchment scrolls are degrading? Or are they? And if one knows that, what can be done?
6. Can DNA provide a final answer as to the identification of the animal bones that have been too long in a hot and too dry atmosphere and can we establish where these animals originate?
7. What can be said about the material culture at Qumran concerning gender, since some tombs have been excavated that contained female skeletons?
8. Can illegible and un-cleaned coins be read by neutron tomography?
9. Can an exegete of the Dead Sea manuscripts obtain information from analytical science research to interpret the text of the manuscripts or to find the site where the scribe was located when he wrote the single pages or complete scrolls? And do exegetes have specific requests from science in future research?
10. What about balsam that has been found near Cave 1 at Qumran?
11. What else can be brought forward to point to celibacy at Qumran; and what can be said about gender altogether?
12. What can be learned from the identification and preservation of textiles that were found in the Cave of Letters from the time of Bar Kochba, a Jewish freedom fighter against the Romans in 135 AD vis-à-vis textiles found in the Qumran Caves?

The workshop at the Lorentz Center would be entitled "Holistic Qumran" that would include text critics, paleography and history, all domains in the Humanities and the Social Sciences (the NIAS context), and to combine it with research executed by the so-called Natural Sciences (the name Hard Sciences is questionable). The use of the word 'Holistic' in the workshop presentation and in the title of the Proceedings involves an inter-disciplinary study employing various analytical techniques and methods of historic research in different domains encompassing all, hence Trans-disciplinarity.

In the present book, the word 'trans-disciplinarity' will be used because the topics treated and the techniques applied solving pending questions on Qumran and the Dead Sea scrolls encompass most of the cutting edge techniques available today. These techniques start with the rather subjective way of making use of paleography to obtain a date for a manuscript or telling apart different writers basing oneself on the way certain letters of the alphabet were written or copied by the scribes on the one hand to sophisticated neutron-tomography and CT-laminography on the other whereby the human eye is less involved, but objective data assembling by up-to-date equipment and its evaluation by highly developed statistics instead.

For those who are unfamiliar with the Qumran story and why is it still so attractive to date, the answer could be that the importance of Qumran in the World today, boils down to a single sentence: "Qumran presents the oldest relics in written form of Monotheism in the Land of Israel".

Monotheism is the technical term used to describe a religion that allows only a single God, without parents, a husband or wife and children.

Monotheism is at present practiced by about 4 billion Jews, Christians and Muslims, about two-thirds of the world population. The nucleus of a monotheistic idea could have been Egyptian, which, however was short-lived and forgotten altogether. One may say that monotheism started with the Jewish religion, 1000 years before the Christians continued it based on Jesus Christ, a Jew, and 1632 years before the Muslims made it theirs too.

It is rare to find hard material- and bio cultural evidence to trace monotheism in time. The only people, who have succeeded to find tangible evidence for the beginnings of monotheism, are the archaeologists. An archaeologist slowly uncovers levels of civilization whose artifacts and written material can be traced to the place where they have been made, as well as dated by various dating techniques.

If one applies this to the find of the Dead Sea scrolls, found at Qumran, one can obtain a treasure of information that one did not possess before the time that these scrolls were found between 1947 and 1956.

But often one asks the question, why are these scrolls still getting the attention and research money during 60 years after they were found? The answer lies, in my opinion, in the recognition of what these texts mean to monotheism, the backbone of the Western Hemisphere.

The story of discovery is quite banal. A young shepherd of the Ta'amireh Bedouin tribe guarding his flock of goats in the Judean Hills lost one and went after it. He found the goat as well as the entire parchment scroll of the Prophet Isaiah 2000 years old. This was just the beginning.

The earliest Hebrew text we have has been preserved in the agricultural calendar at Tell Gezer that dates back to the 10/9th century BC. From this text alone, however, no argument can be made for a monotheistic god since god is not mentioned.

On the other hand, two fragments of silver amulets were found at the Ketef Hinnom (translated: the shoulder of the Ben Gehinnom Valley) at the foot of the St. Andrew's church of the Scots in Jerusalem. The amulets were dated to the end of Iron Age II (7/6th century BC). They quote a passage of the priestly benediction where the name of God is named repeatedly: "*May the Lord (tetragram YHWH) bless and keep you. May the Lord make his face shine upon you and be gracious to you. May the Lord lift his face to you and grant you peace*" Num 6: 24-26. And from the context of the Old Testament in Numbers one may extrapolate that this is a monotheistic text.

The Dead Sea manuscripts consist of an estimated 930 (complete and partial) scrolls. These texts are dated by paleography, whereas others by radio carbon dating (AMS-C14). They provided us with an uninterrupted group of monotheistic Jewish texts from 350 BC—50 AD, two-thirds of them being biblical of character.

Our present Old Testament is based on that of the Masoretes of the 10th c. AD that now is accepted as going back to the Herodian time scrolls found in the Judaean Hills.

On the other hand, in the Qumran texts, we can find variations of the same biblical texts of the Masoretes that did not make it to the canonization of what we have at present by the mere fact that when the process of canonization began, the Qumran texts were already lost to the Jews, since Qumran laid barren since the late 60s AD. Qumran proves the shaping of the biblical texts as we have it today, be it through a long process of variant readings of holy texts that the scribes penned down.

Since 2005, we have access to a complete translation of the entire set of the Dead Sea scrolls that was achieved by a very large team of eminent scholars from all over the world under the supervision of Emanuel Tov as editor in chief.

The reading of a manuscript, as every epigrapher knows too well, is only final if all words and letters can be read. The Dead Sea scrolls are often very fragmentary and one needs a *tour de force* to produce a translation that is indeed final. As long as one has a parallel text in other scrolls or in the Masoretic text, one will perhaps be able to fill in the missing letters or words.

But, in the case that the Qumran text is a variant of an existing biblical text, one needs every letter to interpret the variant, which can sometimes change the entire meaning of a sentence or a thought.

Since 2008, the Israel Antiquities Authority (IAA) has decided to photograph in high resolution once again every bit of a page or a small fragment in order to obtain a second set of updated Dead Sea scroll material and to "read" the letters that have disappeared because the parchment has become dark and to focus on the difficult or almost obliterated letters on the very edge of a broken piece of parchment by the means of Infra-red photography. The tests have started in the last quarter of 2008.

When this enormous task will be finished, the newly detected text will again be open for translation that may be different in certain instances from the translation we have today. We can, thus, foresee that in the coming decades, there will be enough work for tens or even hundreds of text restorers and exegetes.

Simultaneously with all this, we have also to take care to preserve the Dead Sea scrolls themselves, so that also natural science will have an enormous impact, on the condition that the scrolls can be analyzed first and taken care of according to the results of the analyzes that one will obtain.

Present co-editors in the Holistic Qumran Proceedings are my colleagues, Annemie Adriaens of the Chemistry Department of the University of Ghent and Joris Dik of the Material Sciences at the Technical University of Delft.

It is a good *omen* that NIAS, primarily Humanities and the Lorentz Center in the domain of Physics and Mathematics have set up an interdisciplinary research program for its fellows. A decade ago, this would be unheard of.

The order of the following chapters is organized according to the alphabetical order of its principal contributor.

In Chapter 1, Annemie Adriaens, Mark Dowsett, Eberhard Lehmann, Yoav Farhi, Jan Gunneweg and L. Bouchenoire will give an

account of their findings regarding bronze coins found in Israel that, when the pilot study will turn out with good results, will perhaps make illegible coins legible in the future after having been submitted to neutron-tomography. On the other hand, the chemical composition of corroded bronze coins will be discussed too.

In Chapter 2, Joris Dik, Lukas Helfen, Peter Reischig, Jorik Blaas and Jan Gunneweg will show the future approach to study parchment, papyrus and ink by laminography and micro-X-Ray Tomography, a study that first was initiated by Gunneweg, Cotte, Mueller and Murphy at the European Synchrotron Radiation Facility (ESRF) in Grenoble (Proposal EC89) and that now is enlarged with micro-tomography under the guidance of Lukas Helfen at the station ID 21 at the ESRF and Joris Dik in Delft.

In Chapter 3, Katharina Galor will give an overview of relics of gender at Qumran and compare it to what has been found at other sites in the Dead Sea area. The role of women is rather unclear and has also been biased, especially when it concerns a sectarian group of males according to the ancient writers, Flavius Josephus, Pliny the Elder and Philo of Alexandria.

In Chapter 4, Marta Balla and Jan Gunneweg will provide an answer whether the marl/clay found in Pools 71 and 58 was used to make local pottery at Qumran. Also pottery from the Cave of Letters, Engedi, and Masada will be touched upon to show what is the connection, if any, with Qumran, based on the pottery analyzed so far.

In Chapter 5, Jan Gunneweg will give an introduction to the unexpected find of about 100 piles that consist of broken pots together with animal bones that were buried in small pits and that were found primarily on the southern plateau and the eastern area between the buildings and the cemetery where nothing had been estimated to be situated.

In Chapter 6, Gila Kahila Bar-Gal, Tzviki Rosenberg and Charles Greenblatt will explain a new approach to obtain DNA from human and animal bones in spite of the fact that in most of the cases the Dead Sea environment is too hot and too dry and salty to preserve any of the collagen needed for DNA research.

In Chapter 7, Bridget Murphy, Marine Cotte, Martin Müller, Marta Balla and Jan Gunneweg will describe the way our group of scholars dealing with the deterioration of parchment and ink of the Dead Sea scrolls went about at the Synchrotron facility in Grenoble by applying micro-FT-IR spectrography.

In Chapter 8, Hans van der Plicht and Kaare Lund Rasmussen will communicate about radiocarbon dating of Qumran textiles and parchment and how they have been infected by previous castor oil treatment of parchment and textiles, half a century ago. How shall one go about, when one knows that the majority of organic material was contaminated?

In Chapter 9, Ira Rabin Oliver Hahn, Timo Wolff, Emanuel Kindzorra, Admir Masic, Ulrich Schade & Gisela Weinberg will explain the way(s) to use ink as the chemical fingerprint for tracing the various scribes of the Dead Sea manuscripts.

In Chapter 10, Kaare L. Rasmussen, Jan Gunneweg, Johannes van der Plicht, and Marta Balla will provide an account on the age of Jar-35 as well as the shards found in the animal bone piles by Thermoluminescence. Rasmussen will also give the various possible reasons why the dating of the jar was at an early stage set for the 6th-7th centuries after Christ.

In Chapter 11, Kaare L. Rasmussen, Andrew Bond, Greg Doudna and Jan Gunneweg will shed light on the 'mass of iron' that de Vaux found in Room 104 at Qumran, without that someone ever has tried to trace the relic and to submit it to analysis.

In Chapter 12, Emanuel Tov who has been the Editor in Chief of the Dead Sea texts will provide us with an overview of what he expects from science to help an epigrapher, an exegete and a historical or biblical scholar.

In Chapter 13, Jan Gunneweg provides an introduction that led to the Honour Class cooperation of Leiden and Delft Universities to manufacture lye that could have been used for several functions within the sectarian community at Qumran; making soap being one of them. The produced lye could also have served as a tanning product for leather (sandals).

In Chapter 14, Sasja van der Vaart, Alexandra van den Broek, Laura Klerkx en Nanda de Vree will report on their first laboratory test in Archaeometry that they executed in the framework of the Honour Class of the Technical University of Delft and Leiden University in collaboration with the Hebrew University of Jerusalem.

In Chapter 15, Jan Gunneweg will give an overview of the elemental chemical composition of Dead Sea mud as analyzed by INAA, XRF, XRD and SEM, which will be a needed ingredient to reckon with when one will start seriously with an elemental research on the Dead Sea

environment that has probably been the cause of the lifespan of the Dead Sea scrolls.

An epilogue will conclude the Proceedings with a prognosis of future research on Qumran archaeology and the Dead Sea scrolls. The future Qumran research will be complimented with comparisons to cultural relics found at other sites, such as Masada, Engedi and the so-called Christmas Cave that lately has been rediscovered in the delta of the Kedron wadi near the Dead Sea.

Additional published papers of the aforementioned authors have been placed at the end of this volume so that one receives a wide panorama of scientific Qumran research accomplished so far.

The figures run from 1-51. Figures numbered in roman are in color and can be found in the plates. Graphs belong to each author. The Plates run from Figure I-XIII. Most of the photographs are of J. Gunneweg, except that of J. Dik, those of the coins by Roger Gibbons, and where indicated differently.

CHAPTER ONE

THE COIN BENEATH THE CRUST: A PILOT STUDY OF COINS FROM THE MEDITERRANEAN COAST OF ISRAEL

Annemie ADRIAENS[a], Mark DOWSETT[b], Eberhard LEHMAN[c], Yoav FARHI[d], Jan GUNNEWEG[d] and L. BOUCHENOIRE[e]
[a]*Department of Analytical Chemistry, Ghent University, Belgium,* [b]*Department of Physics, Warwick University, UK,* [c]*Paul Scherrer Institute, Switzerland,* [d]*The Hebrew University, Jerusalem,* [e]*XMaS beamline, ESRF, Grenoble, France*

Abstract. We describe the application of synchrotron X-ray diffraction and neutron micro-tomography to three ancient bronze coins from the Mediterranean coast of Israel. Key questions are whether neutron tomography can contribute to the reading of features on corroded coins by allowing the corrosion crust to be stripped away in cyber-space using surface and volume rendering techniques, and to what extent this type of non-destructive measurement can inform possible conservation action. In addition, the X-ray diffraction experiments allow the main composition of the varicolored surface corrosion to be deduced. We show that both X-ray and neutron data are of considerable help to numismatic study of the coins.

Keywords. Coins, Mediterranean coast of Israel, Neutron tomography, numismatics, synchrotron radiation X-ray diffraction, corrosion, legibility

Introduction

Ancient metal artefacts are usually found covered with a crust of corrosion in which some of the burial medium may be embedded. Since conversion of a metal to a corrosion product usually involves considerable swelling of the surface, relatively undamaged artefacts may be concealed beneath crusts of a millimeter or so in thickness.

Using three untreated coins with different degrees of corrosion we explore the hypothesis that neutron micro-tomography (n-μT) might be used to obtain a complete 3-D volumetric absorption map of the artefact in its as-found condition, and then volume and surface rendering techniques, common in tomography, could be used to distin-

guish between remaining metal and corrosion crusts. With sufficient resolution, it might even be possible to read the inscriptions on an artefact such as a coin, without disturbing the corrosion, or at least obtain more information as to the original nature of the artefact.

Since the crusts on the coins exhibited quite distinct patches of red, red-orange, grey-green and black corrosion, we decided to use synchrotron X-ray diffraction (SR-XRD) to look at the average composition of the top 10 μm or so of each. Because of the speed inherent to the combination of synchrotron X-ray intensity (~10^{12} photons s^{-1}) and the use of an area detector, we could obtain a good survey of the coins in an hour of beam time. In future work, it may be possible to correlate the specific nature of the corrosion with the neutron absorption, and this is demonstrated to some extent here.

1. *Description of the coins*

The three coins (designated A, B and C) are discussed in chronological order, starting from the oldest. Photographs of the obverse (front side of the coin) and the reverse (back side of the coin) are shown in figure 1 and Figure I*.

Coin B was found in the old city of Caesarea Maritima and dates to ca 610-641 CE. The coin is severely corroded, having a red-orange crust with patches of grey-green, and no obvious surface marks are preserved. Its visual identification is therefore somewhat conjectural. We suggest that it is a Byzantine coin minted under Heraclius. Coin A was found at the city of Átlit. It is in a better state of preservation. The corrosion is thin, but again red-orange with diffuse black and grey green regions. Nevertheless, it is easier to attribute to the Arab-Byzantine transition period of the mid seventh century CE. Coin C was also found at the city of Átlit. The corrosion on the two faces is different, and intermediate in thickness to that on coins A and C. One face is similar in appearance to the other coins, whilst the other is covered with a redder patina with a black ground. This may indicate that one side of the coin was burnt. The identification of this coin as Islamic is possible only due to the tomographic image (see later) which revealed the Arabic letters under the corrosion. A detailed numismatic report of the three coins can be found in Table 1.

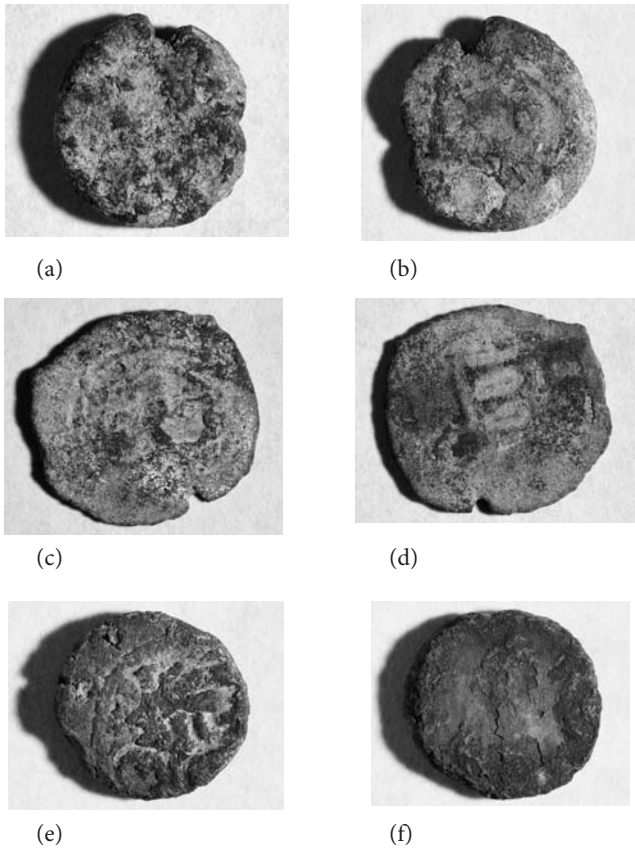


Figure 1. Photographs of the obverse (a, c and e) and reverse (b, d and f) of respectively Coins B, A and C. A color version is available in the plates section (Figure I).

2. *Experiments*

X-ray diffraction experiments were performed at XMaS (Station BM28, European Synchrotron Radiation Facility, Grenoble, France). At this station, a beam with a wavelength of 1.5498 Å and with dimensions of 1 mm × 200 μm was used. A 2D Mar CCD 165 detector (Mar USA Inc., Evanston, IL, USA) was used to record the diffraction patterns. The angle of the camera to the beam was 35° in order to be able to acquire the signals 2Θ values of 15° and 75°. Under these conditions, the diffraction centre is outside the field of view of the camera,

Table 1. Numismatic data of coins B, A and C.

Coin B	**Heraclius (610-641 CE)?** *Obv.* Bust of Heraclius to l. and bust of Heraclius Constantine to r. No legends visible. *Rev.* I+B (?), in exergue: [A]Λ[EΞ] Alexandria, 613-618 CE. Bronze,↑, 6.34 gr., 18mm. *Cf.* Grierson P., *Catalogue of the Byzantine Coins in the Dumbarton Oaks Collection and in the Whittemore Collection*, II, Part I, *Phocas and Heraclius (602-641)*. Washington, D. C. 1968. P. 334-335, nos. 189.1-190.
Coin A	**Arab Byzantine,** 7th Century (circa 650 CE). *Obv.* Standing figure (emperor?) standing, facing; holding long cross in r., and globus cruciger in l., above the cross: O. Dotted border. *Rev.* Cursive m with cross above and pellets or stylized snakes (?) between the uprights of the m. Illegible legend in l., no legends visible in exergue. Dotted border. Bronze,↑, 4.43 gr., 24mm. *Cf.* Album S. and Goodwin T., *Sylloge of Islamic Coins in the Ashmolean, I, The Pre-Reform Coinage of the Early Islamic Period*. Oxford. 2002. P. 79 (Type E); Goodwin T., *Arab-Byzantine Coinage*. London. 2005. P. 40, no. 37.
Coin C	**Islamic –** Umayyad (post reform) or ʿAbbāsid (8th-9th centuries CE) *Obv.* Uncertain Arabic inscription inside a circle. *Rev.* No legends visible Bronze,↑, 3.66 gr., 15mm.

and the camera plane intersects the diffraction cones at an angle to produce elliptical "rings". The images were processed using a new software program, esaProject (© EVA Surface Analysis), which was developed to extract spectra from such images.

Neutron μT experiments were performed at the spallation neutron source SINQ, located at the Paul Scherrer Institute, using the ICON beam line for cold neutrons. The advantage of cold neutrons for imaging purposes is their higher contrasts for most of the sample materials and the higher detection probability. A pixel size of 13.5 μm was used. The set-up is shown in figure 2.

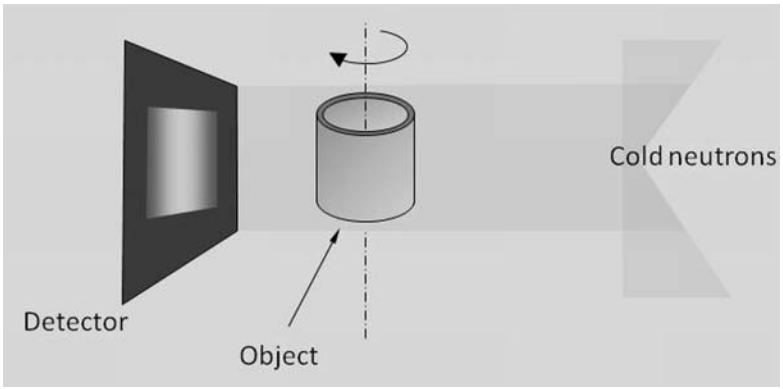


Figure 2. Principle of cold neutron tomography

3. *X-ray diffraction data*

The three coins show mainly the presence of black and red to orange corrosion products which are a mixture of cuprite (Cu_2O) and tenorite (CuO) in various proportions – cuprite being responsible for the red and tenorite for the black coloration. A typical spectrum is shown in figure 3a. The grey-green patches on coins B and C prove to be a mixture of cuprite, tenorite and paratacamite (a copper hydroxychloride), as is shown in figure 3b.

4. *Neutron tomography data*

Photographs and near-surface neutron tomography images for the observe and reserve of the three coins are shown in figure 4 and Figure II*. In the case of coin B (top row) the rendered surface in the tomography passes beneath the paratacamite regions at the bottom of the photograph (third pane from the left). These appear as craters in the tomographic surface in the right hand pane. The rendering also passes beneath the tenorite, revealing surface relief filled by the oxide. This is a clear demonstration that the neutron stopping power of the different corrosion products is sufficiently different for them to be distinguishable. A similar effect is seen for coin A (second row), with details of the embossing on the coin being more prominent in the rendered surfaces from the neutron tomography (second and last pane) in comparison with the photographs (first and third pane). This is again because the

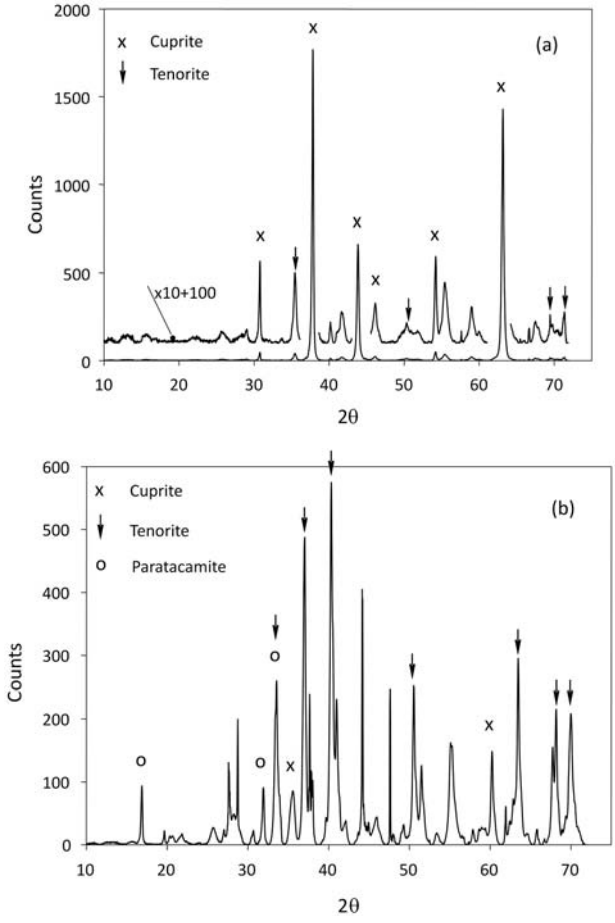


Figure 3: (a) Showing XRD characteristic of the orange-red and black regions on all 3 coins and (b) the grey-green regions on coins B and C.

rendered surface passes beneath the corrosion layer. Of course, photography using controlled illumination, filters and polarized light might achieve a similar effect, but as we show below, the distinction between corrosion and metal can be applied to any part of the volume of the coin to reveal internal features non destructively. The third row shows the comparison between photographs and rendered surfaces for coin C. Here, the improvement in readability is a distinct aid to identification, and even on the reverse side some evidence of embossing is revealed, albeit unidentifiable.

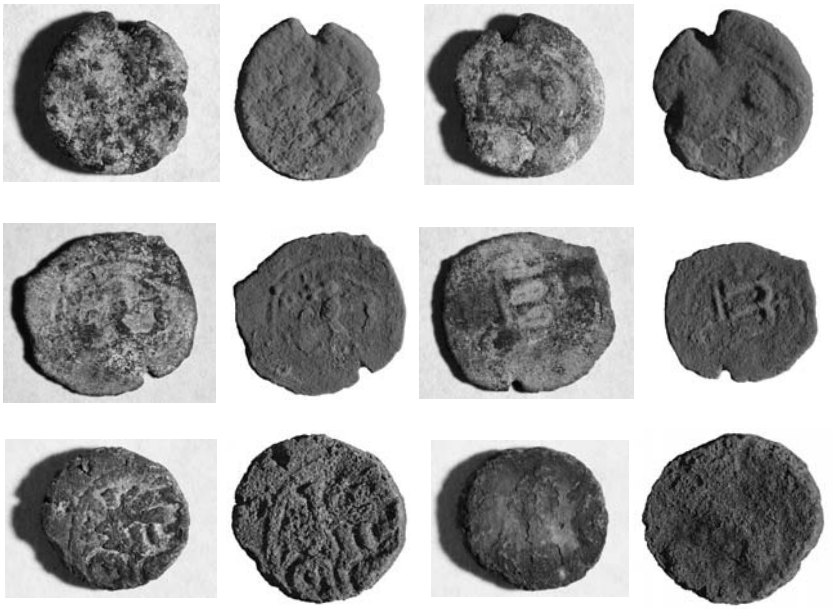


Figure 4. Top row: photographs and neutron tomography images of the surface of obverse (left) and reverse (right) of Coin B. Middle row: idem for Coin A. Bottom row: idem for Coin C. A color version is available in the plates section (Figure II).

Figure 5 and Figure III* show neutron tomography images from inside the three coins. For coin B a series is shown from one end of the coin to the other. The set of images shows a different attenuation for different regions, which is visualized by the different colors: brown, grey and red. It is clear that the coin has corroded from within and without, with internal corrosion occurring in cracks and voids causing the structure to swell. The images show that there is hardly any metal remaining.

The cross section of coin A shows the presence of two different materials and several cracks inside the coin. Some of these show evidence of developing corrosion—perhaps a precursor to the fate of coin B. Coin C on the other hand is composed of a quite homogenous material as judged by the neutron stopping power, although with a different porosity through the coin: the inner part of the coin C seems to be quite porous, whereas the outer part does not.

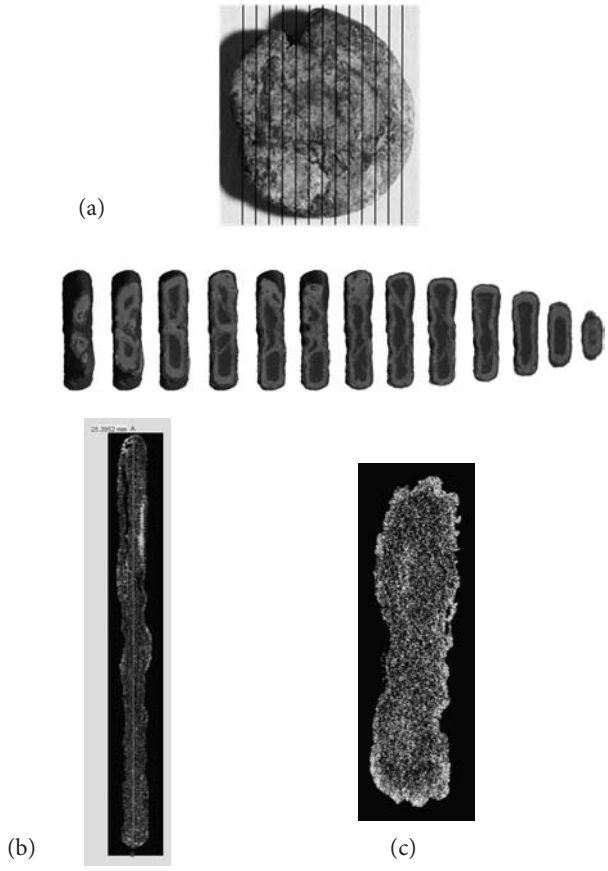


Figure 5. Neutron tomography cross sections of (a) coin B showing a series across the width approximately on the lines in the photograph above; (b) coin A and (c) coin C. A color version is available in the plates section (Figure III).

Conclusions

In this work we have examined three corroded coins which originate from the Mediterranean coast in Israel. Numismatic data show that they are from the 6th to the 9th century CE. The corrosion crusts mainly constitute tenorite and cuprite with patches of paratacamite. Neutron tomography data are able to visualize the inner structure of the three coins showing the heterogeneity and the presence of cracks and different chemical compounds as also indicated by the XRD data.

Surface rendering of volumes in a particular range of neutron stopping power reveal the metal surface and can increase the readability of the markings (if any remain). In particular, for coin C, the neutron tomography data contribute to the identification of the coin through its surface inscription.

Acknowledgements

The authors would like to thank Melanie Salque in helping with the interpretation of the neutron tomography images and Roger Gibbons for the photography of the coins. This work would not have been possible without the support of COST Actions G8 and D42, and EVA Surface Analysis (UK).

CHAPTER TWO

A SHORT NOTE ON THE APPLICATION OF SYNCHROTRON-BASED MICRO-TOMOGRAPHY ON THE DEAD SEA SCROLLS

Joris DIK[a], Lukas HELFEN[b,c], Peter REISCHIG[a,c] Jorik BLAAS[a] and Jan GUNNEWEG[d]
[a]*Delft University of Technology, the Netherlands,* [b]*European Synchrotron Radiation Facility (ESRF), Grenoble, France,*
[c]*Karlsruhe Institute of Technology,*
[d]*Hebrew University of Jerusalem, Israel*

Abstract. The degradation of the Dead Sea Scrolls has been the topic of many scientific investigations. Such studies often involve the analysis of cross-sections, which requires sampling and subsequently the preparation of a fine cut through the parchment and its composing layers. The downside of this type of analysis, however, is threefold. First, it requires destructive sampling of the parchment. Second, mechanical polishing of the cross-section can introduce artifacts. Finally, a cross-section gives a rather arbitrary two-dimensional view on the more complex, three-dimensional structure of the parchment.

Keywords. Parchment, Laminography, Synchrotron-based computed micro-tomography (CT)

Introduction

In this short note we present preliminary, first-time results of a micro-tomographic visualization of the Dead Sea Scrolls. A small piece of parchment of approx. 1x1x4mm was scanned with synchrotron-based computed micro-tomography in order to visualize the internal structure of the parchment non-destructively and in a three-dimensional manner. The first results are promising in that the disentanglement of collagen fibers could be visualized. Interestingly, this process seems to originate from the *interior* of the parchment and then proceeds towards the outside. Further research, notably elemental and structural analysis, is necessary for a complete understanding of this process.

1. *Problems with conventional forms of cross-sectional analysis*

In various fields of conservation science, ranging from paintings to other layered artworks, the preparation of cross-sections is a routine form of analysis [1, 2]. Also in the case of the Dead Sea Scrolls, the stratigraphy of the parchment is of great interest to conservation scientists. A cross-section through the material can include the parchment itself, preparation layers, primer, ink as well corrosion layers or unintentional surface depositions. These individual layers as well as any interfacial reactions can then be examined with an array of (in) organic micro-analytical techniques.

However, sampling of valuable artworks is usually problematic, because it involves the physical removal of authentic material. Minimally invasive sampling techniques have been developed for parchment analysis. Puchinger et al. have published on sophisticated micro-drilling techniques that allows for the removal of samples with a diameter in the order of 0,1 mm [3]. Samples are usually taken from the edge of existing damages in the object, which may not necessarily relate to the questions at hand. Thus, sampling is sometimes determined by opportunity rather than necessity. In the case of the Dead Sea Scrolls, however, even the smallest sample may not be an option altogether.

But even if permission for sampling is granted, a second problem occurs during the preparation of cross-sections [4]. The sample is imbedded in a liquid polymer and processed further after curing. The block of resin is then cut in half and polished until a vertical cut through the imbedded sample is obtained. The grinding and polishing, however, can induce artifacts. This is the case when the sample and the imbedding medium differ in hardness, which notably applies to biological materials, such as parchment. Depending on their mechanical properties, fragments of the sample can be displaced or dispersed throughout the section surface. Another problem can be unintentional chemical interactions between the imbedding medium, notably its radical containing hardener, and the imbedded sample. [5] Another fundamental limitation is that a cross-section is a rather arbitrary 2d section through a more complex three-dimensional layered system.

2. *Structure and degradation of parchment*

The main component of parchment is collagen, which provides the mechanical properties due to its structure at different length scales [6}. At the microlevel collagen is structured in fibers of approximately 50-300 micrometer in diameter. These fibers are arranged in a felt-like network, providing tensile strength in the plane of the parchment. Individual fibers are composed of closely packed collagen fibrils, which are the main components of connective tissues. These fibrils are cylindrical in shape and range from 10-500 nanometer in diameter. Fibrils are in turn comprised of individual peptide chains. This hierarchical order at different length scales determines the mechanical properties of parchment.

The degradation of parchment can occur through oxidation, hydrolysis and gelatinization. These forms of chemical deterioration go hand in hand with a loss of structure. The parchment looses its intact, fibrilar morphology and turns into a more disordered system, as has been determined by diffraction studies. One of the open questions, however, concerns the origin and dynamics of the deterioration process. Where does the loss of structure start and how does it progress through the parchment? Can we visualize the difference between intact, ordered parchment and its more amorphous degraded parts?

In view of the constraints of conventional cross-sectional analysis above, we decided to test another method to visualize the internal structure of parchment: synchrotron-based computed micro-tomography (micro-CT). Over the past years the spatial resolution of micro-CT has been pushed beyond the 1 micrometer level. This has been made possible with the aid of synchrotron radiation, a highly monochromatic and partially coherent form of x-ray radiation. The beamline ID19 at the European Synchrotron Radiation Facility (ESRF) in Grenoble hosts state-of-the-art tomography instruments. Their work on paleontological objects using high-resolution tomography prompted us to consider its application on the Dead Sea Scrolls. [7] Again, the scientific challenge was to see whether we could visualize the micrometer network of fibers and observe structure differences between the intact and degraded parts.

3. *Methods*

Figure 6 shows stereoscopic images of a Dead Sea Scroll fragment on the left. This piece originates from a collection of four fragments that have been analyzed since 1975 and that finally were published in 1980 in *Nature* by Stephen Weiner and others of the Weizman Institute [8]. The fragments are loose pieces of parchment that De Vaux saved for scientific research. Our sample is numbered 4Q922. A caveat is in its place here, because according to the official numbering of the IAA the highest number from Cave 4 is 582 (4Q582). The numbering used in our study is of Milik and de Vaux that was attributed to samples for scientific research before the inventory of Cave 4 was definitive. We have no way of knowing whether this set of samples as treated by Weiner et al. came indeed from Cave 4. On the other hand, a similar case could be made for all other fragments that have been listed so that for the time being, we assume that also these samples came from Cave 4 at Qumran.

We photographed both verso and recto, i.e. what appears to be skin and flesh side. Note that the skin side has been prepared with a reddish ground, but does not bear any inscriptions in ink. We decided to remove a small strip from an edge of the fragment as indicated by the white frame. This strip measured approximately 1x1x4mm. This fragment was then imaged with micro-CT. The imaging took place at the ESRF, making use of the microtomography instrument at the imaging beamline ID19. A set of 1500 projection radiographs (exposure time 0.5 s each) of the fragment were acquired at an x-ray energy of 30 keV. The detector used was a Frelon 2k14 CCD (2048x2048 pixels) developed at ESRF, coupled by microscope optics to a scintillator crystal and yielding an effective pixel size of 0.7 μm. From the 2D projection data acquired we reconstructed several 2d slices perpendicular to the longest axis of the elongated sample. Their planes of view are transparently indicated in the verso image of the fragment on the left of figure 6.

4. *Results*

The right side of figure 6 shows slice reconstructions at different positions of our fragment. It must be noted that the parchment has been best preserved towards the edges, but interestingly shows a worse con-

dition in the *middle*. Both the edges show a fairly solid, homogeneous consistency, which also applies to the area directly below the primer. Small micro-cracks can be noted in these parts, but the overall condition appears pretty much intact. The most degraded volume occurs towards the center of the parchment, where we observe the disentanglement of string-like structures. Given their size (roughly 50 micrometer in diameter) and their morphology we tentatively identified these as collagen fibers. In some sections, it can be seen how these fibers take a sinus-like shape (see lower right slice) before full disattachment occurs from the collagen network.

In one slice we noted a medium-dense, agglomeration of cell structures, which could potentially be a hair follicle. In addition, the reconstructions also shows a multitude of medium to high absorbing particles, most likely minerals, on the surface of the parchment, which must be identified as the primer. More surprising, however, are globular, high-density particles *inside* the parchment, where the disentanglement of the collagen fibers has taken place.

5. *Discussion and conclusion*

The preliminary data above give rise to two questions concerning the deterioration process of the parchment. First, why does the degradation of the parchment occur from the inside out? The main pathways for parchment deterioration being oxidation, hydrolysis and gelatinization, one would expect the first symptoms of degradation at the outside of the material, which is more exposed to ageing factors. Instead, the parchment seems to have retained a more intact structure towards the edges.

The second question is in line with the first. What is the origin and function of the round particles in the parchment interior? Have they been introduced during the preparation of the parchment? Are they some sort of precipitant? We noted their distribution together with disentangled collagen fibers, occasionally caught in between individual fibers. The latter may therefore suggest a relation between these particles and the process of collagen disentanglement.

Further research is required to answer the questions above. In the first place, this will involve elemental and structural micro-analysis, including micro-XRD, micro-XRF and potentially micro-XANES. Such experiments will reveal the chemical composition and hopefully

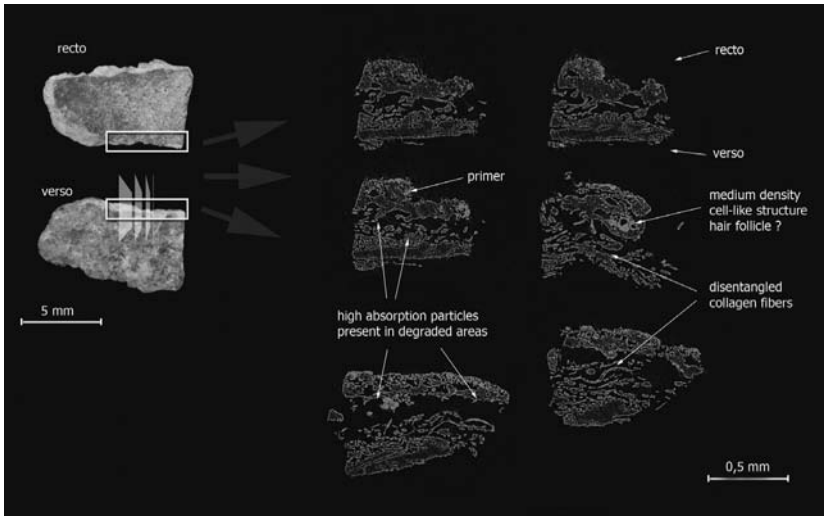


Figure 6. On the left stereomicroscopic images of the Dead Sea scroll fragment photographed on both sides, showing a reddish primer on the recto side. The white frame indicates the area that was imaged with micro-CT. On the right several reconstructed slices. Their plane of view is indicated in transparence on the left. (Color photograph in Figure IV)

the origin of the unidentified globules inside the parchment. Distribution images should be obtained from cross-section, including main and trace elemental components. This will provide information on the interaction between the organic tissue and the mineral components of the parchment. Second, we would like to examine freshly prepared parchment and compare this with degraded sample above.

These additional experiments will still require the preparation of a conventional cross-section through our sample. The added value of micro CT, however, is that we have been able to check our sample non-destructively beforehand for interesting features. Because we have examined a three-dimensional volume, rather than a single 2d section, we have gained a better and more representative understanding of the parchment and its characteristic features. This allows for a more focused second step in this analysis.

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CHAPTER THREE

GENDER AND QUMRAN

Katharina GALOR
École biblique et archéologique française de Jérusalem
and Brown University

Abstract. In this paper a summary is offered to provide the current state of affair in the research of the female presence at the Qumran site, by comparing it to other sites in the Dead Sea area and further inland.

Keywords. Gender, female presence, Qumran, combs, hairnet, perfume.

The study of gender, a popular topic today across academic and non-academic fora, has clearly been addressed previously in the context of Qumran studies (Elder 1994; Schuller 1999; Gruber 2001; Magness 2002; Crawford 2003; Wright 2004). With its potential to clarify the issue of the site's inhabitants and in consequence to carry a sufficient amount of ammunition to either defend or attack the traditional "de Vaux" theory, the topic has been recognized for its importance. The dilemma of deciding in favor of the original suggestion put forth by the excavator, still vigorously maintained by some archaeologists, or instead of adopting the alternative view, still haunts the scholarly community. According to the traditional view the building complex at Qumran was used by a primarily celibate community establishing thus a definitive link with the caves and the sectarian scrolls. According to the alternate, the character of the site—and therefore its inhabitants—mirrors the material culture and population of the larger region. Though, no consensus has been reached among those who reject the traditional view, women in those alternative interpretations are not usually relegated to the site's margins, if the issue of gender is being explicitly addressed.

I tried to summarize the topic of gender and Qumran here above, in a very simplistic way, reducing a truly complex issue to a very basic model. In reality, the problem is much more complicated. To appreciate the current stand of scholarship and the question of the female

presence at Qumran, one should keep in mind that the field of gender studies in archaeology generally speaking has been lagging behind all other fields in the humanities (C. Meyers 2003: 185).

Perhaps even more surprisingly, the area of gender studies in Syro-Palestinian archaeology has emerged much later than in other regions of both the new and the old world, in pre-historic as well as in historic contexts. Carol Meyers, one of the main pioneers in "engendering Syro-Palestinian archaeology" has stated in this context that: "…the agenda of our digs [in Palestine as a whole] has been set by the agenda of the texts" (Meyers 2003: 187). Just like the region as a whole has been long overshadowed by the literary accounts—primarily the narrative of the bible and the gospels—so the site of Qumran has been viewed as a mere reflection of the manuscripts uncovered in the Dead Sea region. The unique and extraordinarily valuable discovery of the scrolls, however, has not always constituted an advantage with regard to a scientific and objective interpretation of the archaeological remains. It may not come as a surprise, that most published works dealing with gender and Qumran address the written evidence, for the most part the scrolls, but also contemporary texts, such as Philo and Josephus. Unfortunately, other than the cemetery, no aspect of the site's material evidence elucidating matters of gender has been submitted to a rigorous study.

Let me only briefly summarize the data we have with regard to the cemetery, in particular aspects that relate directly to matters of gender. After the only cursorily published observations by de Vaux (1953: 95-106; 1961: 37-39, 46-47; 1973: 45-48, 57-58) and about a decade later those by Steckoll (1968; 1969) the subject did not undergo any rigorous analysis for several decades and many of the early claims were uncritically adopted by subsequent scholars interested in the archaeology of Qumran. Only recently, the long dormant anthropological material, underwent scientific evaluation, the so-called "Kurth-collection" by Olav Röhrer-Ertl (2006; and Rohrhirsch and Röhrer-Ertl 2001) and the so-called "Vallois-collection" by Susan Sheridan (2002; as well as Sheridan, Ullinger and Ramp 2004). In addition, several recently conducted surveys of the cemetery have led to a more refined documentation of the locations, distribution and orientation of the still visible graves (I. Vatkin in Hirschfeld 2004: 159; J. Rosenberg and E. Meyers in Sheridan, Ullinger and Ramp 2003: 135; and Eshel, Broshi, Freund and Schultz 2002: 138). The renewed scholarly activity has resulted in several excellent studies that have reevaluated the

funerary evidence from the site of Qumran, both from an archaeological and an anthropological perspective and they appear to come to very similar conclusions (Zangenberg 1999; 2000; Taylor 1999; Norton 2003; and Avni 2008).

Funerary objects that have sparked much interest and speculation are a number of jewelry pieces found in Tomb 1 and Tomb 33 (figs. 7 and 8). Christa Clamer (2003: 171-183), in the second volume of the final report series of the École biblique has shown that the ring, earrings and beads of those tombs are clearly not Bedouin or of recent date (that is ca. 100-200 years old as suggested by anthropologist Joe Zias, 2000: 225-234) but rather of the Roman-Byzantine era. Comparative studies with jewelry from roughly contemporary contexts (Galor forthcoming), however, have convinced me that nothing indicates that the jewelry documented is necessarily later in date than Early Roman. Rings and earrings of this type are common adornments made and worn during both the Roman and Byzantine periods and have not changed sufficiently from a typological and technological point of view that allow to differentiate between early and late Roman and Byzantine eras. The range in date that the bone and glass beads would fit into is even larger. In many cases, it is impossible to differentiate between Bronze Age, Hellenistic, Roman-Byzantine and Early Islamic beads. Unfortunately, no pottery that would allow us to narrow down the dates was found in association with the Qumran jewelry pieces.

Given the statistically minimal amount of professionally excavated burials—only about 5 % out of the total of 1187 tombs—as well as the terrible state of conservation of the available skeletons, all statements made with regard to the cemetery—no matter how rigorous and sophisticated the study—have to be taken for what they are: conclusions based on poorly preserved remains that only constitute a minor test group of the total cemetery. The negligible amount of burial items renders the data hardly less ambiguous.

What we can say with certainty, however, based on the evidence that was available for study is that: The Qumran tombs and cemetery are similar to many others known in the Dead Sea area as well as the region of Palestine as a whole and beyond.

All excavated burial contents indicate a late Hellenistic and Roman date for the tombs and appear to be largely contemporary with the nearby settlement.

The funerary remains do not allow for any kind of conclusions with regard to the interred religious or ethnic identity (Zangenberg 1999; 2000: 51, 66-72; Avni forthcoming); with certainty no specific sectarian distinctiveness can be inferred from the tombs and the associated burial items.

More specifically with regard to the issue of gender, the following statements can be made: The jewelry constitutes the only burial items that are of a gendered nature and can be clearly associated with the female sex. Burials of women and children are present in the main part as well as in the extensions of the cemetery.

The ratio between male and female burials at Qumran is 63.5 % versus 23 %, the remaining 6 % being child burials (Norton 2003: 123), a proportion that can also be observed at other sites in the region.

Wooden combs form part of past and current Dead Sea Scroll and Qumran traveling exhibitions and associated catalogues (Ariel et al. 2007: 124). Important here, is to mention that no combs were found at the site of Qumran itself. Combs featured in the catalogue are from Wadi Murabat and other caves in the area. The fact that no combs were found at this site does not imply that the inhabitants of the building complex did not use any. Rather, the climatic conditions of the site are different from the ones in the caves and do hardly enable the preservation of any kind of organic materials. This said, combs do not elucidate any matters of gender as both archaeological and literary evidence suggest that combs were used by both male and female (Whitehouse 2000: 748).

More significant in this respect are two types of objects found in the nearby caves, both of which have only been rarely mentioned in the literature, by traditionalists and—perhaps more surprisingly so—by non traditionalists alike. One is a hairnet and the other several tunic fragments with a gamma-shaped pattern.

The earliest representations of hairnets feature on pre-historic figurines (Berman 1999). Among the more priced pieces from the Roman period are hairnets made of finely woven gold wires such as the one depicted on a Pompeian fresco portrait of a girl often mistakenly labeled "*Sappho*" (Sampaolo 1992: 104).

The Qumran piece is far more modest than the example from Rome, being made of linen, and clearly represents the more common type regionally as well as in other areas of the Mediterranean. New Testament and Talmudic passages support the iconographic and archaeological evidence indicating that hairnets were primarily worn

by women (Gospel of Thomas 41:24; Babylonian Talmud: Shabbath 57 and 64).

Equally overlooked just like this hairnet, are several tunic or mantle fragments one of which was found in the Christmas cave, which are decorated with a so-called "gamma-shaped pattern." (One piece was shown in a paper presented by Orit Shamir at various conferences and most recently at the last ASOR meeting in November 2007.)

As opposed to the tunics worn by the Romans, which were mostly woven in a single piece, tunics worn by Jews were usually woven in two pieces and joined at the shoulders, leaving an opening for the neck. Lucile Roussin suggests that the Hebrew word for tunic, *haluq*, may be derived from this method of weaving the garment in two pieces (*heleq* in Hebrew) (2003: 183). Stripes of various widths, dependent on the individual's social rank would vertically descend from the shoulders. The stripes, called *clavi* in Latin, were usually woven in shades of purple.

As indicated by the famous Doura Europos wall paintings, tunics decorated with notched bands were worn by men, whereas the ones worn by women were decorated with gamma-shaped patterns (Roussin 2003: 184).

Fragments with both kinds of symbols were found within the Cave of Letters, dating to the second century CE (Yadin 1971: 72 and 76). The gender attribution of the different symbols appearing on the textiles of the Bar Kokhba caves was originally suggested by Yigal Yadin (1971: 69-79) and more recently confirmed by Lucille Roussin (2001: 185-187) who conducted a more in depth analysis devoted to the question of garments worn by Jews.

The sole piece of textile found within the building complex of Qumran itself, and that miraculously has survived the times and climate was found in locus 96 (Bélis 2003: 251-259; Müller et al. 2003: 283, 286; figure 9). Though heavily carbonized, some have pointed out its potential association with the Essene garment which Josephus tells us was white (War II, VII, Sn 5). However, as Roussin has pointed out, in addition to the evidence in Siffre Deuteronomy 115b and various passages in the Talmud (BT Mo'ed Katan 23a; BT Nidda 61b; BT Ketuboth 71a; BT Pesah 109a) the general differentiation between men's white garments and women's colored garments is borne out by the clothing depicted in the paintings of the synagogue of Dura-Europos (Roussin 2003: 186). Though it is impossible to determine the original color of the piece of textile found in locus 96, even if it were

white, it would hardly imply that it was worn by an Essene rather than a random individual.

Spindle whorls are among the most ubiquitous finds linked with the female gender. Though this may not be true for all societies of the new and old world, Carol Meyers (1991: 147) and Miriam Peskowitz (1997a: 24; 1997b: 105-120) have shown that the activity of spinning and weaving in Palestine should be associated with women going back as far as the Bronze and Iron Ages.

The wooden spindle whorls from Wadi Murabaat constitute a rare testimony of wooden whorls (Ariel et al. 2007: 121). The majority of Roman period spindle whorls excavated in Palestine were made of stone, bone or glass. De Vaux has recorded two spindle whorls uncovered at the site at Qumran (Humbert & Chambon 1994: 294 and 299): locus 7 and locus 20. One additional whorl was uncovered at Ain Feshkha in locus 18 (Humbert & Chambon 1994: 359). This is by no means an unusual number if compared to contemporary sites in the region.

Finally, a last category of find I would like to point out that has a potential value for identifying the presence of a certain gender is the one of unguent vessel. Two groups of vessels that are usually associated with oils and perfumes have been found in significant numbers at the site of Qumran: one group consists of glass unguentaria (Donceel 1999/2000: 17, figs. 5, 6, 18 and 20) and the other of clay juglets, primarily of the globular type. Biblical references suggest that perfumes containing essences of several different spices (myrrh, aloe, cassia, and cinnamon) were used for the body (Isa. 3:24; Song 4:10), on clothing (Ps. 45:9) and on bedding (Prov. 7:17). As opposed to modern perfumes, which are solutions of alcohol and distilled essence, perfumes in antiquity consisted of essence in solutions of oil.

In the main, the use of perfumes was a female prerogative. As stated by Susan Stewart (2004: 64) "perfumes [during the Roman period] could be used to great effect by those who wanted to convey messages about gender, and perhaps underline gender difference." Achilles Tatius, a novelist writing in Greek around 300 CE, remarks in a derogative context that: "her beauty is all perfume or hair dye or potions" (Ach.Tat., Leuc. and Cleit. 2.38.2-3). Nevertheless, the use of cosmetics and perfume was considered a normal practice, despite various objections to these products expressed in some of the surviving literary sources.

An active balsam production in the Dead Sea area and its use as a highly prized perfume not only in the region but even beyond is attested by several ancient authors (Patrich 2006: 241). Martial mentions (Stewart 2007: 96) that balsam was a perfume that was also suitable for male use. Its connection with Qumran specifically and more particularly with the globular ceramic vessel has been pointed out by several: Donceel-Voûte (1994: 32-33), Patrich and Arubas (1989), Hirschfeld (2004: 138). In New Testament and Talmudic literature perfume is usually associated with the female gender (John 11:2; B. Ketubot 71b; Y. Ketubot 5:13, 30b and 30c; Baskin 2002: 66-67). If there is some evidence that the globular juglets at Qumran may have been used by men as well, there are numerous other types of unguent bottles that tend to be more readily associated with the female gender.

Rather than claiming to have solved the problem of gender at Qumran, I hope that this paper demonstrates that there are a number of finds that lend themselves to a more thorough investigation of gender specific material remains. The standard conclusion for most paper's dealing with the archaeology of Qumran is to state that as long as the publication process is not finalized, no definitive statements can be made. I will refrain from using this "jargon" as it is highly problematic in two ways. First, the publication of the final report as relevant to most finds of the de Vaux excavations, have been published (Humbert/Chambon (1994); Humbert/Gunneweg: (2003). Second if we are interested in dealing with archaeological remains we have to deal with the deficiencies that this field brings along/is characterized by. The remains are partial and incomplete, and thus quantifications problematic. The absence of certain finds can not be considered as proof that something didn't exist. In other words, let us deal with what we have in terms of gendered artifacts from Qumran and surroundings. Let us state simply and briefly: nothing allows us to differentiate Qumran from other contemporary sites in the region, at least nothing that touches upon gender.

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Figure 7. Jewelry from Tomb 33 (KhQ 2665). Courtesy of École biblique et archéologique française

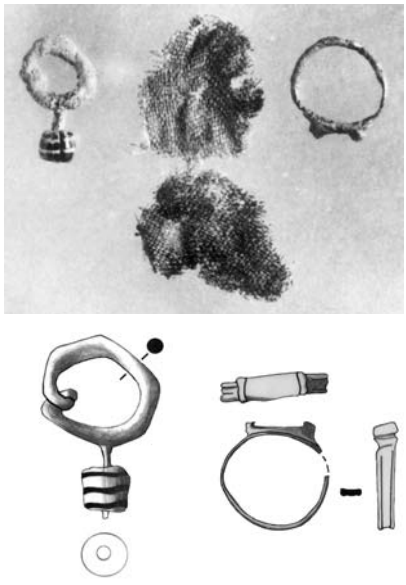


Figure 8. Jewelry from Tomb 1 (KhQ 3651). Courtesy of École biblique et archéologique française

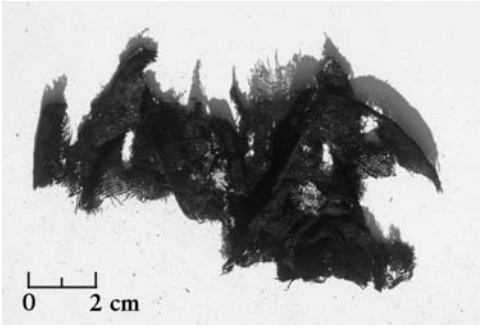


Figure 9. Textile fragment from Khirbet Qumran, locus 96. Courtesy of École biblique et archéologique

CHAPTER FOUR

WAS THE QUMRAN SETTLEMENT A MERE POTTERY PRODUCTION CENTER? WHAT INSTRUMENTAL NEUTRON ACTIVATION REVEALED

Jan GUNNEWEG[a] and Marta BALLA[b]
[a]Archaeometry, Institute of Archaeology, The Hebrew University of Jerusalem, 91905 Jerusalem, Israel; [b]Nuclear Reactor, University of Technology and Economics, 1520 Budapest, Hungary

Abstract. New data will be reported regarding the possibility that the flood deposits accumulated in three of the cisterns at Qumran could have been used to produce pottery, i.e. local Qumran pottery. A pottery waster, found in the ashes east of the Qumran kiln provided the ultimate chemical fingerprint of locally made pottery for which we were searching since 1998. The local chemical fingerprint of Qumran will also serve to distinguish between pots of Qumran and those from Engedi, Masada as well as those that are an integral part of the many (+/- 100) animal bone piles that have been found on Qumran's southern plateau.

Keywords. Neutron Activation, Pottery Provenance, Qumran, Marl/Clay, Animal Bone Heaps, Dead Sea scrolls

Introduction

In 1998, the present authors started a project to investigate where Qumran pottery was manufactured. The technique that was employed was Instrumental Neutron Activation Analysis (hence INAA), which has proved to provide the most objective results regarding identification of the chemical compositions of various potteries. According to the style and the size of the pottery that had been unearthed at Qumran in the 1950ies, it was anticipated that more than one site had produced the pottery that was found in the settlement of Qumran. But, as often occurs in the origin of ceramic vessels, it does not suffice anymore to launch the theory; one has to prove it too.

The rumor had been that Qumran has no clay of its own, which had, therefore, to be imported from elsewhere. This concept of importing

clays is not unique, because in the course of 35 year of INAA research, we have sometimes detected that clay was indeed imported since it was lacking at a specific site. A good example is Black-Red Topped Egyptian ware that was a mixture of two clays, Nile Mud—found everywhere in Egypt alongside the River Nile--and clay that came from the city of Ballas. Also at present, the al-Fakhuri pottery manufacturer in Hebron imports its clay from Beit 'Ummar, a Motza clay formation type of clay, which is then mixed with an even quantity of red hamra clay dug at Hebron itself to which 20% sand is added.

In 1998, one of us sampled mud that is abundant along and in the Dead Sea, mud that might have served the Qumranites to manufacture pottery, in spite of the fact that every archaeologist told us that one could not make a ceramic from Dead Sea mud. After levigation, a small pot was modeled, an inkwell in imitation of the ones found at Qumran, and after firing it, the Dead Sea mud turned into ceramic.

In the same year, also a thin upper layer of marl from a rain puddle was collected from the Qumran plateau itself. The marl was levigated and again modeled into a small inkwell that not only became ceramic but even looked in color highly similar to Qumran pottery. This marl came, of course, from a deposited layer on top of the plateau brought by torrential floods from the Judaean hills that took this deposit for 20-25 kilometers to the Dead Sea by Wadi Qumran and by falls that once in 2 or 3 years come to life and flood the entire Qumran plateau. At present, visible remains of waterfalls and flooding can be seen as gray strokes against a buff-colored cliff above the settlement of Qumran.

By now, we had two ink wells that looked as Qumran pottery, but to prove that one or both of them was the chemical fingerprint of local Qumran pottery, one had to subject both samples to INAA which was done at the nuclear reactor in Budapest's University of Technology and Economics.

At the same occasion, we collected fragments of bricks, sampled clay balls of 1.5 cm. diameter, and two pottery wasters, stoppers of jars and fragments of different ovens and inner linings of kilns; in short all materials that had been made in Qumran and had never left the site. All this was called the Qumran Reference, except for the wasters that did not analyze as anything on our databases.

INAA provided us with the information that the Dead Sea mud did not match the Qumran reference. It further proved that almost all samples of the reference analyzed alike. Moreover, the imitated just-

manufactured inkwell made of rain puddle marl matched the Reference too. This was the proof that we had found a sort of clay that the Qumranites could have used to make their ceramics. In order to prove the latter, we had to analyze pottery found at Qumran. When all this was plotted against INAA data of almost 300 fragments of analyzed pottery found at the Qumran settlement and its caves consisting of all kind of different vessels including the famous cylindrical scroll jars without handles, it became clear that we had obtained the chemical fingerprint of Qumran. This was backed up by rigorous statistical analyzes of which Principal Component Analysis [1] was the decisive statistical indicator, which was also tested by X^2-Square dissimilarity (hence X^2) and Euclidean Distance and Mahalanobis statistics (hence EuD.) as developed in the SEARCH program by Beier and Mommsen [2]. The data were added to the larger data bases; one from the Archaeometry Unit at the Hebrew University of Jerusalem and the Lawrence Berkeley data sets, as well as the Budapest database of INAA data and it was found that this Qumran fingerprint was indeed unique to the Dead Sea area and not found in any site in Cis- and Trans-Jordan, Israel and Jordania, respectively. Gunneweg and Balla published their Qumran results in 2003 [3] to which we refer here.

1. *Magen excavation*

In 2002, during the Brown University's Qumran conference, Yuval Peleg recorded his and Yitzhak Magen's renewed excavations at Qumran and made mention of a clay source that he and I. Magen had found at the bottom of cisterns 71, 56 and 58 [4]. Because the clay layers were found within the cisterns, the excavators assumed that the cisterns had been used to collect clay and concluded that Qumran had been an enormous center for making pottery that was exported to many sites everywhere. This new conclusion made news not that it brought new data, but because it was accompanied with the still unproven hypothesis that Qumran was a mere pottery production site and nothing more than that, thereby also excluding the Dead Sea scrolls that had had been found there but that had nothing to do with the site of Qumran, as was suggested.

Our request of obtaining a sample of these alluded clay deposits was repeatedly refused, whereas meanwhile a new myth was built around potters making ceramics at the Dead Sea without having any connec-

tion to either a sectarian group of people that settled there or the Dead Sea scrolls that were seen as the sectarian legacy. Important local as well as international newspapers all around the world heralded the new interpretation, but without any of them could actually prove a word of what they wrote down.

In June 2007, five full years after the Brown conference, Gunneweg got a full year fellowship at NIAS (Netherland Institute of Advanced Studies) in conjunction with the Lorentz Center in Mathematics and Physics at Leiden University. He was offered to organize a five-day long workshop, which he chose to have on "Holistic Qumran" a trans-disciplinary approach to what had been found at Qumran and in the Dead Sea scrolls. The present paper is a part of the workshop's proceedings.

It became time to lay hands on the clay deposits at Qumran and since taking samples from the Cisterns 71, depicted in figure 10, Cisterns 56 and 58 was not allowed, J.G. went one day to the other side of the road that passes Qumran and that runs perpendicular to the delta of Wadi Qumran and took a sample of the torrential flood that had been deposited there for centuries by Wadi Qumran, depicted in figure 10a. This sample, however, would prove the chemical composition of torrential rain floods but could not be used as a substitution for sampling Magen/Peleg's marl/clay from the three aforesaid cisterns.

It was anticipated that this layer should be the same layer of deposited clay that had been collected by Magen and Peleg since it came from the same direction; the Judaean Hills. In following the tradition of the last 10 years, again an inkwell (figure 10b) was modeled and fired into a ceramic. Nonetheless, the *caveat* remained that the INAA result of this inkwell would still not prove that the obtained sample was of the same composition as the clay found by Magen-Peleg in the Qumran cisterns.

Just a week before departure to the Netherlands, with the full heartedly recognized help of two of his ex-students, Yuval Peleg and Yoav Zioni, J.G. was granted permission to sample the clay deposit from the Qumran cisterns 58 and 71 (see figure 10c), whereas he also sampled a potter's waster from the kiln dump and a fragment of a sealed jar that was supposed to have contained date honey. K.L. Rasmussen would date the jar using Thermoluminescence at the University of Southern Denmark at Odense. Three ostraca were given to us as a bonus, one with an inscription made before firing that means that a Qumranite did it.

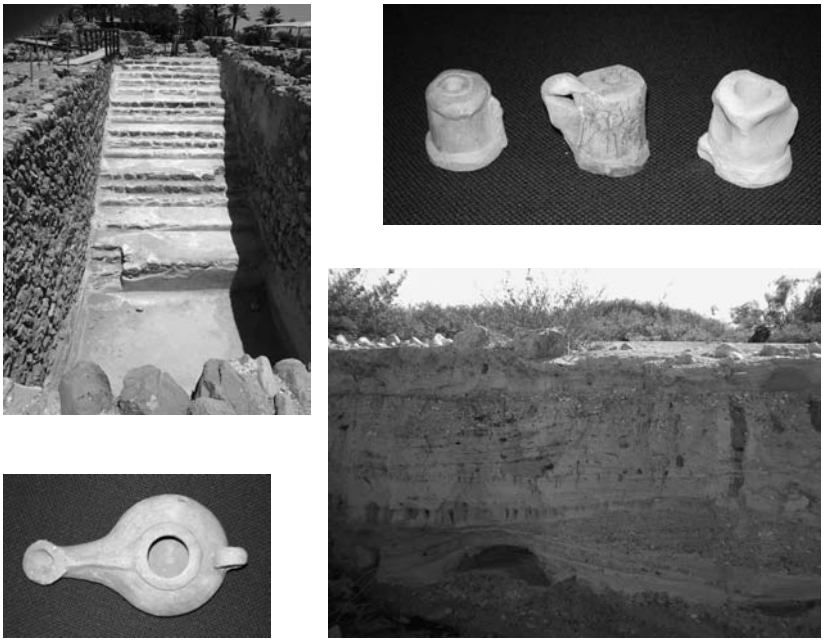


Figure 10 a-d. At the down right corner the torrential deposit of marl/clay and above it the three inkwells made of Dead Sea mud, the rain puddle and the marl/clay deposit, respectively; all subjected to INAA. In the upper left corner is depicted Cistern 71 from where marl/clay was analysed. In the down left corner is the oil lamp from the Price-Gutfeld expedition at Qumran

In the short period that was left before departure, the clay was levigated and made into plaques that then were fired above 860 degrees Celsius for 40 minutes to get rid of calcium carbonate and all organic matter. After that, powder was drilled from the clay plaques and placed into plastic vials and sent off to Marta Balla at the nuclear reactor in Budapest to have the results at hand before the start of the Workshop on Qumran planned for April 2008.

2. *Randall Price excavation*

Since 2001, Randall Price of Connecticut University and Oren Gutfeld of the Hebrew University are excavating somewhere at the center of the Southern Plateau outside the Qumran settlement (see Map 2). This

plateau ends as a spur with barely recognizable ruins of scroll caves 7-10.

In the past, INAA as well as TL was applied to Jar-35 that Randall Price had found sealed with a bowl that was removed from the jar *in situ* in J.G's presence. Within the jar, on the bottom, laid a deposit of a transparent material. The jar was sampled and analyzed by INAA. The deposit within the jar proved to have been wine. The jar itself came from Jerusalem, as INAA was able to prove, whereas the origin of the wine could not be ascertained [5]. The jar was also dated by TL, which will be dealt with in the present proceedings by Kaare L. Rasmussen.

Since then, new interesting materials were excavated consisting of a completely preserved long disk-shaped oil lamp with erected nozzle, depicted in figure 10d, generally thought to have been of the Late Hellenistic period, as well as a shard of a large jar from a large animal bone deposit and a cooking pot fragment from a smaller animal bone deposit. Price and Gutfeld told us that these were two such deposits out of a total number of about sixty that they dug west of the 'Long Wall' that runs N-S at the east of the settlement and the entire southern plateau. Both deposits were important to establish from where the pottery came among which the animal bone remains had been buried since we all are curious to be able to prove of who settled there outside of the Qumran settlement. The bones from these two burials were submitted to DNA to establish which animals have been slaughtered and Gila Kahila will describe these in chapter 5.

3. *Gideon Hadas excavation, Engedi*

There are always scholars who will tell you that they have found something that they have recognized as being from Qumran by just looking to the shape of the vessel. Such was the case with the excavator of Engedi Village at an area at the foot of a small hill, Gideon Hadas who found a house with pottery and ovens [6]. Among the pottery finds was a jar that looked to the naked eye similar to KhQ1465 QUM162 from L. 80 at the Qumran Complex, previously analyzed and published in 2003 as a local Qumran pot (Gunneweg and Balla 2003, 11/2).

The Engedi jar has three pierced ledge handles on the shoulder of the jar whereas the Qumran jar has two small loop handles. They are approximately of the same size. Both jars have a high upstanding rim.

Figure 11. Three similar jars: On the left the Qumran Jar (KhQ1465 QUM162), in the middle the jar from Engedi (Courtesy of G. Hadas) whereas on the right jar 1655/1 of the Schoeyen Collection that will be treated in a different publication (Courtesy of M. Schoeyen). All three are complete and have pierced knob-handles.

The Engedi jar has a ring base bottom, as has the Qumran jar. Finally, the Qumran jar has a straighter body than the Engedi jar has. Nevertheless, Hadas' question was; is it possible that Qumran provided Engedi with pottery of which this jar was the most important representative because it looked exactly like a jar dug at Qumran?

Moreover, a second large jar was sampled that had been found within the waters of the Dead Sea and seemed to have fallen from a boat that either arrived with the jar from the Jordanian side of the Dead Sea, or it was on its way to Moab at the other side of the Dead Sea in Jordania. INAA would be able to find the manufacture site of the jar, it was hoped. We have analyzed three vessels from Engedi, the two already mentioned and a cooking pot.

4. *Ehud Netzer's and Guy Stiebel's excavation of Masada*

The rock-castle of Masada deserves a holistic research similar to the one that we have performed on Qumran. What follows, therefore, covers a tiny piece of the cultural relics left at Masada.

In 2002, Andre Lemaire of the Sorbonne in Paris, when writing his chapter on Qumran ostraca remarked the *LXI* inscription on a wine jar at Qumran (figure 12) and interpreted it as the number 61 in Latin

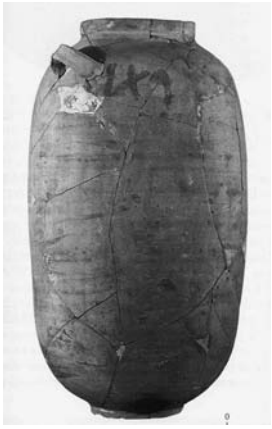


Figure 12. Qumran jar (KhQ 1401) with the LX1 inscription.

script, which, he claimed, is similar to wine jars that have been found in southern Italy [7]. INAA showed that the jar was made of the Motsa-Hebron clay [8] so that the jar was made in Israel and the inscription could have been copied from similar jars found elsewhere that perhaps had been imported from Italy.

A similar jar with a Latin number inscription was also found in Masada and we wanted to know whether this jar was also made from Hebron-Motsa clay or that this one was imported from Italy as suggested by Lemaire (Lemaire 2003, 354).

Besides this jar, we analyzed more storage jars with inscriptions, but these will have to wait until the final publication of Masada pottery.

5. *Freund's excavations at the Cave of Letters*

Mid March 2002, Richard Freund from Hartford University at Connecticut in the United States approached us to solve one of the most interesting questions he and his team had encountered in connection with the Cave of Letters. He asked: Is the content of the cave that of 130-135 AD on the basis of the letters of Bar Koziba (Bar Kochba) found in this huge cave, as originally shown by Yigael Yadin, or was the cave also earlier occupied by pre-70 AD sectarians who had fled either Jericho or Jerusalem or Qumran?

The Cave of Letters' pottery has been dug from beneath tons of fallen cave ceiling. The cave is almost impossible to be reached. It is

located halfway a steep cliff. The cave is 150 meter long with two extending arms of 60-meter depth. The climate is so constant and dry that bones, for example, have still their collagen preserved so that dating them by C14 is easy.

Freund received the answer that the neutron activation technique was not designed to provide a date to artifacts and that we would need a reference to match the chemical composition of his samples. However, we gave him to understand that if we would be able to trace the origin of the cave's pottery to either Jerusalem or Qumran, we would indirectly obtain a date since both sites were entirely destroyed by the Roman general Titus in 70 AD. Pottery in the Cave of Letters being traced to either one or both of these places would provide a date that antedates the destruction, so that some of the pottery in the Cave of Letters might be of pre-70 AD.

The Cave of Letter's sample consisted of red ware, white limestone-imitation ware, two storage jars and oven lining.

The samples were collected from Loci 1 (Hall B), 2 (Hall C), 6 (BC Passage), 8 (kiln lining), 16 (AB Passage), 29 (Hall A) and 34 that is the entrance of the Cave. The collaborator of Freund, J. Clark told us that the fifteen chosen pottery samples represented, stylistically, the bulk of pottery found in the Cave of Letters and so, we commenced [9].

6. *INAA as a nuclear technique and the modus operandi of preparing samples*

The technique used to analyze the pottery samples is Instrumental Neutron Activation Analysis (INAA). This nuclear analytical method has the capacity to determine quantitatively about 25-35 chemical elements-for the most part trace elements- that are sufficient to provide the chemical fingerprint of the vessel.

The basic idea of INAA is that irradiation by neutrons induces nuclear reactions in the nuclei of the sample and that the interaction results in reaction products, which are usually radioactive. The decay of these radioactive nuclides is accompanied by the emission of gamma quanta of characteristic energies, and the radiation can be used both to identify and accurately quantify the elements of the sample.

Experimental

Powder samples weighing approximately 50 mg are taken for the analyzes. These samples are heated in a furnace (for 1 hour at 600°C) to remove absorbed moisture and organic remains before being placed in polyethylene vials. A batch of fifteen such vials plus another containing reference material is then packed into an irradiation capsule.

Irradiations are performed in the nuclear reactor of the Institute of Nuclear Techniques (8 hours on 100kW power, 2.4 x 10^{12} $ncm^{-2}s^{-1}$ thermal neutron flux). Gamma-spectrometric measurements are performed after cooling times of 5–6 days and 25–30 days, using a HPGe (germanium) well-type detector (Canberra, FWHM: 1.95 keV, rel. efficiency: 20.5%) connected to a Canberra S100 Analyzer. Spectrum evaluation is done with the use of GENIE 2000 software.

Standardization is performed by the so-called single comparator method, using gold as the comparator element and zirconium foils as flux-monitors. To control the accuracy of the measurements, a reference, GBW 07313 Marine Sediment, is analyzed as well.

Elemental data is attached. The given uncertainties are combined standard uncertainty values of the measuring process, but do not contain the uncertainty of sampling.

List of new Qumran samples to be discussed hereunder:

Qumran
QUM 370 Oil lamp QP C2 209.4L 1062 (Price/Gutfeld).
QUM 371 Marl/Clay from Pool 71 (Magen/Peleg)
QUM 372 Upper deposit of marl/clay from Pool 58
QUM 373 Lower deposit of marl/clay from Pool 58
QUM 374 Ostracon, 1385 Locus 9027, with three incised letters in Hebrew before firing
QUM 375 Ostracon, 4025 Locus 9227, scratched letters in Hebrew
QUM 376 Wadi marl/clay inkwell
QUM 377 Jar shard from large bone deposit
QUM 378 Jar shard from small bone deposit
QUM 379 Cooking pot ware shard from small bone deposit
QUM 380 Brown substance content of Jar 2, Magen 2160
QUM 381 Two-handled Jar 2 itself. Magen 2160.
QUM 383 Magen stopper of Jar 2
QUM 386 Content of Jar 2 for NAA

QUM 387 Soil content of Jar 2 for TL and C14
QUM 388 Kiln waster from East Dump L. 9020

Masada
Masada 201 N3. 20A 449 High neck wine jar with long loop handles. Red inscription and tar at the inside of the mouth for about ten cm.
Masada 202 N3. 20 261 Inscribed shoulder of amphora
Masada 203 N3. 51 517 large body shard with inscription
Masada 204 N3. 69 616 The *LI*-inscribed wine amphora (imported?)
Masada 204 Imported(?) inscribed wine amphora
Masada 205 N3. Registration unknown. Large storage jar with four loop handles
Masada 206 N3. 51 465 Inscribed before firing jar
Masada 207 459/1516 Large pithos bottom and rim [rim analyzed]
Masada 208 G59/95 N1 Stamped handle of amphora [*ER*…..]

7. *Qumran results*

The next paragraphs concern the provenance of Qumran pottery analyzed by INAA. The X^2 and EuD values will be stated after every single sample. Both are taken with only one single outlier, or security of a single standard deviation.

QUM 370, the oil lamp of the Price-Gutfeld excavation on the mid-southern Qumran plateau was similar in composition to that of an oven cover, a platter, a decanter, pseudo-Nabataean pottery and a storage jar that all were shown to be local to Qumran (Group-I in Gunneweg and Balla 2003)[10]. This oil lamp was thus locally manufactured at Qumran for people who camped on the southern plateau at Qumran X^2 0.99, EuD .98.

The marl/clay deposit of Pool 71 (QUM371) did not analyze as any of the previously analyzed local pottery found at Qumran. One cannot, therefore, conclude that this deposit—as such—was used for making Qumran pottery as its chemical fingerprint shows. The latter can be stated with confidence, in spite of the thorough levigation of the deposited marl/clay found in Pool 71 and its subsequent firing at 860 degrees Celsius. This information goes straight against any conclusion that Magen and Peleg published in Jerusalem (Magen and Peleg 2007) X^2 .65, EuD 1.2.

The upper clay deposit found in Pool 58 (QUM372) was similar in composition as that of Pool 71, which means that both deposits had a common background, but nothing more than that. The same flood could have filled them.

The lower deposit of Pool 58 (QUM373) too, does not match any of the Qumran pottery that we have analyzed in our previous work. It matches the chemical composition of the newly made inkwell made of the marl/clay (see figure 10b) sampled from the delta of Wadi Qumran on the other side of the road that bypasses Qumran X^2 .64, EuD 1.6.

Although no pottery was made of the marl/clay deposit, all three samples (QUM371-3) show a statistical match with material that we have collected at Qumran before we started INAA project in 1998. They include the inner lining of the kiln, stucco for the plastered table, an oven cover, a clay ball and the inkwell that was made of the rain puddle. One can, therefore, state that this marl/clay is the prime potter's clay/marl but not as it was found.

QUM374, Ostracon #1385 came from the East Dump near the potter's shop. It bears three incised letters that were written on the shard before firing. This information alone is not enough to state that it is local because the shard could come from elsewhere. Nevertheless, this ostracon analyzed as the previous flood deposit samples, especially Pool 71 X^2 .69, EuD .11. This is therefore the first testimony to the final ceramic product of a local Qumran potter who also incised the pot with three letters that seem to be the only letters he used.

All this proves that the flood was homogeneous in composition and deposited everywhere being it within pools or across the entire surface of Qumran. That is then also the reason why we have stated at the start of this paper that the rain puddle chemical fingerprint was similar to local pottery and that it was there as a result from a flood that covered the Qumran surface at times.

QUM375, ostracon # 4025 found in Locus 9227 is a shard plus handle scratched with letters on the shoulder, a pot that originated in Jerusalem. It matches the wine Jar-35 (QUM359) previously proved the only pot brought to Qumran from Jerusalem (Gunneweg and Balla 2006, 106). The ostracon and Jar-35 are the only samples that matched the Motsa fingerprint as it is found in Jerusalem and that is out of a total of 280 samples that were analyzed. They therefore came from the capital. Nevertheless, a *caveat* is in its place, because both Jerusalem-made pots were found outside the Building Complex of Qumran X^2

.67, EuD .32 and have, as such, nothing to do with the Qumranites of the Building Complex itself.

QUM376, the freshly made marl/clay inkwell of August 2007 matches QUM371-4, all flood deposits, as well as the inkwell that was formed from the Qumran rain puddle, directly south of Pool 71 in 1998 and the Bedouin *Tabun* X^2 .56, EuD .44. It also matched two clay balls, a brick, two fragments of kilns and a pipe that was found in Locus 51 built into the latrine there.

QUM377, a large storage jar from the Northern bone deposit—the larger one of the two bone burials treated in the present proceedings--and excavated by Price/Gutfeld was similar to two cups, pseudo-Nabataean pottery, a jug and a scroll jar that previously were shown being made in Jericho (samples QUM171, 218, 256, 259, 274; Chemical Group III in Gunneweg and Balla 2003, 19) X^2 .50, EuD .38. The meaning of the many bone deposits will be treated elsewhere. They cannot be explained as remains of sacrifices, because it suffices to mention Flavius Josephus who stated that the Essenes did not sacrifice, but that they had rites that were purer, as he calls it.

A Jar from the small bone deposit (QUM378) is similar to an ostracon (QUM291) and a jar that is local to Qumran (QUM139) that has been found in Cave 39 and the so-called "Negev" ware sample whose origin is not certain. It could well be that the Cave 39 content and the pit of Rice/Gutfeld that has matching pottery will have repercussions when all other pottery has been published, which at this stage is not yet available.

A cooking ware shard (QUM379) also of the small bone burial deposit as QUM378 is similar to pottery that have been called Motsa-Hebron ware, since it matched clay from Beit 'Ummar i.e. fourteen scroll jars and scroll jar lids that all belong to Chemical Group II from Motsa-Hebron X^2 .42, EuD .55.

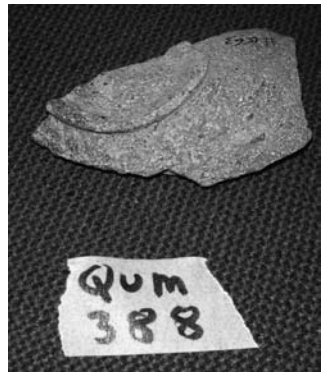
A dark substance (QUM 380) found in one of the group of jars that were sealed (QUM381) i. e. the two loop-handled Jar 2 (Magen No. 2160).

The jar under study was one of the thirteen that were found east of the dump near the potter's workshop, all with a content and sealed with a clay stopper. Previous work performed on the content of this jar at the Weizman Institute (Israel) identified the content being date honey (Magen and Peleg 2007).

Jar-2 is high in potassium, 3.9%, seldom found in local pottery of the Dead Sea area. In order to verify the date-honey connection, we

b

a



c

Figure 13 (a-c). Content (QUM380) of Jar-2 (a) and Jar-2 itself (QUM381) (b). On the right (c) the kiln waster that proved the chemical fingerprint of Qumran pottery.

might obtain a chemical analysis of dates as found at the site of Qumran and compare that with that of QUM380, the honey content of Jar-2.

It should be emphasized that the elemental chemical composition of the organic matter is low in Ca (2.2%) and high in Hf—16 ppm—the latter being 3-4 times higher than Hf in all other samples analyzed. The deposit also has a high Ce abundance—106ppm—as well as Fe of 5.1%, whereas other materials have Fe values in the range from 0.8-4.6%. We will have to continue to study this material.

Jar-2, QUM381 (figure 13 a and b), as well as its soil from within (QUM 386/7) are the only samples that have high bromine contents.

Br varies between 80-91 ppm whereas Br in all other samples varies from 8 to 35 ppm. What the higher Br content means is as yet unclear, but is logical because of the high Br in Dead Sea water and even in the air round the Dead Sea. From the INAA results on Dead Sea mud, we have learned that bromine was present as almost 2 percent, which is indeed high.

For Jar-2 itself, we have not found a match with pottery stored on our databanks. The origin of this jar remains, therefore, unknown. We have no idea if that is also true for the other jars, since we have analyzed one jar only. Nevertheless, all other jars look exactly the same as the one we have analyzed. They are also sealed, whereas Jar-2 was only chosen for analysis because it was already broken and thusready for taking a destructive sample.

If we cannot determine from where the jar came, it will be interesting to explain why these jars were filled with local date-honey (still to be analyzed) because the jars do not belong to the Qumran pottery assembly made *in situ*.

QUM383, one of the stoppers that were found with the Jar-2 type is an outlier, accentuating that the stopper came with the jar for which we have no provenience too.

QUM388 (figure 13c), the pottery waster that was discarded in Locus 9020 of Magen/Peleg in the pile of ashes to the east of the kiln complex is the most important sample, in our mind, that we have analyzed, because it matches the local composition of pottery that we have called Group-I at Qumran by X^2 .40, EuD .35. The match comprises samples such as the torpedo jar (QUM136), cup QUM136, lid QUM182, jar QUM210 and bowl QUM213 and clay ball QUM127, all local to Qumran (Gunneweg and Balla 2003, p.11).

The chemical composition of the waster QUM388 is different from two pottery wasters that we have analyzed previously and that had no chemical similarity with any of the pottery found at Qumran. From now on, we have proof that Qumran made its own pottery in kiln 84 of which the discarded ashes at the east side of the potter's workshop testify and that there are hundreds of complete vessels as well as pottery fragments that match that composition and never left the site.

That the two previous wasters had no link with the chemical composition of Qumran pottery may perhaps be explained that these were remains of pottery that collapsed in the kiln and its clay was not further used to manufacture pots for Qumran.

8. *Cave of Letters results*

The Red Wares from the Cave of Letters are chemically similar to those analyzed at Qumran and its chemical composition indicates that it could have been made in Qumran. In case the pottery was made in Qumran, it could well be from before 70 BC. There is, however, a twofold *caveat*: 1) This red ware pottery is not found in Qumran itself and 2) since local Qumran pottery was made of marl/clay, this prime constituent is present at almost every site along the Dead Sea where it was deposited by torrential floods from the Judaean Hills. Therefore, we should be very cautious to draw a conclusion concerning the relations of the population of the Cave of Letters with those of Qumran

Five Red ware body shards were chemically all similar to one another with minor variations that probably point to different potter's practices. All red ware match the general chemical pattern that is characteristic of Qumran, which as we have seen is the marl/clay deposit from torrential flooding. A stylistic research must be combined with the chemical outcome and only when they agree can the INAA results become finalized.

A cooking pot chemical composition matched that of Michniewicz's sample of clay from the Hidden Waterfall at Nahal Arugot which is near to the Cave of Letters (see the study of Jacek Michniewicz of Poznan University in Humbert and Gunneweg 2003, 51-127, esp. p. 63 sample P12/2).

Four "limestone imitation" ceramic samples are yet of unknown provenience, whereas two oven linings analyzed as Dead Sea mud. The latter does not mean that they were also made of mud from the Dead Sea, but they could have been made from the mud layers as they are found along the entire western Dead Sea area, partly the bottom of the Dead Sea and part the mud flood that came down from the cliffs.

9. *Engedi results*

Also the Engedi samples showed no connection with Qumran. Engedi 1, the jar with the three pierced knob/ledge handles did not match anything that we have on the database that comprise the Judean desert, the Judean Hills and the Dead Sea area together. This means that stylistically a certain shape of a vessel may be imitated, but to conclude from stylistic similarity alone that two pots have the same origin is

without importance if one cannot prove it. Therefore the three-ledge-handled jar did not originate in Qumran. On the other side, the form of the jar was imitated and the orinal one (which one?) must have been on display somewhere otherwise imitation is impossible.

Engedi 2, a Roman cooking pot, showed a statistical match with a vessel in Beersheba and three vessels that are "Negbite" pottery of the Iron Age II from the Negev desert. It may mean that the Engedi jar had its origin in the Negev, nearby Beersheba.

The second Engedi jar that was sampled, found on the bottom of the Dead Sea was, of course, laden with salt. As a result, INAA data should be taken with the necessary need of concern. In fact, the jar matched no other pottery on our database. Of course, it could be that it came from Jordan for which we have no chemical fingerprint. However, it could with the same security come from Israel too. The latter is a good example that one should not philosophize too much concerning provenance of pottery, if we have nothing to go on.

10. *Masada results*

Of the eight samples that have been analyzed by INAA, only two, Masada 3 and 5 show the Masada connection with its surroundings. Masada 3 matches pottery that was made in Jerusalem, Iron Age pottery also made in Jerusalem as well as a shard of Pseudo-Nabataean pottery that was established as having been made locally in Jerusalem. An additional sample is Masada 5, which matched Motsa clay found in Beit 'Ummar and two vessels from Masada that have previously been analyzed in the Jerusalem Archaeometry Unit in the late 1970s.

Whether one of the inscribed jars, especially Masada 204 had any relation with a town or district in Italy, has still to be seen. The database is under construction; a part of it will reside in the large database of the nuclear facility at Missouri University in the USA where Michel Glascock has initiated to combine all INAA data into a large database.

11. *Analytical conclusions*

The two sets of samples consisting of, on the one hand, the inkwell made in 2007 from a torrential flood marl/clay layer, the inkwell from a rain puddle outside the settlement and, and on the other hand, clay

collected from the cisterns 58 and 71 within the Qumran building complex, prove beyond doubt that they are part of the same marl/clay that covered the entire area surface because they all analyzed alike.

The main characteristic of all marl/clay plaques and inkwells is that they have a calcium abundance that varies from 26 to 33 percent, which is very high and explains the high calcium in the Judaean Hills from where the floods came. The local Qumran pottery on the other hand has an average of around 7-8 percent calcium. If one adjusts for dilution, due to the calcium that is rather free from trace elements, then the pottery can be brought into line with the flood deposits of marl/clay so that one may conclude that a certain amount of pottery is indeed locally made at Qumran.

After a long period of collecting the samples, preparing and running them, evaluating the data and going over the database, we finally saw the INAA results staring us in the face: Also the new imitation 2007-inkwell as well as Magen's marl/clay from the cisterns and the recent kiln waster had exactly the same composition as the rain puddle ink well of 1998 (QUM225) and was highly similar to the Qumran reference that we had established nine years prior to the present results.

Nevertheless, there are still other potteries found in the Qumran settlement--large groups of them--that came from either Jericho or Hebron-Motsa as we have communicated in previous studies.

Finally, we must disappoint those who wanted the outcome of the cistern clay deposit to be the Motsa/'Ummar clay of which many scroll jars have been made. The Motsa did not reach Qumran, at least not via the rain floods as decribed above.

12. *General conclusions*

1. With a selection of pilot studies in pottery provenience determination from four different sites, we are obliged to present conclusions that are based on a relative few number of samples that have been analyzed. Nevertheless, we can state with great confidence that the Qumran riddle of the past seven years has been solved. In a nutshell, the in 2003 determined locally made pottery at Qumran itself is hereby proved to be so, because of the analyzed pottery waster, QUM388 in this paper, from the kiln area that has a highly similar composition as our previous chemical Group I, the local fingerprint of Qumran.

2. The rain puddle, our homemade inkwell QUM225, proved that torrential floods covered at times the Qumran plateau and filled unprotected cisterns. It also proves that the cisterns, at least cisterns 58 and 71 were never cleaned, but new water covered the deposits with marl/clay found at their bottom. Cistern 58 is a good example of the latter; because two separated deposits of the same clay were found, called the upper and the lower deposits, QUM372&373 respectively.

3. Our home-made inkwell made in 2007 of the marl/clay deposit on the other side of the road that passes Qumran, proved that it is the same in composition as the clay collected from cisterns 71 and 58 and can even today be used to make pottery in contradiction to all those who have repeated for 50 years that Qumran did not have potter's clay. On the larger vessels, we have detected a thin layer that might be a kind of wash [11] that possibly could have strengthened a jar in order to make up for the lesser quality of the marl/clay that was used to form the body of a jar or large bowl. It could, however, also be the result of certain materials that came from within the clay and settled at the outer surface during the firing process in the kiln. This might be an interesting study for the future, some of which we have already proposed in a poster presented at the Biannual Archaeometry Meeting at Mexico City in 2000 (Gunneweg and Balla, 2000 abstract).

4. Although there is no direct statistical match between the marl/clay and Qumran Group I, there are certain types of pottery that were unique to Qumran and are similar to the fingerprint of local Group I. Such are the torpedo shaped jar and the funnel-shaped jar, as well as Qumran reference materials of various sorts as stoppers, clay balls, inner lining of kiln and a Bedouin oven (*tabun*) that was dated to the 19th century. The latter proves that even the Bedouin used the marl/clay at Qumran to produce their ceramics locally.

5. The two marl/clay deposits in pool 58 at different levels show that this pool was twice flooded by flood debris, having the same composition that originated somewhere in the Judaean Hills.

6. An additional extremely important conclusion is that the so-called Motsa-Hebron clay fingerprint is not found among all the new clay samples that were collected from cisterns 58 and 71, from the flood deposit at the foot of Qumran in the Delta of Wadi Qumran and, last but not least, the rain-puddle marl/clay that was collected from Qumran's surface. The latter does not infer that the Qumranites were collecting clay from rain puddles. It means that there has to be a spot where the same marl/clay as that of the rain puddle was deposited,

perhaps for millennia. The deposition might have changed the calcium content from 30% to about 7%, which cannot be answered so far with our research.

7. Jar-2 that was filled with a substance that might have been date-honey and that was found in the context of 12 identical jars that were sealed too, has an unknown origin. It does not analyze as local Qumran ware or any of the pottery that was traced to either Jericho, Jerusalem or Hebron. The sample of the content is in the hands of a botanist in Delft University.

8. What concerns the three-handled jar found in the small village near kibbutz Engedi, that is the jar that was similar in shape to the Qumran jar KhQ 1465 it has nothing else in common but its shape. Thus, not every pot that looks alike–Engedi proves it —has the same origin. The ancient Engedi inhabitants brought it by themselves or others in from the Northern Negev. Therefore, Engedi cannot be brought in connection with Qumran based on that specific vessel. On the other hand, it also proves that Qumran did not export its pottery to Engedi in spite of the proximity of the two sites. Of course, more pottery should be analyzed to substantiate this statement, but it was true for the imitated Engedi jar that was thought to be the finest candidate to prove a relation between the two sites.

Finally, previous pottery wasters that have been analyzed by us in the late 1990s did not analyze as pottery vessels found at Qumran. This can mean that the clay was insufficiently good to make ceramic or that the two wasters belong to a previous period, when other pottery was made, which we have not analyzed so far. The new waster, QUM 388, however, analyzes as the local pottery from Qumran.

The local fingerprint of Qumran proved by our previous work as well as by further analyzes of pottery and clays obtained from Magen-Peleg is not found in any pottery outside the Qumran settlement. The latter means that there was no export of pottery to elsewhere and that the estimated 5000 ceramic vessels were meant to be for local use in Qumran itself. The latter may emphasize the fact that many vessels were needed because of purity practices at Qumran. The latter brings us back to the original thought of de Vaux that Jewish puritans inhabited the site.

If, on the other hand, the same chemical fingerprint of Qumran will in the future be found somewhere else, it must date to the Hasmonean period, which still is not contradictory to our thought that the real sectarian Qumranites inhabited Qumran from the late forties BC until

the late fifties AD, in all a mere 90 years. This seems to be corroborated by what can be learned from the biblical scrolls found at Qumran, which are of the Herodian period, and also unique to Qumran exclusively because other bible manuscripts, for example those found at Masada, are of the type as preserved in the Masoretic tradition.

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Appendix

List of individual elemental abundances for the Qumran samples. Concentrations are in ppm if not stated otherwise.

	1	2	3	4	5	6	7	8
	QUM 370	QUM 371	QUM 372	QUM 373	QUM 374	QUM 375	QUM 376	QUM 377
As	7.4+–0.3	13.0+–0.5	12.2+–0.4	11.6+–0.4	12.6+–0.4	7.4+–0.3	11.8+–0.4	8.8+–0.4
Ba	333+–50	330+–40	360+–50	460+–40	312+–40		530+–40	320+–50
Ca%	15.66+–0.91	33.20+–1.8	33.08+–1.78	28.31+–1.44	31.31+–1.60	6.24+–0.44	26.17+–1.41	9.54+–0.56
Ce	66.5+–2.4	53.6+–2.3	49.7+–1.8	39.0+–1.4	52.5+–1.9	66.6+–2.4	43.5+–1.6	85.2+–3.1
Co	12.8+–0.5	10.6+–0.4	9.46+–0.40	8.92+–0.32	10.8+–0.5	12.1+–0.5	10.6+–0.4	15.9+–0.7
Cr	137+–6	230+–8	242+–9	160+–6	230+–7	112+–5	174+–6	170+–6
Cs	1.52+–0.19	2.01+–0.15	1.66+–0.22	1.85+–0.14		5.55+–0.32	1.48+–0.20	2.80+–0.32
Eu	1.58+–0.08	0.97+–0.06	0.88+–0.08	0.77+–0.05	1.12+–0.08	1.41+–0.10	0.78+–0.06	1.84+–0.11
Fe%	3.97+–0.13	2.90+–0.09	2.74+–0.10	2.19+–0.07	2.80+–0.09	4.58+–0.15	2.64+–0.09	5.35+–0.17
Hf	7.94+–0.40	3.95+–0.23	3.58+–0.24	5.25+–0.26	4.86+–0.24	5.48+–0.32	5.50+–0.28	9.15+–0.37
K%	1.78+–0.12	0.96+–0.07	0.93+–0.09	0.81+–0.07	0.78+–0.09	2.10+–0.12	0.85+–0.10	1.22+–0.13
La	31.7+–0.9	25.0+–0.7	24.0+–0.6	19.1+–0.5	24.6+–0.7	27.1+–0.7	21.7+–0.6	38.0+–1.0
Lu	0.38+–0.02	0.28+–0.01	0.29+–0.02	0.21+–0.01	0.36+–0.02	0.38+–0.02	0.30+–0.01	0.45+–0.02
Na%	1.16+–0.04	0.33+–0.01	0.50+–0.02	0.26+–0.01	0.72+–0.02	0.60+–0.02	0.29+–0.01	0.72+–0.02
Nd	26+–4	23+–3		16+–2	31+–13	30+–5	22+–3	40+–7
Rb				30+–5		100+–12		70+–10
Sb	0.68+–0.04	1.22+–0.05	1.29+–0.05	0.91+–0.04	1.23+–0.04	0.40+–0.02	2.64+–0.08	0.89+–0.04
Sc	12.8+–0.3	9.61+–0.23	9.25+–0.22	7.38+–0.18	9.57+–0.23	18.6+–0.5	8.19+–0.20	16.6+–0.4
Sm	6.86+–0.2	5.33+–0.17	5.06+–0.16	4.03+–0.14	5.38+–0.17	6.43+–0.20	4.62+–0.15	8.09+–0.26
Ta	1.51+–0.14	1.03+–0.33	0.70+–0.11		1.07+–0.12	1.09+–0.14	0.82+–0.11	1.64+–0.18
Tb	1.00+–0.15	0.86+–0.16		0.54+–0.08	0.62+–0.10	0.79+–0.16	0.79+–0.11	1.35+–0.18
Th	6.80+–0.33	5.82+–0.28	5.16+–0.25	4.58+–0.26	5.53+–0.23	8.20+–0.30	4.96+–0.24	9.12+–0.37
U	3.90+–0.21	9.68+–0.35	11.5+–0.5	7.60+–0.31	11.7+–0.4	2.88+–0.15	8.64+–0.35	4.27+–0.23
Yb	2.83+–0.10	2.19+–0.21	2.28+–0.10	1.75+–0.09	2.70+–0.10	3.05+–0.11	2.42+–0.10	3.52+–0.54
Zn	146+–10	240+–13	260+–15	167+–9	230+–13	136+–9	240+–13	265+–16
Br	39+–2	17+–1	35+–2	8+–1	29+–1	19+–1	21+–1	26+–2
Se			6.1+–1.0					

	9	10	11	12	13	14	15	16
	QUM 378	QUM 379	QUM 380	QUM 381	QUM 383	QUM 386	QUM 387	QUM 388
As	4.7+–0.2	5.1+–0.3	7.6+–0.3	9.3+–0.4	4.0+–0.2	5.1+–0.3	4+–0.2	7.7+–0.4
Ba		182+–43	490+–50			310+–30	335+–34	178+–30
Ca%	4.09+–0.29	6.02+–0.43	2.17+–0.32	11.2+–0.7	35.1+–5.5	23.6+–1.2	21.3+–1.1	7.42+–0.48
Ce	47.8+–1.5	66.7+–2.4	103+–3	47.5+–1.7	10.6+–0.6	28.3+–1.0	21.9+–0.8	66.2+–2.1
Co	7.20+–0.30	20.8+–0.7	22.3+–0.8	9.08+–0.4	2.27+–0.15	4.99+–0.21	4.53+–0.19	11.1+–0.4
Cr	73.3+–3.1	107+–5	133+–5	116+–5	24.6+–1.7	76.7+–2.8	62.0+–2.6	116+–4
Cs	3.16+–0.24	7.35+–0.36	1.76+–0.17	5.17+–0.35	1.10+–0.10	1.0+–0.1	1.57+–0.12	4.97+–0.30
Eu	1.06+–0.07	1.36+–0.08	1.67+–0.16	0.98+–0.08	0.26+–0.04	0.51+–0.04	0.49+–0.04	1.41+–0.07
Fe%	2.66+–0.08	4.63+–0.15	5.08+–0.16	4.34+–0.14	0.82+–0.04	1.29+–0.05	1.30+–0.04	4.59+–0.15
Hf	3.97+–0.23	4.39+–0.30	16.0+–0.6	3.54+–0.27	0.71+–0.09	3.18+–0.16	2.06+–0.14	5.68+–0.28
K%	1.26+–0.11	2.84+–0.19		3.87+–0.26	0.54+–0.09	0.91+–0.13	0.85+–0.14	1.94+–0.20
La	19.4+–0.5	27.8+–0.8	35.4+–1.0	20.2+–0.5	4.90+–0.16	12.6+–0.3	9.58+–0.26	27.8+–0.8
Lu	0.26+–0.01	0.38+–0.02	0.48+–0.03	0.25+–0.01	0.07+–0.02	0.21+–0.01	0.15+–0.08	0.40+–0.02
Na%	0.46+–0.01	0.56+–0.02	1.02+–0.03	1.05+–0.03	0.15+–0.01	0.61+–0.02	0.66+–0.02	0.45+–0.01
Nd	26+–5	29+–5	36+–7	25+–5			16+–2	33+–4
Rb	48+–8	103+–12	37+–8	99+–12			24.8+–4.2	80+–10
Sb	2.59+–0.08	0.56+–0.03	0.73+–0.03	0.44+–0.03	0.2+–0.01	0.48+–0.03	0.35+–0.02	0.60+–0.03
Sc	11.4+–0.3	19.3+–0.5	15.0+–0.4	17.5+–0.4	2.98+–0.07	5.48+–0.13	5.40+–0.13	18.9+–0.5
Sm	4.73+–0.15	6.35+–0.20	7.96+–0.25	4.92+–0.16	1.15+–0.04	2.83+–0.10	2.24+–0.07	6.60+–0.21
Ta	0.61+–0.09	0.91+–0.11	1.93+–0.17	1.09+–0.12				1.03+–0.12
Tb	0.67+–0.12	0.81+–0.17	1.21+–0.17			0.41+–0.09		0.71+–0.13
Th	5.35+–0.22	8.08+–0.33	10.4+–0.4	6.93+–0.29	1.36+–0.10	2.65+–0.13	2.61+–0.13	8.33+–0.34
U	2.27+–0.12	2.88+–0.23	2.94+–0.18	2.54+–0.22	1.32+–0.12	5.10+–0.21	3.88+–0.18	3.59+–0.19
Yb	1.80+–0.07	3.14+–0.13	3.77+–0.14	2.02+–0.12	0.48+–0.03	1.62+–0.08	1.04+–0.06	3.02+–0.11
Zn	52+–6	136+–9	105+–7	51+–6	22+–2	52+–9	59+–4	79+–8
Br	17+–2	19+–2	19+–2	80+–3	22+–2	80+–3	91+–3	16+–1
Se				15.5+–1.5		7.8+–0.7		

CHAPTER FIVE

INTRODUCTION TO THE "BURIED BONES"

Jan GUNNEWEG

Abstract. Hundred piles of animal bones were found at the Qumran settlement and its plateau. All were found with intact (some) or destroyed pottery (majority). Were these piles, typical for Qumran alone, remains of meals, of sacrifices or did they serve another purpose? INAA, TL, DNA and C14 will try to give an answer.

Keywords. Neutron activation, Thermoluminescense dating, DNA, Radio Carbon dating

Buried bones have been found since the time that de Vaux started to excavate in the 1950s. He even marked every single one on the map of Qumran. Except for the markings, however, the relics were lost being it ceramic or animal bones. The first dialogue on 60 deposits of animal bones buried within pots or beneath pottery shards took place on June 24, 200. Randall Price wanted us to analyze everything that had a connection with the 60 bone burials he and his team unearthed. This, however, means a major project. So I proposed a pilot study instead that consisted of the following. We would take bones from two such piles to be analyzed by DNA and dated by radiocarbon dating, whereas the pottery shards of the bone burials would be submitted to INAA to learn where they came from and dated by TL to corroborate the C14 date of the bones. We were curious to know who were the people who left these relics in pots, which they either brought with them or that they were given at Qumran.

Secondly, DNA should be performed to the various kinds of animal bones, whether relics of sheep, ibex, goat or cow. Moreover, we wanted to get a date of the bones by radiocarbon dating them as well as a corroboration of the date by analyzing the potshards by Thermoluminescence to corroborate C14 dates of the organic material found within the context of the broken pots (figure 14 a,b).

Two of the aforesaid deposits of Price/Gutfeld have been excavated on the southern plateau of the Qumran Complex, the majority of

Figure 14. At the left, the small burial bone pile as found and packed in plastic for further research. At the right, a bone, intended to be analyzed with DNA and a shard submitted to INAA.

which along the "Long-Wall" that runs in N-S direction of the eastern border of the site with the cemetery. The deposits extend far beneath this wall towards the East, meaning that this particular wall is of a later date, since it was built over the deposits of animal bones and date kernels. The duo Magen/Peleg excavated the eastern side of the southern plateau along the corridor between the settlement and the cemetery and their finds of animal bone burials have as yet to be published; however, they have been found toward the East too.

After having been granted permission to have access to two such animal bone deposits, I wanted to let the same samples of pottery and animal bones circulate among the various laboratories in order to verify the provenience of the pottery as well as to know to what period the animal bones and the pottery can be attributed. I considered it a pilot study because the sample was only a tiny bit of the hundreds of shards and bones that were found. It was, however, hoped that a specific date would also help to identify when the plateau people operated the pits on the southern plateau that were used as cooking facilities and when they had been in use. The DNA would perhaps make a bridge between the Dead Sea scroll skins and the animals that were slaughtered and eaten.

Three shards have been submitted to INAA in Budapest (Marta Balla) to learn their chemical fingerprint and therewith its connection with the local population or their relation with others from more remote sites, each with its specific chemical fingerprint. INAA provides in its total of about 30 elements also those three that are neces-

sary to date ceramics by Thermoluminescence, i.e. the abundances of thorium (Th), potassium (K) and uranium (U). These three are needed to correct for the alpha, beta and gamma radiation after a pot had been in use and was subsequently left and buried in the soil.

The bones were subjected to DNA analysis by Gila Kahila who performs DNA at the Koret School of Veterinary Medicine at the Hebrew University in Rehovot. The pottery and soil was sent to K. L. Rasmussen and his team in the University of Southern Denmark at Odense who performed Thermoluminescence on the pottery at the University of Southern Denmark at Odense. During my fellowship at NIAS at Wassenaar and the Lorentz Center in Leiden, in the Netherlands, I sent a part of the same bones to the Isotope Laboratory of the University of Groningen, where J. van der Plicht informed me within a week that no collagen was found in the bones and thus also no date could be established.

This lack of collagen has been explained that the temperature at the Dead Sea area is too hot and it is too dry to preserve collagen in bone. A different explanation was offered by Olav Roehrer-Erttl (Zangenberg & Galor 2006, 182) who claimed that collagen in Qumran bones "*had already been reduced to less than 1% due to the high salt content in the soil*". This should be further researched *ad fundum*.

In March 2008, also G. Kahila told me that also she did not find any collagen either in order to receive a DNA fingerprint of the subjected animal bones. The good news was that Gila had received access to more bone deposits and that there, perhaps, would be a few where DNA could be extracted from in the future.

The INAA data of the three pottery shards that have been found with the bones are described in Gunneweg and Balla's INAA paper in chapter 4 in this volume. The Thermoluminescence date of the two bone heaps, by means of the TL data of the analyzed pottery will be treated in chapter 10 by Rasmussen et al..

Only after that we have received a satisfactory answer from the analyzes can we start to formulate further questions that may help in the interpretations of this phenomenon of piles of bones that were buried beneath shards of pottery, which is unique to Qumran solely and may one of the many indications that the site is exceptional too.

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CHAPTER SIX

ANIMAL REMAINS FROM KHIRBET QUMRAN: A CASE STUDY OF TWO BONES (QUM 392 AND 393) FROM TWO BONE BURIALS

Gila KAHILA BAR-GAL[a], Tzviki ROSENBERG[a] and Charles GREENBLATT[b]

[a]The Koret School of Veterinary Medicine, The Robert H. Smith Faculty of Agriculture, Food and Environment, The Hebrew University of Jerusalem, Rehovot, 76100, Israel. [b]Kuvin Center for the Study of Infectious and Tropical Diseases, Faculty of Medicine, The Hebrew University of Jerusalem, Jerusalem 91120, Israel

Abstract. Two animal bones from the so-called 'Bone Burials' were submitted to DNA to be used as genetic markers to identify the animal whose bones were buried. The second aim of this study was to try to link the ancient DNA (aDNA) to the animals whose hide served the manufacture of parchment for the Dead Sea scrolls.

Keywords. DNA, Single Nucleotide Polymorphism, Mitochondrial gene.

Introduction

The interpretation of the archaeological remains found in Khirbet Qumran, dated to the late 1st century BCE to the early 1st century CE, has been debated for the last 40 years. During the excavations, from 1947 to 1956, scrolls were found in 11 caves along Wadi Qumran and in Khirbet Qumran. The findings consist of approximately 930 scrolls including books of the Hebrew Bible, sectarian writings of the local Qumran community, and about 200 previously unknown Jewish exegetical, homiletic and liturgical texts (Broshi, 1999[2]). Most of the scrolls were written on parchments made out of animal skins (Ryder, 1965). Some of the scrolls were nearly complete when found but the majority was greatly fragmented. Some of these fragments could be pieced together by matching text patterns, scribal characteristics, ink, and characteristics such as physical damage (Stegemann, 1995). The

origin of the manuscripts, the identity of the Qumran community and the relationships between the archaeological site and the scrolls are the subject of dispute among scientists (Broshi, 1999; Gunneweg, 2006). Molecular genetics and in particular ancient DNA (aDNA) study of the scrolls had indicated the possibility to use genetic markers to determine the animal species from which the parchments were made (Kahila Bar-Gal *et al.,* 2001; Kahila Bar-Gal, 2006). Moreover, the identification of two fragments as derived from the same individual was shown to be feasible. Identification of the fragments to the individual level will enable the grouping of fragments together to match new text and/or verify matches that have been done based on various other methods (Woodward *et al.,* 1996; Kahila Bar-Gal *et al.,* 2001; Kahila Bar-Gal, 2006).

Since their discovery the Dead Sea Scrolls (DSS) have been a subject of fierce controversy. In spite of the advance of scientific research of the DSS, the question of their provenance remains unresolved. A comparison of the DSS genetic profiles with the animal remains (from the site) would determine the relationships between the two sources and can shed light on this issue. A close relationship will suggest that the parchments were made out of those animal remains found at the site, meaning they were made from a herd kept/raised/slaughtered at Qumran. Apart from the scrolls 408 Caprinae bones were identified in the assemblage together with other archaeological artifacts (Zeuner, 1963). Unfortunately, these animal bones of the early excavations at Khirbet Qumran are not available. Therefore, this study was not carried out.

New excavations, on the southern plateau of Khirbet Qumran, carried out by Randall Price and Oren Gutfeld exposed deposits of animal bones buried within pots or beneath pottery shards. The location of the animal deposit at the southern plateau along the corridor between the settlement and the cemetery requires a determination of its date. If the animal remains are dated to the same period as the DSS, late 1st century BCE to early 1st century CE, they can be used in a study that will genetically characterize them. Genetic comparison of the DSS and animal remains will assist in determining the provenance of the DSS.

The goal of this study was to conduct a preliminary genetic study on two bones sampled directly from two piles of bones and shards excavated by Price and Gutfeld in 2006. Any success in this research will allow the genetic characterization of the bones to be compared to

the genetic profile of the DSS to determine the relationships if any between them.

1. *Materials and methods*

Jan Gunneweg of the Hebrew University of Jerusalem sampled one bone from a large pile of bones and shards (see figure 15a). The pile measures approximately 40 cm in diameter. In figure 15b, a bone is depicted that came from a small heap, about 35 cm in diameter.

The animal bones were in the two deposits together with pottery of which three pots, which have been analyzed by INAA and dated by TL (Thermoluminescense), see chapters 4 and 10 in this volume. The bones sampled received the laboratory code Qumran 392 and 393.

DNA extraction: The DNA from the two specimens was extracted using guanidine thiocyanate (GuHCL) and silica-based purification (Boom *et al.* 1990, Hoss & Pääbo 1993). This method was found to be the best method for the extraction of aDNA (Rohland *et al.,* 2005).

DNA amplification: The DNA samples were amplified via PCR (Saiki *et al.*, 1988) using multiple sets of primers to amplifying fragments of a mitochondrial gene cytochrome b (Kahila Bar-Gal *et al.,* 2002a,b). ABI-*Taq*Gold DNA polymerase was used to increase success rates. At the aDNA laboratory the work was performed in UV-irradiated hoods with protective clothing and face shields, while equipment and bench tops are cleaned with bleach and UV irradiation, in addition to other routine precautions (Cooper & Poinar, 2000).

Sequencing: Degradation of primers and unincorporated fragments were accomplished with Exo/Sap (USB), prior to direct sequencing with BigDye Terminator kits (PE ABI) and electrophoresis and detection on an ABI 3730 capillary sequencer. Sequences were inspected using Sequencher© 4.8 (GeneCodes®.) and initially BLASTed via NCBI's Mega-BLAST algorithm online (http://www.ncbi.nlm.nih.gov/blast/Blast.cgi) in order to verify amplification of cytochrome b genes.

Single Nucleotide Polymorphism (SNP) characterization: In order to determine the animal species we used also our Single Nucleotide Polymorphism (SNP) assay of the Coat color Melanocortin receptor 1 (MC1R) gene. The amplified amplicon (50 bp) has one SNP, Y13958: 183 C > T, difference between sheep and two ancient breeds of goats Shamit and Baladi (Figure 16A). Using a High Resolution Melt (HRM) feature on the Real Time PCR machine (Rotor-Gene TM 6000,

Corbett) the genetic difference was recorded (Rosenberg *et al.*, in preparation).

2. *Results*

DNA was extracted four times independently from each bone sampled. For each extraction five sets of primers were used to amplify the mitochondrial gene, cytochrome b. In order to increase the success rate every DNA extraction was amplified at least three times in different conditions. In spite of all these efforts the success rate was very low. There were only three positive amplifications: two amplicons from the "large heap" and one from the "small heap". The positive amplicons were directly sequenced to determine the species of animal. The comparison of the obtained sequences with the world database, using BLAST software, indicated a high similarity to bacterial sequences.

The extracted DNA was also amplified using a Real-Time PCR assay for a coat color gene, MC1R. The assay was designed to amplify aDNA from Qumran samples together with one other archaeological specimen from the same region, a skin dated to the 4st century BCE. The additional aDNA sample was used as a control to indicate that the SNP assay is valid for ancient samples. The results of this assay determined that aDNA can be amplified successfully (Figure 16B). Unfortunately, the amplification of the DNA extracted from the two animal bones from Qumran failed, meaning no DNA was recovered (Figure 16B).

3. *Conclusions*

In the last century the opportunity to study the genetic code of ancient samples became feasible (Pääbo 1993). It is important to note that the recovery of DNA from ancient samples is far from routine, as the researcher has to contend with the fact that *if* DNA is present it is degraded and often *no* DNA survives in the ancient specimens (Pääbo *et al.,* 2004; Yang, 1997). Extraction and amplification of DNA from ancient samples may be a challenging procedure due to small amounts of authentic aDNA, the high rate of foreign DNA contamination, *post-mortem* degradation and inhibition (Pruvost and Geigl, 2004). This study was an attempt to determine the feasibility of recovering aDNA

from two animal remains from the newly excavated deposit at the southern plateau of Khirbet Qumran. Unfortunately we failed to amplify successfully DNA from these two bones. The three positive amplifications probably represent a foreign contamination (Pääbo *et al.,* 2004; Pruvost and Geigl, 2004; Yang, 1997).

The failure of amplifying aDNA from these two bones can be by chance (we would be happy with 30% recovery) or can represent the low preservation of aDNA in the animal remains from the southern plateau of Khirbet Qumran. It is important to note that also C^{14} date could not be established on theses bones due to lack of collagen (Personal communication Hans van der Plicht of the AMS-C14 in Groningen). Theses finding can be explained also based on Salamon *et al*., (2005) study. They found that aDNA relatively better preserved in crystal aggregates inside fossilized bones, in addition the existence of insoluble collagen may indicate on a good preservation state. Other studies such as the unsuccessful amplification of aDNA from olive pits from Khirbet Qumran (Elbaum et al., 2006) and the missing collagen in the human remains from the site (Sheridan et al., 2003) can also support the degradation/absent of the DNA in the two animal bone remains. On the other hand, research conducted on the DSS favor the existence of aDNA (Woodward *et al.,* 1996; Kahila Bar-Gal *et al.,* 2001; Kahila Bar-Gal, 2006) and collagen (Broshi, 2004). This indicates that differences in preservation of biological material can be found among organic remains from the same site.

The correlation between the DNA preservation and the biological source was also shown in the Real Time PCR amplification assay for MC1R gene (Figure 16). The addition aDNA was extracted from leather found in a cave at the Judean Desert dated to an earlier period (4st century BCE). The leather, as the scrolls, preserved the DNA better than the bone material. The study on the DSS focused only on recovery of mitochondrial DNA (cytochrome b and D-loop) and indicated high preservation (Woodward *et al.,* 1996; Kahila Bar-Gal *et al.,* 2001; Kahila Bar-Gal, 2006). These results may be explained due to the tanning process (Sudha and Gnanamani, 2008; Vuissoz et al., 2007). It was published that aDNA was recovered from various manuscripts such as the Greek manuscripts (Poulakakis *et al.,* 2007). Real Time PCR and melt curves are extremely helpful when it comes to the amplification of very low copy numbers of target molecules and of reaction mixtures containing inhibitors as is common in aDNA studies. Pruvost and Geigl (2004) have shown that this methodology

increases the success rate of ancient samples and adds a higher reliability to ancient DNA studies. The high sensitivity of the technique can explain the false amplification (probably primer dimmer) of the Qumran sample (Figure 16).

Although aDNA was not recovered from the two animal bones from Qumran, aDNA was extracted and successfully amplified from animal remains from various specimens along the Judean Desert including Masada and Nahal Heimar (Kahila Bar-Gal, 2000). Therefore, we believe that the absence of DNA in two sampled remains of this study do not necessarily represent the preservation of the entire assemblage excavated by Price and Gutfeld. The fact that the animal remains from the early excavation are not available increases the importance of studying the specimens from the new excavations.

Ancient DNA studies of the archaeological specimens contributed to the interpretation of the site answering specific questions that cannot be answered using a different methodology (Kahila Bar-Gal, 2006). In order to answer the question concerning the DSS provenance we need to determine the genetic profile of the animal remains from the site and compare it to the known genetic characterization of the DSS (Woodward *et al.,* 1996; Kahila Bar-Gal *et al.,* 2001; Kahila Bar-Gal, 2006). We are compelled to continue this study due to the importance of this question.

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Two bones from the Price-Gutfeld excavation at Khirbet Qumran were sampled by Jan Gunneweg and used in the study: Qumran 392 and 393.

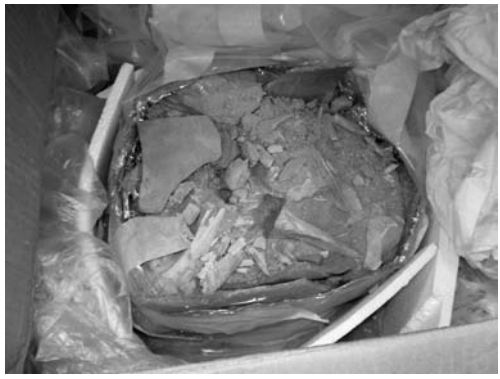
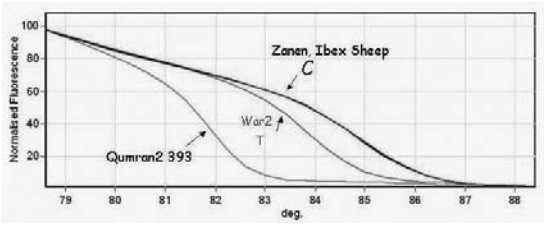
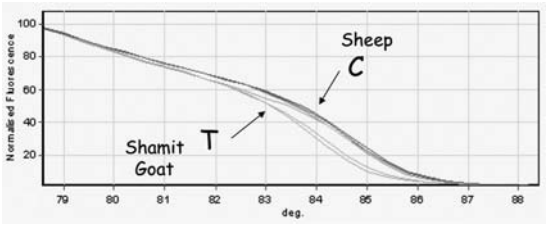


Figure 15a. "Large Pile of bones".

Figure 15b. Two fragments of the same bone found with the "Large pile of bones".



A.



B.

A= Positive control samples to determin the difference between goat and sheep. Sheep is recognized with a C (cytosim) while the goat breed Shamit is recognized with a T (tymin).
B = Positve control of a sheep with two ancient samples indicating that the DNA extracted from Khirbet Qumran bone had very low amounts of DNA preventing its successful amplification. The other ancient sample was successfully amplified and was found to be a goat.

Figure 16. High Resolution Melt (HRM) analysis after Real Time PCR amplification of an amplicon from the coat color gene MCA.

CHAPTER SEVEN

DEGRADATION OF PARCHMENT AND INK OF THE DEAD SEA SCROLLS INVESTIGATED USING SYNCHROTRON-BASED X-RAY AND INFRARED MICROSCOPY

Bridget M. MURPHY[a], Marine COTTE[b], Martin MUELLER[c], Marta BALLA[d] and Jan GUNNEWEG[e]
[a]Kiel University, Germany, [b]Laboratory, The Louvre, Paris, [c]GKSS Research Centre Geesthacht, [d]Nuclear Reactor at the TU of Budapest, [e]Archaeometry, Hebrew University of Jerusalem

Abstract. In this study, we are presenting the various analytical techniques that have been used to obtain information about the state of the Dead Sea scrolls at present and what could have been the cause of its deterioration. Did it start at the moment they were written due to the preparation of the parchment or to the binder that was used to make the ink fluid for writing. Synchrotron-based techniques will be employed to obtain a new look at the deterioration of the parchment and ink of the Dead Sea scrolls, using SR-based micro-X-ray and Infrared analyses.

Keywords. Synchrotron-based X-ray fluorescence (XRF), Fourier-transform Infrared (FT-IR), INAA.

Introduction

Since the early nineteen fifties, a continuous process of conservation of the Dead Sea scrolls and fragments of them has taken place under the supervision of the Israel Antiquities Authority. Nevertheless, the scrolls have become darker and more brittle by the day. Therefore, at this stage, the degradation of parchment in general and that of the Dead Sea scrolls in particular continues to present intriguing questions regarding restoration, preservation and conservation. Given the enormous importance of the texts on the Dead Sea scrolls as cultural heritage, answers should be given very soon before the material becomes illegible and eventually destroyed.

In general, degradation can be of environmental character, for example due to humidity, oxygen, sunlight as well as artificial light, temperature, or is induced by fungi, insects and airborne pollutants. The climate in the Dead Sea region is generally favourable for the preservation of organic materials as has been shown in recent studies on textiles (linen = cellulose fibers, wool = protein fibers) from differently exposed Qumran sites and from the Cave of Letters (Müller 2007, 877). In manuscripts written on parchment (collagen = protein) or papyrus (cellulose), degradation can be also due to the composition of the ink that was used, with such binders as Arabic gum, vinegar or wine, bees' honey, oil and animal bone glue. Further degradation may be caused by the tanning process that was applied on the preparation of the Dead Sea parchment with the use of lime and perhaps even ammonium, the latter deriving from stale urine. The degradation of parchment is therefore a complex problem since it involves either one or more or all of the above.

Degradation and alteration are sometimes used as one and the same phenomenon. Nevertheless, alteration in parchment would make the text illegible due to exfoliation of the uppermost stratum of parchment while the parchment has kept its characteristics whereas degradation would destroy the parchment on which the text was written.

The latest publications on the subject by scholars such as Steckoll (1968, 91), Plenderleith (1955, 39) and Nir-el and Broshi (1966, 157) have been focused on the composition of ink and on the composition of parchment with and without ink (see next section). Yet, the question of what are the various material processes that work together to degrade parchment has not been touched upon. The influence of tanning as well as that of ink with its complex composition has also been overseen because of the difficulties to point to specific degrading agents as well as to measure them.

1. *Previous work*

Already in 1955, during the excavations at Qumran, Plenderleith (1955, 39) analyzed the ink by spectroscopic methods and found that it was carbon with traces of Ca, Mn, Fe, Cu, Sn, Pb and Ag. These data were eventually published by Steckoll (1968, 91).

Energy-dispersive X-ray fluorescence (EDXRF) analyzes of Qumran parchment (Broshi 1985; Nir-el 1996, 157) showed high concentra-

tions of Cl, K, Ca and Br that all are expected if the skins were dehaired and tanned with the use of salt water from the Dead Sea that is high in the above-mentioned elements (Haran 1985, 21). It was argued that due to the hygroscopic nature of alkali and alkaline earth metals, the moisture of the Dead Sea scrolls would increase, causing denaturation of the parchment collagen to gelatine as a result of which also the strength of the parchment would be attacked (Nir-el 1996, 157; Lukas 1962, 38).

In 1988, the Smithsonian Institution tested the relationship between the properties of collagen and stress in an environment that changes from the relative humidity in which the scrolls have survived and to that in which they are stored now (Mecklenburg 1988, 231-244). It was found that the ink is the primary cause of flaking and cracking of the underlying parchment. The latter was suggested as coming from the different contraction properties of the glue that was used on parchment. This result reawakened interest in this topic and became one of the two triggers for the work presented here. The Smithsonian Institution concluded that the degradation of the Dead Sea parchment (especially of the Genesis Apocryphon) was due to environmental conditions such as relative humidity and temperature as well as to the composition of the binding matter of the ink such as gum and animal bone glue. Nevertheless, the conclusion was that "*these binding materials in the Qumran scrolls have never been analyzed or identified*" (Nir-El 1996, 157). This quote was the second trigger for the present research.

2. *Iron in ink and parchment*

Returning to the above statement that the ink used for writing the Dead Sea scroll was carbon-based (Steckoll 1968, 91), one should still

Table 1. List of elemental concentrations in fresh and ancient parchment (Balla 2004). Abundances in parts-per-million (ppm) if not stated otherwise.

Element	Fresh parchment	Ancient parchment
Ca (%)	4.9 +/– .1	2 +/– 0. 1
Fe	149 +/– 6	1500+/– 10
Na (%)	0.11 +/– 0.01	1.4 +/– 0.01
K	418 +/– 13	819 +/– 25

consider the possibility of iron-containing ink as a source of parchment degradation, starting with the corrosion of iron. Excluding iron in ink would in turn corroborate the hypothesis of the binder being responsible for parchment degradation starting at the letters written on it.

Ginnell (1993) at the Paul Getty Conservation Institute published X-ray fluorescence (XRF) research on degraded fragments of the Genesis Apocryphon and other Qumran parchment and decided that iron was not a constituent of the ink used. Nir-El and Broshi (1996, 157) set it as their goal to identify the black ink of the Qumran scrolls by its pigments as well as to study the degraded Genesis Aprocryphon in the context of its ink. The paper can be summarised as follows: The base of the black ink on the Dead Sea scrolls is carbon that could have been obtained from lampblack or soot etc., thus corroborating Plenderleith's conclusion (1955, 39). Since the addition of metallic compounds to obtain black ink has been invented after the Qumran period of the 1st century AD, iron cannot have been the main constituent of ink that was used in the Dead Sea scrolls. Measurements done on parchment alone and those of parchment plus ink provide similar values. Whereas on the one hand iron was almost absent in ink, parchment, on the other hand, contained a high amount of iron. The PIXE analyzes (Broshi 1985) showed varying iron values that range between 114-800 nanograms per square millimetre, high compared to that of other chemical elements, but not constant and uniform over the fragment, but on the contrary reaching abnormally high levels at several spots on the fragment.

In order to finally clarify the role of iron in the parchment of the Dead Sea scrolls, we analyzed freshly made parchment and a scroll parchment fragment by the means of instrumental neutron activation analysis (hence INAA) at the nuclear facility at Budapest (Balla 2004). The results provided the elemental composition of the parchment as shown in Table 1. The data showed 149 ppm and 0.15 % iron, respectively. The old parchment contains a tenfold quantity of iron more than fresh parchment. It may point to the use of an iron knife to remove flesh, fat and hair of the goatskin 2000 years ago. The same is true for the sodium content; Na in ancient parchment is ten times higher than in fresh parchment. On the other hand, the calcium content of ancient parchment is the half of that in fresh parchment.

We shall not discuss the possible sources of iron in parchment here. The relatively low iron content of both fresh and also ancient parch-

ment might well contribute to an overall degradation. However, since the ink is carbon-based the reason for the accelerated degradation is very probably not related to iron but rather linked to the composition of the ink binder that was used.

3. *Aims of the present study with synchrotron radiation*

Based on our previous knowledge (see above), we can formulate the following questions to be answered by the present study:

What ink binder has been used for the Dead Sea scrolls? Is it possible to see the remains of fluid ink that travelled down into the surface and deeper into the parchment? What was the tanning process for the Dead Sea scrolls? Are there differences between data obtained from the recto and retro side of the parchment due to a lime treatment or perhaps tanning? Can one locally measure the lime and/or tanning material penetration? Does the latter contribute to parchment degradation in the long run? What does the ink binder contribute to the degradation of parchment? Is there organic matter connected with the ink layer? Are there differences in the elemental composition in regions with and without writing on them? What can be the way to stop degradation in the light of our results? How can one improve the present status of parchment in taking into account: Light, humidity, dust particles, chemical compounds and organic material?

In order to answer the above questions, we first need to establish the state of parchment degradation of the Dead Sea scrolls by measuring the physical and chemical differences of ancient parchment juxtaposed to those of fresh parchment by using thin cross sections and scanning through the various strata of dirt, dust, skin and ink. Residues (scrapings) from inkwells should be taken into account as well. To this end, we used two different techniques based on synchrotron radiation. The inherently low divergence of synchrotron radiation allows for microfocusing, thus providing the required microscopic spatial resolution of the experiments. The first experimental technique applied was X-ray microscopy. X-ray microscopy mainly relies on X-ray fluorescence (XRF); a common technique for elemental analysis. The second technique of Fourier-transform Infrared (FT-IR) spectroscopy measures the strength (frequency) of molecular bonds and is used to identify certain molecules or molecular groups.

A similar study has been carried out on the skins of mummies and on hair (Cotte 2005, 159). However, mummy skin could only be studied at its surface due to limitations in sampling the mummy skin. In terms of spatial resolution, we profit from the fact that we have parchment pieces at our disposal, small pieces of which could be sacrificed for the preparation of cross sections.

In our study on the degradation of the Dead Sea scrolls using synchrotron radiation, we tried to keep the most relevant three players in mind:

The prepared skin of an animal and what the lime treatment or perhaps the use of a tanning agent to dehair the skin process, would do to this skin.

The use of ink and the binder to combine the carbon and binder into a fluid state as well as other materials to make the ink well adhering and resistant.

The notion of time and place; what conditions are playing in a two-millennium period of time and in the place where the manuscripts have been found and where they are preserved at present.

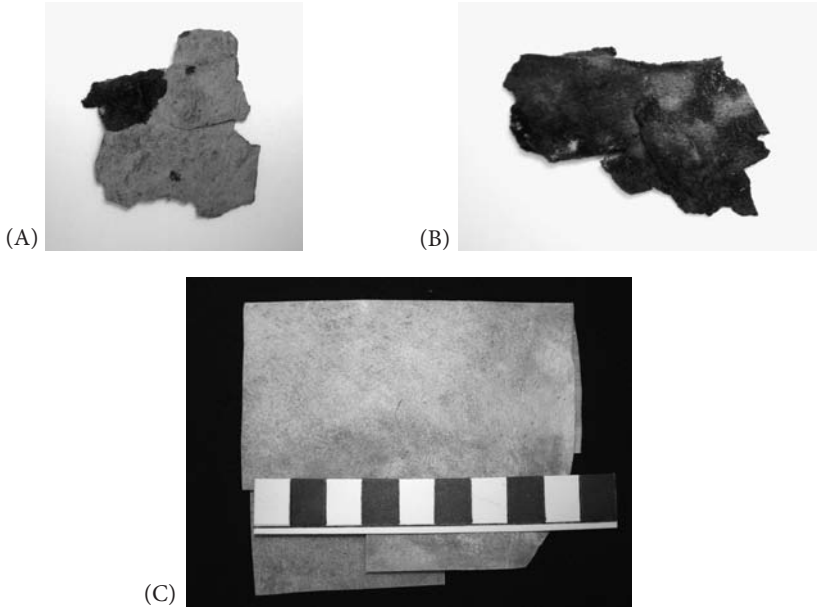


Figure 17. Representative fragment of Dead Sea parchment used in these experiments are shown in the figure. All pieces came from 4Q992: (A) containing an ink dot; (B) in an extreme state of degradation and (C) a fresh piece of goat parchment measured as a reference sample. The black and white bars are 1 cm long.

Table 2. A list of specimens investigated in this study.

Sample Code New Code	Qumran Registration	Description	
Scrapings			
QUM 222	KhQ 4638	Black substance scraping from an inkwell	
QUM 223	KhQ 2989	Scraping from black material of a large Qumran jar	
QUM 230	KhQ 1465	Scraping from a small jar	
QUM 233	Cave X:2	Scraping of a substance adhering to inside of QUM 234, a small jar.	
QUM 235	Cave X:2	Scraping of cement-like material adhering to outside of QUM 234	
Parchment			
QUM 1	Fresh goatskin made into a sheet of parchment		B1
QUM 2	Cave 4	Suspected piece of Papyrus	B3
QUM 3	Cave 11	Parchment	B4
QUM 4	Cave 4	Parchment with a dot of ink	C1
QUM 5	Cave 4	Parchment with an incised line	C2
QUM 6	Cave 4	Parchment in a very degraded state	C4
QUM 7	Cave 4	Parchment with a dot of ink	C5

4. *Experimental details*

Important questions in the search to understand degradation of the Dead Sea Parchment are:

1. Which elements are present in the parchment samples and are the same elements also found in the ancient and the modern samples?

Investigating calcium content and location can give information on the preparation of the parchment as it is thought that lime was used in the tanning process and sulphur which is a constituent of skin and additionally is present in high concentrations in urine also thought to be used in the tanning procedure, additionally chlorine, and potassium may give information on the general state of parchment. One method very well suited to the chemical analysis of these key elements is X-ray fluorescence. In order to achieve a position resolution of the parchment in profiles, micro fluorescence is required. This is only possible with special focusing optics and the intense X-ray beam available at modern

synchrotron sources. In addition by scanning the X-ray energy it is possible to determine the local chemical environment of the relevant components using the Near Edge X-ray Absorption Fine Structure (NEXAFS) method. Again utilizing a micro-beam provides a high position resolution.

2. Which organic material is present and in what condition?

The presence and state of the organic material, namely proteins, carbonate and oxalate can give vital clues to the degradation state of the parchment as shown in a study of Mummy skin (Cotte 2005, 159). All of the above methods are available at the X-ray microscopy beamline, ID21 at the ESRF and the experiments discussed here were carried out there.

The samples available for these investigations were six fragments of ancient parchment of the Dead Sea scrolls previously investigated (samples No. 27-32 in Weiner, 1980,820) and in order to have a comparison, a piece of modern goat skin parchment. Pictures of a representative selection of the investigated samples are shown in figure 17 A-C.

QUM 4 contained a partially lifted off ink dot and QUM 5 an impressed line into the parchment. Most of the samples were moderately degraded apart from QUM 6 that appeared to be in an extreme state of degradation, the surface being blackened and the sample very brittle to touch. Additionally four samples of scrapings from different vessels found at Qumran of which at least two could have been the raw or ready prepared materials used for ink have been chosen for these experiments. The find location of each sample has been recorded in Table 2.

The experiments were carried out at the X-ray microscopy beamline ID21 and on its Infrared facility, at the European synchrotron radiation facility (ESRF) in France. As it was intended to investigate the samples in absorption mode thin cross sections were required. Samples of parchment were cut into small pieces and were then prepared by embedding them in a fluid resin that hardened over 24 hours. Each resin block of about 0.5 x 1.0 cm^2 containing one sample was then cut with a microtome so that one obtained cross-sections of 10 μm, 6μm and 4 μm respectively. The cuts of cross-sections were placed between two sheets of parafilm to facilitate handling of the samples. One must take care that the resin, in its fluid state, may have interacted

with the sample; in particular, it might have diluted some of the ink on the parchment. The suspected ink scrapings were in powder form and they mixed with potassium bromide and pressed into pellets for the Fourier Transform Infrared experiments.

The μ-beam Scanning X-ray microscope at ID21 (set up shown in figure 18) benefits from an intense undulator beam focused by a zone plate objective lens element and defined by an order sorting aperture. The beam size at the sample is 1.0 x 0.5 μm. The sample sits on an X-Z translation stage and may be scanned vertically and horizontally in the beam to obtain profile information. It is possible to obtain both absorption and fluorescence data simultaneously allowing a position resolved elemental analysis of the samples in the investigated energy range. For the following measurements an X-ray energy of 4.1 keV was chosen.

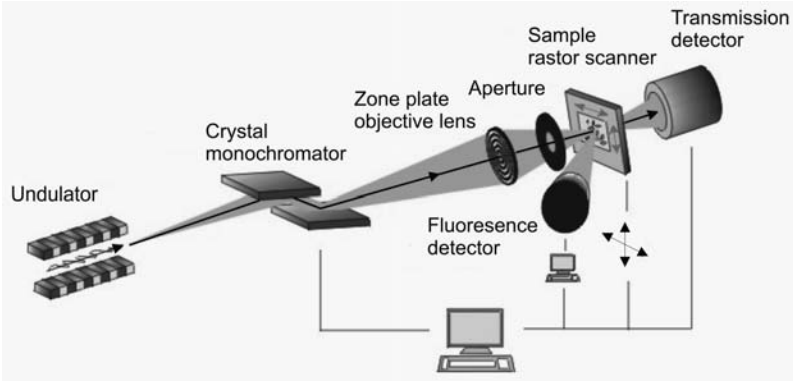


Figure 18. The experimental set up at the scanning X-ray microscope on ID21 at the ESRF (color photo in Figure V).

At the Fourier Transform Infrared (FT-IR) facility scans were carried out on a FT-IR spectro-microscope. It is composed of a Thermo Nicolet Nexus Infrared bench and an Infrared Thermo Nicolet Continuum microscope. The novel feature of this instrument is that it is possible to use IR from the synchrotron or from an internal source. Our data were collected on the same sample cross sections as in the micro fluorescence experiments in transmission/absorption mode over an energy range of 4000 cm^{-1} to 1000 cm^{-1} with a beam size of 8 x 8 μm^2 generally in 5 μm steps. A combination of all three above tech-

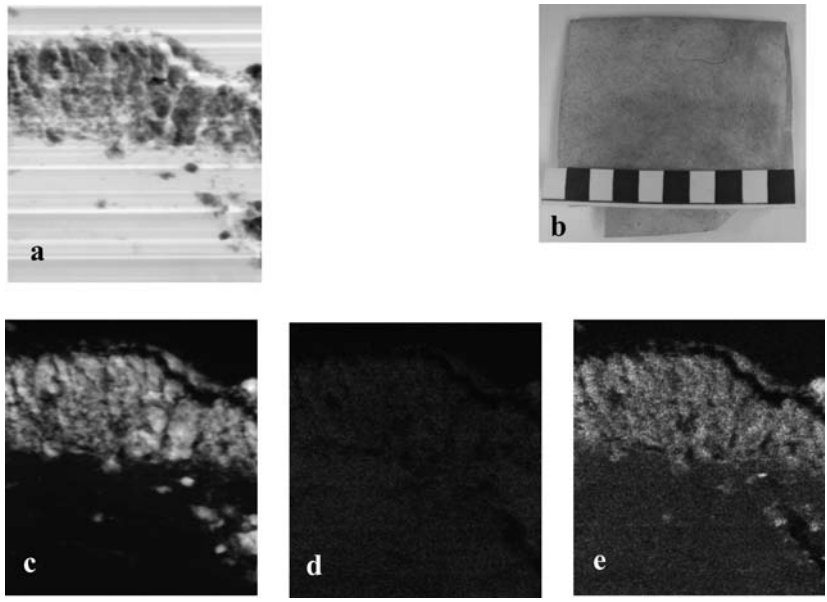


Figure 19. Absorption and fluorescence maps of modern goat parchment (QUM 1; 90 x 100 μm^2): (a) absorption profile (b) photograph of the fresh parchment sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map. It is possible to see the embedding resin surrounding the section of parchment in the absorption profile. Color photos in Figures VI a through c.

niques allowed us to obtain new important data on the chemical composition of ancient parchments and carry out a direct comparison with modern parchment.

5. *Results and discussion*

First let us consider the micro-fluorescence experiments. The results from sections of fresh parchment, Dead Sea parchment with no ink and parchment containing ink are shown in figures 19-22. Of all the three thickness cuts, it turned out that the 4-micron thick samples gave the best results and these are the results shown here. Scans of 90 x 100 μm^2 in 0.5 μm steps were performed on all samples. In each case an absorption map, maps of calcium, chlorine, sulphur and potassium were simultaneously collected with a counting time of 0.5 s for each step.

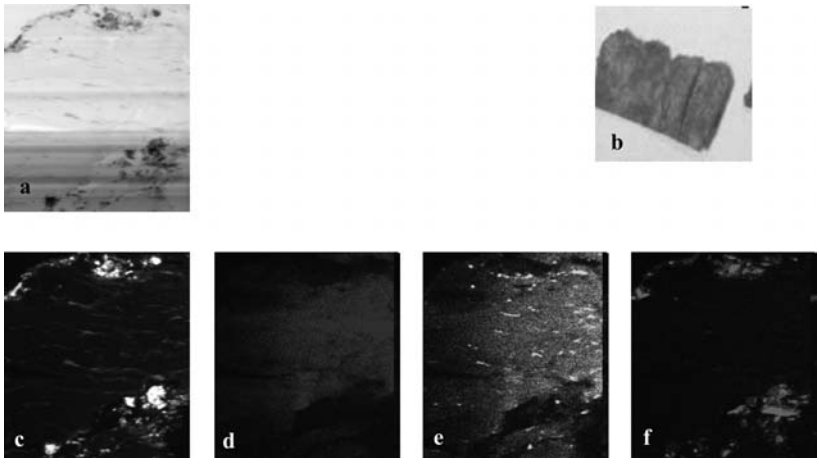


Figure 20. Absorption and fluorescence maps of Dead Sea parchment (QUM 3; 90 x 100 μm^2): (a) absorption profile (b) photograph of the sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map and (f) potassium.

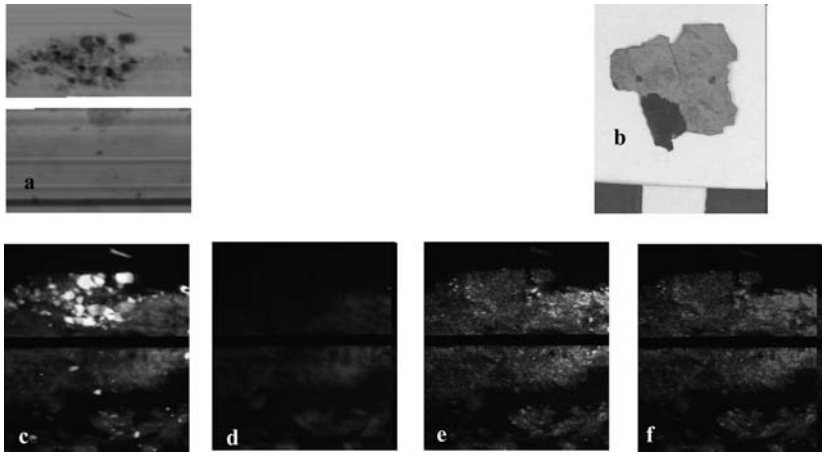


Figure 21. Absorption and fluorescence maps of an ink dot on Dead Sea parchment (QUM 4; 90 x 100 μm^2): (a) absorption profile (b) photograph of the sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map and (f) potassium. The white/black stripe running through all images is due to a loss of the X- ray beam during the measurements.

The data collected on the modern goat parchment is shown in figure 19. The images show the top 100 μm of the parchment cross-section. In figure 19 (a) an absorption image is presented. Figure 19 (c), (d) and (e) show the chemical maps for calcium, chlorine map and sulphur. The data show that the elements distribution is homogeneous through the cross-section. Similar data, not shown here, was collected at other positions on the sample.

In the case of the Dead Sea parchment without ink (figure 20) the absorption image tells a different story, areas of high contrast are present close to the parchment surface. Additionally there are some small-elongated structures 1 to 2 μm high and 10's of microns long present. The chlorine map shown in figure 20(d) has an even distribution but for calcium and sulphur and potassium there are very visible differences. Let us first consider the calcium map in figure 20 (a). Close to the top and bottom surface we observe agglomerations containing calcium, also there are some smaller calcium containing structures present in the center of the cross-section. The image has a lamellar structure perhaps associated with degradation. It is very interesting to note that unlike the near surface calcium agglomeration the deeper structures are also present in the sulphur map. In contrast, the potassium map has a high contrast very similar to that of the surface near calcium deposits. This is a first indication that the form of the calcium in the near surface deposits may be different from those deeper in the parchment. The data collected on other sections of this sample exhibited the same behaviour. The chlorine map was, as in the case of the modern parchment, homogeneous over the sample cross-section area.

Highly interesting findings were obtained in the case of the section containing the ink dot. The image cross sections shown in figure 21 were taken at the position of the ink dot. The absorption image (figure 21 (a)) exhibited an extremely high contrast close to the ink dot position extending over 40 μm in a radial direction. There are additional areas of high contrast in the center of the parchment larger than those seen in the parchment without ink. They range in size from 1 μm^2 to 5 x 10 μm^2. These features were clearly reproduced in the calcium map. In general all the chemical maps are less homogeneous than for the no ink case or the modern parchment. Interestingly, a feature of this cross-section was that a major delamination was observed in the center of the cross section, perhaps an indication that the parchment close to the ink suffered greater degradation than parchment where no ink was present. The chlorine map exhibits a degree of structure not seen

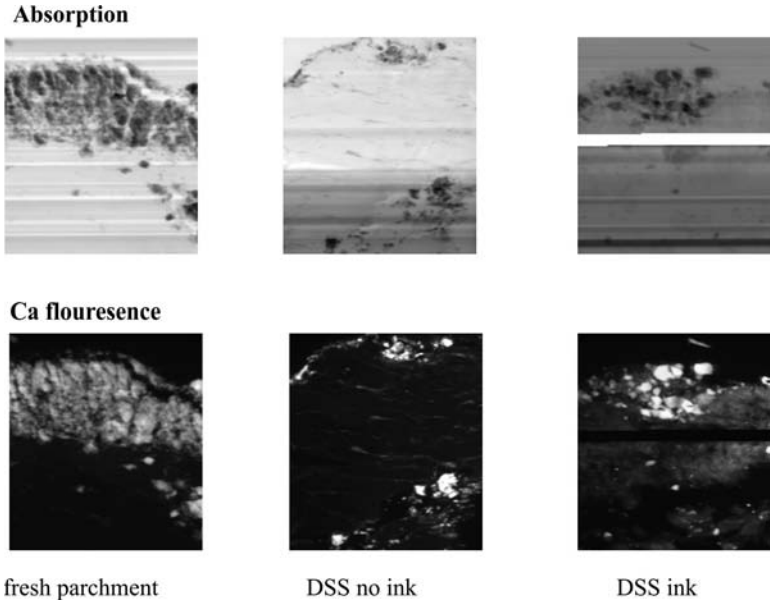


Figure 22. For comparison absorption and calcium fluorescence maps of fresh parchment (QUM 1, QUM 3, QUM 4; 90 x 100 μm^2), Dead Sea parchment with no ink and Dead Sea parchment with ink are shown. The increasing degree of degradation can be seen in each case.

in the other samples. There are some conglomerations present in both the sulphur and the potassium map but neither displays a correlation to the calcium map.

As a discussion aid, absorption and calcium fluorescence maps of fresh parchment, Dead Sea parchment with no ink and Dead Sea parchment with ink are shown together in figure 22. The absorption image and the calcium distribution are in good agreement in all cases indicating that calcium is one of the main absorption constituents in the parchment samples. In the three cases shown a different distribution is observed. For the freshly prepared parchment, the contrast in both the absorption and calcium fluorescence image is very homogeneous; we therefore take this as an indicator that little or no degradation is present. When we consider the Dead Sea parchment with no ink there appear to be two processes at work. On the one hand, the calcium distribution is no longer even throughout the sample and has formed conglomerations close to the surface. On the other hand, there are some stripe-like calcium structures present in the deeper area or

the cross section. This raises the question if it is the same form of calcium in both cases. We will return to this question shortly. In the section with ink present there is an even greater degree of inhomogeneity. In the absorption scan the contrast at the surface is so high that it is no longer possible to get useful information in the center region. With the calcium map we can see there is a build up of calcium close to the surface in the form of many conglomerations. The extent and contrast of these structures is much higher than that in the parchment without ink (figure 22). This is a strong indication that either the ink or the binder is a major cause of degradation in these parchment samples. As one observes the three samples side by side it is clear that the healthy skin like structure of the fresh parchment is no longer visible in either of the Dead Sea samples also indicating degradation.

As the calcium appeared to perform a critical role in indicating the state of the parchment we investigated this topic in more detail for the sample with the ink dot. Scanning the energy of the X-ray beam around the absorption edge of calcium, near Edge X-ray absorption spectroscopy was performed locally at points of specific interest in the energy range 4.02 to 4.14 keV. The points where NEXAFS scans were carried out are indicated in figure 23. Generally close to the surface the NEXAFS scans indicate the presence of calcium carbonate ($CaCO_3$). One example is shown in figure 23. Scans were performed along a line starting from the surface travelling towards the center of the cross sec-

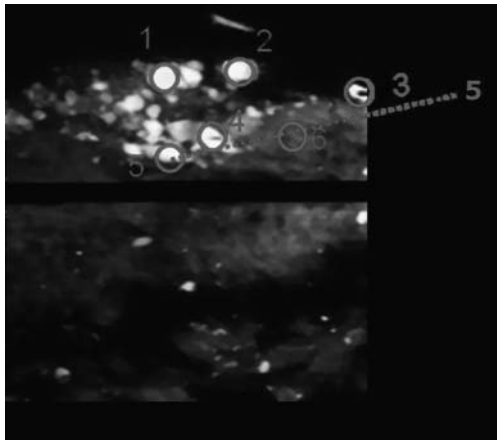


Figure 23. On the right hand side, a NEXAFS scan performed at point 5 in the calcium fluorescence map is shown. The scan shows that at this point calcium carbonate ($CaCO_3$) is present. (Sample QUM 4)

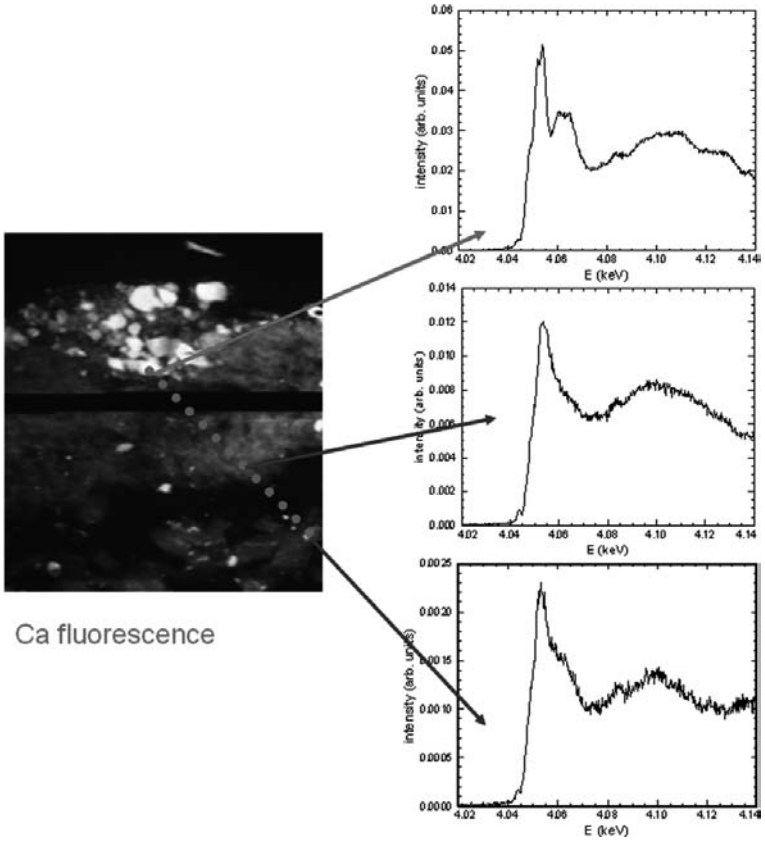


Figure 24. Profile of the calcium content in the parchment with ink cross-section showing that the calcium carbonate ($CaCO_3$) signature is strong close to the surface. Tricalcium phosphate $Ca_3(PO_4)_2$ becomes more dominant as one travels further from the surface into the body of the parchment but drops off in intensity the further away from the ink dot one travels. (Sample QUM 4)

tion. As one penetrates into the parchment the NEXAFS indicate that the signal of Tricalcium phosphate $Ca_3(PO_4)_2$ becomes more dominant. The signal intensity dropped off rapidly indicating that the concentrations of $Ca_3(PO_4)_2$ are much lower as the distance from the ink dot increases. One possible interpretation is that the calcium carbonate is a remnant from the tanning process (lime, whitener). In this consideration, the apparent build up of calcium carbonate may have protected over the centuries the ink. Calcium phosphate or bone ash is a constituent of bone glue and may be associated with the ink binder.

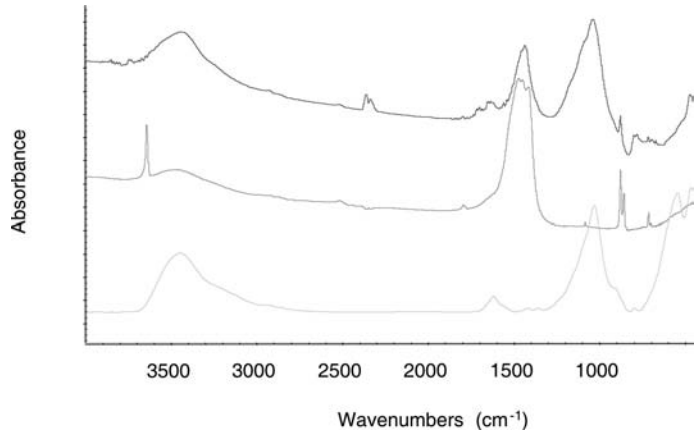


Figure 25. FT-IR spectrum of jar scraping QUM222 (top) with reference spectra of calcite (middle) and burnt umber (bottom).

The scrapings of the jars (QUM 230,233 in Table 18) were only analyzed by FT-IR spectroscopy. Only inorganic material could be found and identified, there were no traces of any organic compounds. QUM 230 and QUM 233 (from the inside of jars) are mainly gypsum ($CaSO_4 \cdot 2H_2O$) with a very small amount of calcite (crystalline modification of $CaCO_3$). The outside of the same jar (QUM 235) contained calcite as well; however, there is a major clay contribution with a spectral similarity to burnt umber, a brown clay pigment. The same mixture is found in the QUM 222 powder sample that was actually a black substance from an inkwell. The other black scraping (QUM 223) is better matched with a different clay, bentonite. Even though bentonite has the property of adsorbing relatively large amounts of protein molecules from the aqueous solution, also here no traces of organic material are found.

The FT-IR results on the jar scrapings did not yield any hints on the type of binder that might have been used. It is not unlikely though that all organic material simply did not survive the 2000 years before excavation. The minerals found in the powders analyzed are most likely just particles from the soil and the jar material. The presence of burnt umber is not definite (figure 24) as other clays have similar Infrared spectra. It could be thought that the inhabitants of Qumran burnt their own umber to produce brown pigment, using it as paint or ink, which was then applied either on parchment, on mortar or on pottery shards, called ostraca.

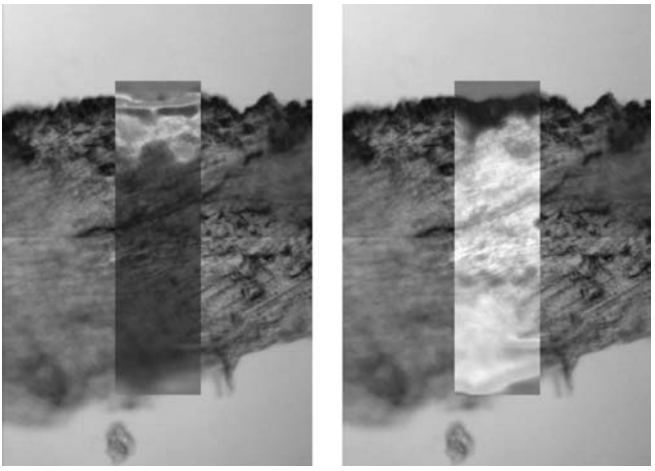


Figure 26. Microscope images of a thin section of fresh parchment (QUM 1) with an overlay of FT-IR maps of carbonate band intensity (left) and amide I band intensity (right). FT-IR map size is 80 × 272 μm² with 8 μm step size in the two directions For color photo see, Figure VII).

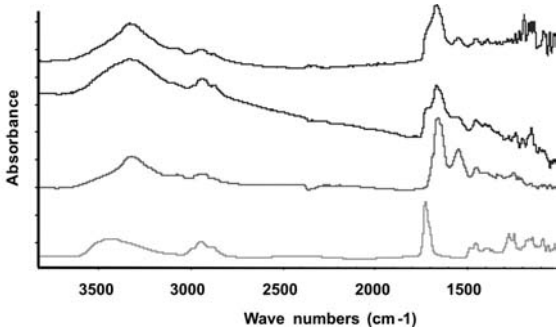


Figure 27. FT-IR spectra taken on two positions of a thin cross-section of QUM 4 (two upper spectra) in comparison to fresh parchment (third spectrum from top) and the signal of the embedding resin (bottom). The amide band domain lies between 1650 and 1500 cm^{-1}.

Infrared microscopy maps of fresh parchment (QUM 1) showed a clear distinction of a layered structure (figure 25). The main characteristic of the upper layer of about 30 μm on thickness is the presence of an intense carbonate band a ~1450 cm^{-1}. As it coincides with a strong Ca concentration found by XRF, this upper layer is an effect of treating the writing surface of the parchment with calcium carbonate to

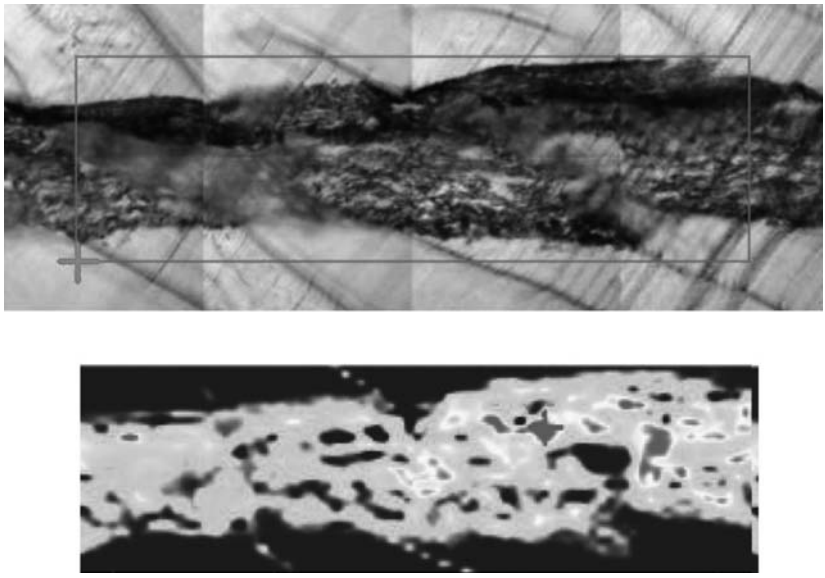


Figure 28. Microscope image of a thin section of QUM 7 (top) and amide II band intensity (bottom). FT-IR map size is 860 × 270 μm² with 10 μm step size in the two directions. The ink dot is located on top of the section in the center (horizontal position: 6100 μm) with a hole well visible underneath the ink area (for color photograph see Figure VIII).

brighten it up. The signal from the lower layer is classical of animal tissue (proteins, here collagen). The high intensity of the characteristic amide bands (figure 25, third spectrum) can be used to extract a map of tissue concentration in the cross-section (figure 25, right). The tissue appears very compact and homogeneous across the parchment. The embedding resin signal does not interfere in the amide band domain (figure 26, bottom).

The Infrared spectroscopy results on all Dead Sea scroll parchment fragments are very similar to features obtained on modern parchment (figure 26). A main difference is that on modern parchment the upper layer contains a high quantity of carbonate, which cannot be discerned in ancient samples. It might be present but at very low concentration or more in homogeneously distributed, as suggested by the occasional agglomeration of calcium compounds found by XRF (see above). The structural integrity of the fresh parchment (see amide map in figure 27) has changed into an inhomogeneous, porous structure with signs

of delamination (amide map in figure 27). In contrast to what was expected, there are few chemical signatures characteristic of degradation. Even on the visibly highly degraded and dark Dead Sea scroll parchment fragment QUM 6 a signal typical of proteins is observed, in association with some minerals among which are calcium oxalates. Oxalate had previously been observed on mummy skin (Cotte 2005, 159) as a sign of degradation. On the very fragile and brittle sample QUM 4, weak degradation signs could be present as well. The generally good preservation of the parchment tissue may be explained by the tanning treatment performed when producing parchment. Tanning seems to highly limit skin degradation.

Of particular interest was the association of parchment degradation with ink. Figure 28 shows a cross-section through an ink dot in sample QUM 7. In the micrograph (top), it is well visible that a thin black surface layer covers a hole underneath. The surface layer is completely invisible in the amide II band intensity map (figure 28, bottom). All collagen in the parchment must have been destroyed in this layer under the influence of the ink and the binder therein. Another small difference should be noted around the ink dot, with a higher signal at ~1630 cm^{-1} versus the signal at ~1655 cm^{-1}. This shift is usually interpreted as the highest proportion of the β-sheet structure of proteins secondary structure, versus α-helix, and thus indicates a local change of the collagen structure (which is actually based on triple α-helices). One should note, however, that the amide band intensity shift could also be an edge effect due to the presence of a hole under the ink.

6. *Conclusions*

Modern synchrotron-based Infrared and X-ray microscopy techniques have shed some light on the nature of degradation in the parchment of the Dead Sea scroll found in Qumran. Returning to the aims, we state the following conclusions:

It is likely that a bone glue based ink binder was used. The decay of the calcium phosphate (bone ash) signal further away from the ink spot into the bulk of the parchment suggests that the ink permeated into the parchment.

Though we could not identify the tanning process, we could verify that it provides an effective preservation. The organic components of

the parchment are still present. However, structural degradation is observed: the homogeneous, compact appearance of fresh parchment is changed into a more porous structure. Calcium carbonate may have been used to lime-whiten the Dead Sea scroll parchment, sometimes even on both sides. In particular, large agglomerates are still present under the ink.

The ink apparently destroyed the parchment in its immediate vicinity, as no organic matter could be found in the parchment regions penetrated by ink, and holes are visible underneath them. The neighbouring regions also show signs of organic denaturing.

We hope that these indicators will be useful for the preservation of the Dead Sea scrolls and other ancient parchments. Keeping the scrolls in a temperate dry environment will decelerate the chemical processes harmful for the parchment and reduce the diffusivity of attacking radicals.

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CHAPTER EIGHT

RADIOCARBON DATING AND QUMRAN

Johannes van der PLICHT[a,b] and Kaare L. RASMUSSEN[c]
[a]Center for Isotope Research, University of Groningen, [b]Faculty of Archaeology, University of Leiden, the Netherlands,[c]Institute of Physics and Chemistry, University of Southern Denmark, Odense, Denmark

Abstract. Samples from Qumran dated by Radiocarbon are potentially contaminated with preservatives or other contaminants. This includes some of the Dead Sea Scrolls, which were treated with castor oil in the 1950s. This castor oil is not removed by the standard AAA pre-treatment used for ^{14}C samples. We developed a pre-cleaning step, to be applied before the standard ^{14}C procedure, which has shown to remove such contaminants effectively.

Keywords. Radiocarbon, dating, contamination, Qumran, Dead Sea Scrolls

Introduction

The Radiocarbon (^{14}C) dating method was developed during the years around 1950 [1]. Since that time, several "revolutions" have improved the method considerably. Among the most significant ones are improvements in measurement precision, the introduction of AMS, and calibration of the ^{14}C timescale.

AMS (Accelerator Mass Spectrometry) enables small (milligram size) sample analysis [2]. This is a factor of 1000 less than the original, so-called conventional method. AMS therefore enables ^{14}C dating of precious and intrinsically small samples—artwork, Neanderthal bone, delicate artifacts, pollen, and the Dead Sea scrolls.

Calibration now enables absolute dating back to more than 10.000 years ago [3]. In turn, this spawned "revolutions" in many fields of application, among which archaeology. It is important to note that Radiocarbon provides a "yardstick of time", enabling the measurement of past time by scientific means, independent of cultural assessments.

The method enables chronological comparison of different areas at excavation sites and also between sites and regions. This is essential for proper interpretation of archaeological or stratigraphical layers and association with data from other fields [4].

While the method is basically simple, it is complex in detail and errors in matters concerning both fieldwork and technical laboratory aspects. Therefore, quality control is necessary to build up reliable ^{14}C chronologies. This involves regular laboratory intercomparisons, duplicate measurements of samples, issues such as conventional versus AMS, sample selection, association, contamination, and others [5], [6].

In this contribution, a short review of the principles of the ^{14}C method, the conventions and the most recent developments are given, including the 2004 calibration of the timescale. Quality control issues and sampling strategies will be discussed. This covers the state of the art of ^{14}C dating with an emphasis on matters relevant to Qumran.

1. *The ^{14}C dating method*

The element Carbon consists of 3 isotopes in nature: ^{12}C, ^{13}C and ^{14}C. These three isotopes are all forms of Carbon with different atomic masses (12, 13 and 14, respectively). The isotopes ^{12}C and ^{13}C are stable and have abundances of about 98.9 and 1.1%, respectively; the isotope ^{14}C is not stable but radioactive, and has an extremely small natural abundance of about 0.0000000001% or 10^{-12}.

The isotope ^{14}C is continuously produced in the earth's atmosphere by cosmic radiation. Radiocarbon decays with a half-life of 5730±40 years [7]. A stationary state of production, distribution between the main carbon reservoirs (atmosphere, ocean and biosphere) and decay results in a (more or less) constant ^{14}C concentration in atmospheric CO_2 [8].

The ^{14}C enters the biosphere via photosynthesis in the plants. Next, it finds its way in all living organisms via the food chain. Upon death, there is no longer equilibrium, and the amount of ^{14}C decreases because of radioactive decay. This is schematically shown in figure 29. The half life is defined as the time needed for half of the radioactive ^{14}C atoms to decay. Thus, by measuring the amount of ^{14}C remaining in the sample its time of death can be derived. This is the simple basic principle of the ^{14}C dating method.

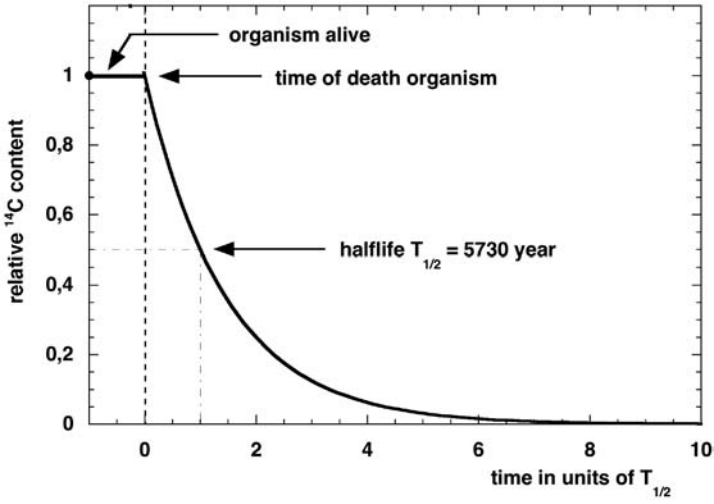

Figure 29. The radioactive decay curve for ^{14}C

In practice, samples back to 50.000 years ago (about 9 half lives) can be dated.

Although this principle is straightforward, in practice there are many complicating factors.

First, the half-life needs to be accurately known. This is a problem because in the early days of ^{14}C dating, the so-called Libby value of 5568 years has been used for the half life [1].

Second, it appears that the ^{14}C concentration of atmospheric CO_2 has not always been the same in the past. In tree rings, natural variations of the atmospheric $^{14}CO_2$ abundance were discovered on a time scale of one decade to a few centuries [9]. Later it was discovered that these variations can be attributed to variations in solar activity [10], which in turn influence the production of ^{14}C in the atmosphere. Also changes of the geomagnetic field strength influence the production of ^{14}C in the atmosphere [11]. This is understood because both solar activity and geomagnetic field strength determine the amount of cosmic radiation impinging on the earth, and thus the ^{14}C production rate in the atmosphere [12].

Third, there are mass dependent effects. This is called isotope fractionation. Biological, physical and chemical processes are usually mass dependent, which means they change the isotope concentration [13]. Translated in terms of ^{14}C, this changes the ^{14}C date.

Fourth, the accurate and precise measurement of ^{14}C is not straightforward. The AMS method is based on a particle accelerator, needed to separate the very small amount of ^{14}C from other isotopes, present in abundances larger by many orders of magnitude [14],[15].

Fifth, only the ^{14}C that was part of the organism when it died should be measured. Any foreign carbon that has entered the sample since that time is contamination and must be removed. A mixture of physical and chemical means does this pre-treatment. The pre-treatment also isolates a stable chemical fraction of a sample for dating—for example, cellulose for wood, or collagen for bone [16].

The solution to the complicating factors 1-3 mentioned above, is to define or standardize the ^{14}C measurements.

By definition, the ^{14}C timescale is expressed in BP = Before Present, where "Present" refers to the "standard year" 1950 AD [17]. Radiocarbon measurements are always measured with respect to a standard (=Oxalic Acid with a radioactivity of 0.226 Bq/gC) that corresponds to that year.

By convention, the original half-life (5568 years) used in the early days of the ^{14}C dating method [1] is used for this defined timescale.

Also by convention, the definition includes correction for mass dependent effects (fractionation). This fractionation can be determined by measuring the isotope ^{13}C content of the sample. The isotope ^{13}C is stable and thus not (as is ^{14}C) subject to decay. The fractionation is expressed as $\delta^{13}C$ in per mil deviation from the $^{13}C/^{12}C$ ratio from a standard [13]: $\delta^{13}C = [^{13}C/^{12}C]_{sample}/[^{13}C/^{12}C]_{standard}-1$. The standard value for $\delta^{13}C = -25‰$.

In summary, the ^{14}C timescale is *defined* and has to be *calibrated* to establish the relationship between ^{14}C time and historical time. Note that calibration takes into account not only the "wrong half-life" but also the natural ^{14}C variations. Both effects cause the ^{14}C clock to run at a varying pace, different from real clocks: ^{14}C time does not equal historical time. Calibration connects both independent clocks.

2. *Calibration*

Calibration involves measuring samples by both the ^{14}C method (in BP) and another method. Ideally this other method has to be independent from ^{14}C, yielding absolute dates (in AD/BC), and the samples have to be from the terrestrial (or atmospheric) reservoir. Calibrated ^{14}C dates are reported as calBC or calAD [18]. The unit calBP is used as well; this is defined as calendar years with respect to 1950 AD (calBP=1950-AD).

The most ideal samples for calibration are tree rings, because they can be dated absolutely by means of dendrochronology. Following the early work of Suess et al. [19], the ^{14}C community has issued special issues of the journal *Radiocarbon* with calibration curves based on dendrochronology. The latest and presently recommended calibration curve is Intcal04 [3]. The dendrochronological record now extends back to 12.400 years ago [20]. This calibration curve is shown in figure 30. The insert shows a detailed view if the wiggles for the fourth millennium BCE. These data are measured with the best possible precision [21]. The calibration curve has been extended beyond the tree ring limit by using marine data. Using these marine data, the Intcal04 curve

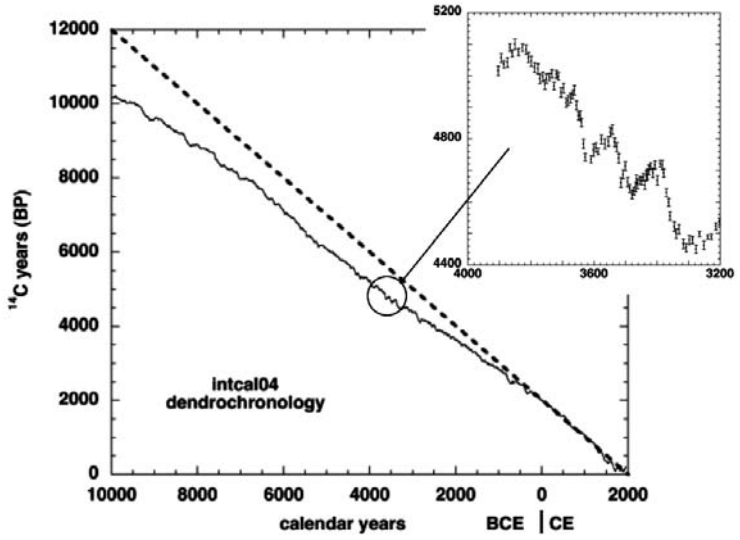

Figure 30. The ^{14}C calibration curve Intcal04, based on dendrochronological datasets. The insert shows wiggles in detail.

has been constructed [3]. It is the presently recommended calibration curve back to 26.000 years ago (26.000 calBP).

Because of the irregular shape of the calibration curve, the translation of a ^{14}C age (in BP) into a calendar age is not straightforward. Special calibration software has been developed, producing calibrated age ranges with 1σ or 2σ confidence intervals [22],[23],[24]. These programs (updated with the Intcal04 datasets) can be downloaded from www.radiocarbon.org.

An example calibration is shown in figure 31. It is the calibration of one of the Dead Sea Scrolls: 1QpHab (Habakuk *Pesher* from cave 1), as measured by the Arizona laboratory (AA-13417) [25]. The figure shows the relevant part of the calibration curve Intcal04, with the uncertainties (1σ). This part of Intcal04 is constructed through the dendrochronological datasets with a temporal resolution of 5 calendar years [3].

Figure 31 shows two probability distributions. First, along the vertical axis, the probability distribution corresponding to the measurement 2054±22 BP is plotted. This is a so-called Gaussian, which represents the probability distribution of data around the mean value. The deviations from the average value (in this case, 2254 BP) are given in terms of the standard deviation σ. The meaning of this term is that the probability of observing values between 2254+σ and 2254-σ is

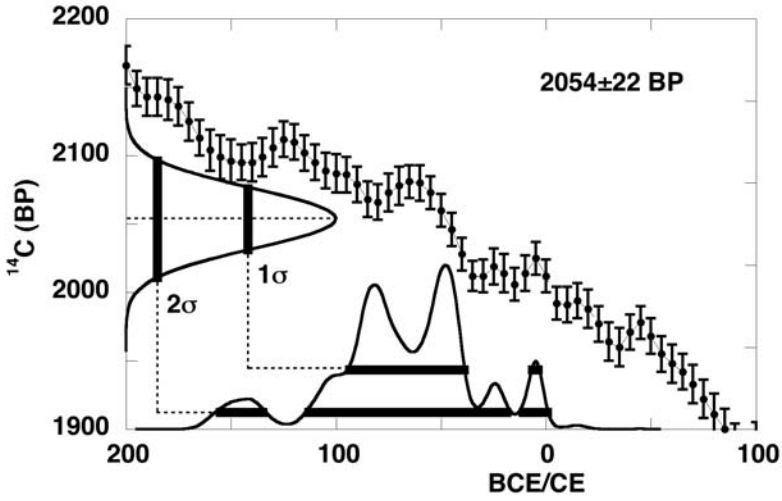

Figure 31. Calibration of the ^{14}C age 2054±22 BP, the published date for the scroll Habakuk (1QpHab)

68.3%, and between 2254+2σ and 2254-2σ is 95.4%. The 1σ and 2σ ranges for the Gaussian distribution are plotted as vertical bars in the figure.

Along the horizontal axis, the calibrated probability distribution is plotted. This distribution no longer has a Gaussian shape (it would be Gaussian only in the case that the calibration curve would be a straight line). As can be seen, the distribution has a complex shape due to the "wiggles" in the calibration curve. In theory, one ^{14}C date can correspond to several calendar ages. Computer programs are needed to calculate the errors in terms of confidence intervals. The program calculates the 1σ and 2σ confidence interval for the calibrated probability distribution, corresponding to 68.3 or 95.4% probability, respectively. These confidence levels are indicated as horizontal bars in the figure. This means that for these date ranges, the area under the probability curve is 68.3 or 95.4% of the total area, which corresponds to 100% probability.

For the ^{14}C date 2054±22 BP, the 1σ calibrated age range thus determined is 95-38 and 6-2 BCE; the 2σ calibrated age range is 157-133,114-16 BCE and 12 BCE-2 CE.

For completeness, we mention here that beyond 26.000 years ago, calibration datasets are available but they are not consistent.

Therefore, no calibration can be recommended for the time range 26-50.000 calBP. Hence the name "Notcal04" [26]. For a more updated discussion see [27].

3. *The measuring of ^{14}C*

The measuring process of the ^{14}C content of archaeological samples such as bone, charcoal, wood and so on, can be viewed from two perspectives: measuring technique and sample preparation. For ^{14}C, there are two methods for measuring ^{14}C: radiometry and mass spectrometry. The so-called conventional method is based on radiometry. This method requires large samples (around 1 gram of C) and is not further discussed here, as it is not relevant for Qumran. For detailed information we refer to [28] and [29]. The technique of AMS is based on mass spectrometry, for which method milligram size C is sufficient (e.g. [14],[2]).

Sample preparation follows similar procedures for both methods. The general rules are that contaminants have to be removed (physi-

cally and chemically) and that a reliable datable fraction needs to be isolated. Such contamination usually comes from the burial environment. Only the ^{14}C that was part of the organism when it died should be measured.

The commonly used chemical pre-treatment method for the samples is known as AAA (Acid-Alkali-Acid). The first Acid step is designed to remove soil carbonates and infiltrated humic or fulvic acids. Usually one uses HCl. The next step, Alkali, is performed with NaOH and removes soil humates. The final Acid step (again HCl) removes any CO_2 that is absorbed during the Alkali step.

The strength, temperature and duration of the Acid/Alkali treatments depend on the nature and quality of the sample material.

The AAA method is the standard, used for charcoal, charred material, wood, peat, and organic deposits. Details may differ depending on the sample material. A more or less complete overview of various recipes can be found in [8] and [16].

For fossil bone, the organic matrix collagen is isolated.

The sample pre-treatment chemistry provides quality parameters for the sample materials. A very important one is the organic carbon content (often denoted as C%) of the sample. For wood and peat, this must be in the range 45-55%; for charcoal, around 70%; for bone, 45-50 %.

When the organic carbon content is much lower, the material is degraded and the ^{14}C dates become less reliable.

Another quality parameter is the ^{13}C content of the sample. The $\delta^{13}C$ value is measured because it is needed for fractionation correction. But this $\delta^{13}C$ value also needs to be in a specific range for various materials. For (C3 plant type) charcoal, wood and peat this must be about –25‰; for bone, in the range –19 to –21‰. We note that for bone there are exceptions; the stable isotope content depends on the food intake of the organism—see, for example, [30].

In exceptional cases, the AAA pre-treatment method may not be completely sufficient to remove contaminants. For example, this is known to be the case for materials heavily impregnated with contaminants, or which are treated with conservation materials. It is possible to remove such contaminants by applying an extra pre-treatment step, using a so-called "soxhlet" extraction [31].

After proper pre-treatment and isolation of the datable fraction, the ^{14}C content of this fraction needs to be measured. The AMS requires solid graphite. This is produced by first combusting the datable mate-

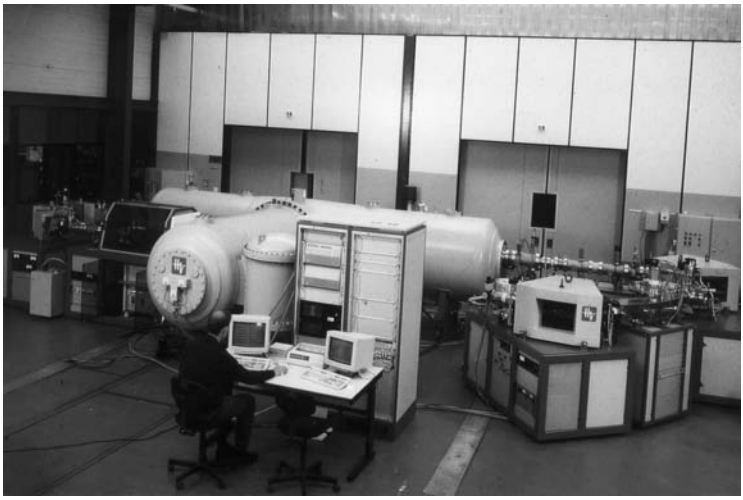

Figure 32. The Groningen AMS facility, dedicated for ^{14}C measurements

rial into pure CO_2. Next, this CO_2 is reduced to C powder (graphite); this graphite is pressed into sample holders (so-called targets). A carousel of 58 such targets can be loaded in the source of the AMS. The AMS facility of Groningen University is shown in figure 32.

In the ion source (on the left in figure 32), negatively charged C atoms are produced from the graphite. This beam of C^- ions is steered into the accelerator, which is the large T-shaped tank in the center of figure 32. A high voltage of 2.5 MV (Million Volt) accelerates the C particles to high energies. A set of magnets (on the right of figure 32) separates this high energy beam of C particles according to mass: 12, 13 and 14 for the isotopes ^{12}C, ^{13}C and ^{14}C, respectively. The ^{12}C and ^{13}C beams are measured by current meters, and from this the ratio $^{13}C/^{12}C$ is determined. This is used for the fractionation correction, necessary for the ^{14}C dates. A particle detector measures the ^{14}C counts, so that also the $^{14}C/^{12}C$ ratio can be determined. From all of this, the ^{14}C ages in BP are calculated. More technical details and status reports can be found in [32] and references therein.

4. *Quality control issues*

Radiocarbon (^{14}C) is the most common radiometric-dating tool applied in disciplines such as archaeology. Stringent quality control

(or "how good are ^{14}C dates") is required to build up reliable ^{14}C chronologies. Important aspects of quality control involve regular laboratory intercomparisons, multiple analyzes of selected samples, sample material selection, archaeological association, and sample size (i.e. conventional versus AMS as measuring technique) and contamination.

True point dates cannot be achieved with ^{14}C dating, as there will always be a standard deviation. Both equipment resolution and measurement stability, as well as the random nature of radioactive decay [8] causes the results of repeated measurements to spread around a 'true' value. The possible discrepancy between a measured value and the 'true' value is indicated by the standard deviation (σ). Multiple measurements will theoretically result in an average date that is both more accurate and precise than can be achieved with single measurements, provided that the ^{14}C laboratory does not have a systematic bias towards older or younger dates.

The quality of the BP date—i.e. the measured date, before calibration—forms always the basis for every radiocarbon age determination. It must be realized that a ^{14}C date *does* provide a very important universal physical measurement of time, independent of cultural-historical viewpoints and associative reasoning.

Sample selection is a critical component in the ^{14}C dating process. The layers from which archaeological or geological samples are taken during excavations have not always remained static and may have been affected by different kinds of post-depositional processes. Perturbation by plants, animals or human activities (e.g. digging) may cause migration or contamination of carbon in samples used for ^{14}C dating.

Another key question is the relationship between the age of the sample and the archaeological or historical question addressed: "how is the ^{14}C event related to the human event to be dated" [5]. A well-known problem in this respect is the so-called "old wood effect". Wood used (or re-used) to construct a building may have a ^{14}C date that differs from the human construction event by several centuries, depending on the age of the wood. It must be emphasized that the ^{14}C date of the wood (or charcoal) in such a case is not a measurement mistake. Rather the age of the wood sample is older than the age of the archaeological layer or building in which it was found.

The ^{14}C community has formulated general recommendations [33], which can be summarized as follows:

1. the sample needs to come from a closed archaeological context or secure stratigraphic layer
2. the sample must represent the event of archaeological interest
3. the sample needs to come from a context with artifacts pertaining to a specific cultural phase
4. the sample should not be contaminated
5. short lived samples are preferred for ^{14}C dating
6. more than one date per context or phase is recommended
7. the ^{14}C laboratory must adhere quality aspects as is common practice by the ^{14}C community (such as organic content and $\delta^{13}C$ of the sample)
8. a ^{14}C date can not be dissociated from the archaeological context; this means that statistics on sets of dates (like averaging) can only be applied to single archaeological contexts
9. the ^{14}C dates must be reported according to the convention—i.e. in BP, which is defined as measured relative to the oxalic acid standard, including correction for isotopic fractionation using $\delta^{13}C$ of the sample
10. the ^{14}C dates are to be calibrated using the most recent calibration curve (at present Intcal04), recommended by the ^{14}C community
11. calibrated dates are presented in calBC or calAD (or equivalents like calBP, BCE, CE)
12. upon publication, the archaeological context and the ^{14}C determination details need to be published
13. the ^{14}C laboratory must take part in the internationally organised intercomparison studies.

Another important matter related to sample selection is the respective choice of "conventional dating versus AMS". There can be a temptation to collect and submit all isolated seeds and tiny flecks of charcoal. The dating of such isolated samples by AMS should be discouraged, if larger samples (seed or charcoal clusters) are present in the same layer. If sufficient material is available, samples can be dated more cheaply and often more accurately by conventional means. The possibility of dating erratic post-depositional influences is considerable when isolated small fragments of charcoal or seeds are used, which are liable to movement by faunal or human digging activity. Such tiny samples have to be derived from a clearly defined context or association to justify dating. Lanting and van der Plicht [34] presented a detailed discussion about these issues, including examples. It is a "myth" that

AMS is better than conventional radiocarbon dating: standard deviations are usually not smaller.

Time-width effects represented by a sample have to be considered. Bulk samples of peat layers, for example, are centimetres thick for conventional ^{14}C analysis. Such a sample comprises many years of sedimentation or growth. Isolated seeds, macrofossils, and grains represent single-year samples and are typical AMS material, due to their small sample size, but the stratigraphic context must be clear, as noted above.

The correct calibration procedure of ^{14}C dates from multi-year or single-year samples needs to be carefully contemplated. Smoothed curves are recommended for multi-year samples, while single-year samples ought to be calibrated with the most detailed calibration curve available [8].

Intercomparison is a major part of quality assurance. By intercomparison is meant that different laboratories date the same samples. This may involve either samples of known age or blind samples. Thus a laboratory can "check" its performance—in particular, the sample (pre)treatment and ^{14}C measurement procedures. Intercomparison is a well-recognized issue in the ^{14}C community, and various exercises form a continuing process. The latest large-scale intercomparison is FIRI (Fourth International Radiocarbon Intercomparison) in which 84 laboratories participated worldwide. Several publications were generated by this program [35], and the final report is a special publication of the *Radiocarbon* journal [36].

This Fourth International Radiocarbon Intercomparison had aims of evaluating the comparability of routine analysis of both AMS and conventional laboratories, quantifying of the extent of and the sources for any variation, and investigating of the effects of sample size, precision and pre-treatment on the results.

The FIRI intercomparison results for the two Groningen laboratories (conventional and AMS) can be found in [6]. Other intercomparison measurements involving selected laboratories concern high precision measurements on dendrochronologically dated wood for calibration purposes [37], the development of working standards [38], and a presently ongoing Israel Iron Age project [39].

5. *Qumran: ^{14}C dating*

Thus far, there has been a minimal employment of ^{14}C dating of materials from the actual site of Qumran since the excavations in the 1950s. In the early days of Radiocarbon, the measurement errors were large, and there was no calibration into absolute ages. As it happens, one of the first ^{14}C dates in history was performed on a linen scroll wrapper, dated in 1949 by the pioneer Libby himself. The result was 1917±200, corresponding to 167 BC—233 AD or 33±200 AD [1]. This illustrates both the large measurement error, and the reporting relative to 1950, i.e. before the discovery of natural ^{14}C variations making calibration necessary. Also this was before the time of fractionation correction by ^{13}C measurements, and only large samples (grams of material) could be dated.

With the development of AMS, dating of small (milligram size) samples became possible. In the 1990s, fourteen Dead Sea texts were dated in Zürich [40], followed by another set dated in Tucson [25]. The dates suggest a possible range from the third century BC to the first century AD for texts from caves near Qumran, with a strong concentration of probable dates in the second or first century BC.

This set of ^{14}C dates obviously form a very important date list for our cultural heritage. Nevertheless, there are discussions concerning the dates as well. As an example, we mention a critique by comparing the ^{14}C dates with palaeography [41]. This critique, however, is based on a wrong understanding of concepts of measurement errors and calibration procedures, as is explained in a rebuttal by van der Plicht [42]. We mention this here as an illustration of discussions and misunderstandings between scholars representing humanities and sciences, going on to the present day. It illustrates the need for a multidisciplinary approach of research concerning Qumran and the Dead Sea Scrolls.

There is also a discussion concerning the original set of ^{14}C dates for the Dead Sea Scrolls in the scientific community. Rasmussen et al. [43] have shown that the use of castor oil by the original team of scroll readers may have contaminated some of the ^{14}C dated scrolls; contamination that could not be removed by the standard AAA pre-treatment procedures used prior to ^{14}C dating.

Also this "castor oil problem" pointed out by Rasmussen et al. [43] was criticized [44] and in turn rebutted [45].

It is necessary to state that the ^{14}C dating method is a trustful method. It is a scientific measurement of past time, independent of other dating methods like palaeography, pottery assemblages or (pre/proto) historic data. When two independent dating methods produce different results, then at least one of them must be wrong. It is of course possible for a ^{14}C date to be wrong, or better: not yield the expected result. There can be many reasons for this. One of the most common reasons is: contamination with foreign material (but see also the list in the paragraph on quality control).

The castor oil question is an example of this. The ^{14}C content of the samples was undoubtedly measured correctly by the two laboratories involved, following standard procedures. In the event a sample was contaminated with castor oil, the date would be too young because the standard sample pre-treatment could not remove this. We note that in the ^{14}C dating community, this is a very exceptional situation. Tens of thousands of dates are produced annually by the laboratories, continuously checking themselves through programs like intercomparisons, and castor-oil like problems only happen when materials are treated with preservatives—like the Dead Sea Scrolls and samples from museum collections. These are exceptional cases, and usually require additional chemical treatment to remove the humanly applied foreign carbon.

For the Qumran samples to be dated by ^{14}C, a collaboration was started between the Universities of Southern Denmark and Groningen. In Odense (laboratory code KLR), the extra chemical pre-treatment (soxhlet and others) and contamination tests were applied to sample materials. In Groningen (laboratory code GrA), these samples were consequently treated by the standard AAA method and dated by AMS.

A battery of ^{14}C dates of wood, linen and parchment excavated during the 1950s at Khirbet Qumran and in the caves at Qumran is obtained. We distinguish dates of (charred) wood or seeds, linen and textile, and scrolls/parchment. We will emphasize here mainly the castor oil tests of scrolls and parchments, and review here only a few results on other materials dated. A full report on contamination tests and decontamination experiments will be published shortly [46].

5.1. *(Charred) wood and seeds*

Radiocarbon dates for wood samples obtained thus far are summarized in Table 1. Dates were obtained for date stones (KLR-2610a2 and

2612); the latter one was charred. A sample of wood (KLR-2611) was contaminated with paraffin preservative, which was removed by pre-cleaning with soxhlet extraction, prior to standard AAA treatment.

A special find at Qumran, known as Jar-35 has been investigated thoroughly by various archaeometric means. Both TL and 14C date it. A full report will be published [47]; only the ^{14}C dates are shown here. Three samples of charcoal from inside the jar were dated. They all produce the same date within error; the weighted average for the dates is 2035±25 BP, which calibrates into 50-1 BC (1-sigma).

Table 1. ^{14}C dates for samples of (charred) wood and seeds.

Sample	excavation	KLR	GrA	^{14}C date (BP)	$\delta^{13}C$ (‰)	%C	calibrated age
date stone	KhQ-519	2610a2	17393	1955±40	-23.29	63.9	5-85 AD
wood	T-18	2611	17394	1970±40	-23.13	52.1	20 BC-75 AD
charred date	KhQ-519	2612	17395	1955±40	-23.20	65.6	5-85 AD
JAR 35		6624	33950	2060±30	-24.52	62.0	
JAR 35		6624	34165	1965±35	-25.71	60.9	
JAR 35		6624	34170	2075±35	-24.10	61.6	
JAR 35			averaged	2035±25			50-1 BC

The ^{14}C dates themselves cannot be used as an indicator of the reliability of the dates. But the carbon content (%C) and stable isotope values ($\delta^{13}C$) can, because these values lie in certain ranges independent of age. The can also be used as an indicator of contamination.

For example, a wood sample QUM-515 (not shown in table 1) KLR-3327 (GrA-17412) is considered suspicious in terms of ^{14}C dating. The $\delta^{13}C$ value for this sample which is very deviating: $\delta^{13}C$=-10.62‰. For reasons not discussed here, we think that this sample is probably contaminated. However, a C4 plant which has a different photosynthesis pathway yielding $\delta^{13}C$ values in the observed range can not be excluded at this stage.

5.2. *Linen/textiles*

The $\delta^{13}C$ values for linen and cotton samples, which are manufactured from plant fibers, are expected to fall in the same range as wood

samples, i.e. around –25‰. Samples made from animal tissue, such as wool, have $\delta^{13}C$ values around –21‰.

For samples characterized as "textile" there remains uncertainty since it is not known if the sample consists of plant fibers (cotton) or is made from animal tissue (wool).

Our Radiocarbon date list includes a piece of textile fabric stored in the Palestine Exploration Fund collection: GrA-24262 and 25588 (KLR-5466). This is a duplicate date of linen from a sample known as sample AF-25. The average of these 2 dates is 1985±30 BP, which calibrates into 40 BC-55 AD (1-sigma). Before dating, the sample was subjected to soxhlet pre-cleaning in Odense. In Groningen, the sample underwent standard AAA treatment and was dated by AMS. The results are shown in Table 2.

For a full discussion of archaeological implications of the linen date shown here we refer to [48].

Table 2. ^{14}C dates for samples of linen and textile.

Sample	material	KLR	GrA	^{14}C date (BP)	$\delta^{13}C$ (‰)	%C	calibrated age
AF-23	linen	5466	24262	1995±40	–24.10	40.8	
AF-23	linen	5466	25588	1975±35	–24.45	36.6	
			averaged	1985±30			5-55 AD

The $\delta^{13}C$ and C% sample quality data for these samples are within normal range.

5.3. *Bones*

A few samples of fossil human bone from the Qumran cemetery were submitted to Groningen for dating. For bone, the datable fraction is collagen. The bones were very fragile, and for that reason they dissolved immediately at the start of the chemical treatment. No collagen could be produced from Qumran bone material thus far.

Bones and textiles from the graves (as well as other materials from the desert) are very fragile, which is a good reason to apply preservatives for conservation purposes. Obviously this causes contamination for ^{14}C dating; it requires the materials to be exposed to additional pre-cleaning before the standard AAA treatment used for ^{14}C dating. This can be problematic for fragile materials because they can be destroyed

during the treatment before the desired datable fraction could be extracted.

We have performed extensive testing of samples in this respect. One series was conducted on samples treated only by the standard AAA procedure. In the other series, samples were pre-cleaned prior to AAA and ^{14}C dating. A detailed datelist will be published shortly [46]. Only a few results are shown here (tables 1 and 2).

Based on our results, we recommend that samples from Khirbet Qumran and the surrounding caves residing in museums should be analyzed and cleaned prior to AAA pre-treatment and ^{14}C dating.

Our findings also apply more in general to ^{14}C samples from museum objects, and are not limited to Qumran.

5.4. *Scrolls and parchment*

It is feasible that castor oil and similar components could lead to a reaction with the proteins of the parchment. If this indeed takes place, modern carbon atoms originating from the oil would be fixated to the parchment, and thus constitute a serious obstacle to any attempt at de-contaminating samples of castor oil polluted parchment prior to ^{14}C dating.

It is known that the original team of editors of the Dead Sea scrolls, both to clean the texts and to make the readings more clear, used castor oil extensively.

As far as castor oil is concerned, it is essential to determine whether the standard AAA pre-treatment procedure is capable of removing all traces of this specific contaminant from manuscript fragments.

Contamination of a sample with an oil derived from a modern natural plant (such as castor), if not removed by the pre-treatment, would give a younger age than the true age. Fossil oils (petroleum products from the oil industry) would give an older age than the true age. The effect is about 100 ^{14}C years for a contaminant 1% in weight.

We conducted experiments by contaminating pieces of French medieval parchment with oil, both modern and fossil. The results are shown in Table 3.

The untreated samples yield ^{14}C ages corresponding to the age of the parchment. The effect of oil contamination is obvious.

From these results we calculated how much of the oil was removed by the AAA treatment. The conclusion from this experiment is that the ^{14}C age offset is 2-3 centuries, for samples fully saturated with oil.

Also pure oils were dated. Old (fossil) oil is infinitely old on the ^{14}C timescale; indeed the ^{14}C concentration could not be distinguished from the background, which corresponds to a ^{14}C age of about 45.000 BP (GrA-14051). Modern oil indeed has a ^{14}C activity of 111.5%, which means it dates from after 1950.
For more details and calculations, we refer to [43].

Table 3. ^{14}C dates for scrolls and parchment testing the effects of castor oil

KLR	GrA	treatment	weight natural (mg)	weight with oil (mg)	weight after AAA	^{14}C date (BP)	$\delta^{13}C$ (‰)	%C
1894	13929	untreated	27.0	27.0	22.4	750±40	–22.43	46.7
1895	13930	old oil	24.7	31.5	22.8	2030±40	–23.10	49.8
1896	13931	modern oil	27.9	36.5	25.9	540±40	–23.48	49.6
2315	14044	old oil	31.7	38.2	27.3	1670±45	–23.04	46.1
2316	14043	new oil	24.9	32.7	23.3	475±45	–23.53	45.8
2317	14042	untreated	23.9	23.9	19.3	770±45	–22.61	43.7
2318	14038	old oil	24.0	30.3	17.9	1670±45	–22.84	44.4
2323	14051	pure old oil	–	–	–	>45000	–27.00	–
2324	14052	pure modern oil	–	–	–	111.5±0.6%	–28.80	–

After these initial experiments, we investigated various methods to remove contaminants (in particular castor oil) prior to the standard AAA pre-treatment.

Experiments for decontamination of the scroll materials by cleaning using this *soxhlet* extraction method, or by other techniques such as ultrasound cleaning and supercritical CO_2 cleaning, have proven to be successful [49]. The *soxhlet* method was chosen to be the best decontamination method; the others had a lower success rate, because of the fragile nature of the samples.

We have succeeded in devising a pre-cleaning strategy using soxhlet extraction that allowed complete removal of castor oil and linolic oil from both medieval parchment (KLR-6850 to 6854) and for samples of un-inscribed Dead Sea scrolls (KLR 7080 and 7081). These measurements, together with other pre-cleaning test results are shown in Table 4 [49].

Table 4. ^{14}C dates for scrolls and parchment after soxhlet cleaning of the contamination with oil

KLR	GrA	contamination	^{14}C date (BP)	$\delta^{13}C$ (‰)	%C
6850	37802	none	795±30	–22.14	47.5
6851	37803	castor oil	795±30	–22.02	47.6
6852	37897	linoleic oil	785±30	–22.21	47.9
6853	37898	castor oil	795±30	–22.03	47.6
6854	37899	linoleic oil	(failed)	–21.98	47.4
7080	39727	none	2120±70	–18.47	41.7
7081	39728	castor oil	2200±70	–18.11	39.5

The samples were cleaned using soxhlet with 1 hour in ethanol, 4 hours in hexane, and again 1 hour in ethanol.

The sample KLR-6854 was lost in the process due to a technical problem. The dates of the other four samples are (almost) identical, showing that the cleaning was completely effective.

Two samples of un-inscribed Dead Sea Scroll were tested; one was left untreated (KLR-7080), whereas the other was contaminated with castor oil (KLR-7081).

The samples were quite small, which explains the relatively large uncertainty (70 BP).

The results from Table 4 can be seen as "the ultimate test" for the castor oil (de)contamination experiment. The decontamination method we developed proved to be effective in removing castor oil from Dead Sea Scroll parchments, and is mechanically subtle enough not to damage the fragile material during the cleaning process.

We are in the process of selecting Dead Sea Scrolls, previously dated by ^{14}C in either Tucson or Zürich, which would be candidates for re-dating. This concerns, among others, 4Q258 Community Rule (d), 4Q266 Damascus Document (a), 4Q171 Pesher Psalms (a), 4Q521 Messianic Apocalypse (see Plate XI), Xhev/Se 8a Kefar Bebayou/Kefar Baru, and 1QH Thanksgiving Hymns (a).

We propose to re-date these Dead Sea Scrolls, after pre-cleaning with the soxhlet procedure we developed for this purpose.

6. *Conclusions*

Since its conception around 1950, Radiocarbon (^{14}C) is developed into a well-established and reliable dating method. Calibration of the ^{14}C timescale enables absolute dating and therefore provides a scientific measure of past time.

The AMS method enables the dating of small (milligram size) samples, opening new horizons since the 1980s.

The application of the ^{14}C dating method can be limited by the temporal resolution, in particular in (proto)historic periods. It depends on the exact shape of the calibration curve during the relevant time frame, and may vary from a few decades to centuries.

In the context of Qumran, ^{14}C samples are fragile and therefore treated by preservatives or oil. This is a contamination that is difficult to remove from the sampled materials using chemical treatment protocols, which are standard for the ^{14}C method. This includes samples of the Dead Sea Scrolls, dated by ^{14}C almost two decades ago. Some of these scrolls were potentially contaminated by castor oil, which had been applied during the 1950s. Since this castor oil is not removed effectively by the standard chemical pre-treatment, some ^{14}C dates are affected and possibly too young.

We have tested pre-cleaning techniques, of which the so-called soxhlet extraction was selected for use. This pre-cleaning should be applied before the standard ^{14}C procedures are used. By performing a variety of experiments, we have shown that parchments, heavily contaminated with oil, could be cleaned effectively.

Therefore, we can now re-date by ^{14}C those Dead Sea Scrolls, which were contaminated reliably.

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CHAPTER NINE

CHARACTERIZATION OF THE WRITING MEDIA OF THE DEAD SEA SCROLLS

Ira RABIN[a], Oliver HAHN[a], Timo WOLFF[a,b], Emanuel KINDZORRA[a,b], Admir MASIC[c], Ulrich SCHADE[d], Gisela WEINBERG[e]

[a]Federal Institute for Materials Research and Testing, 12200 Berlin, Germany, [b]Technical University of Berlin, 10623 Berlin, Germany, [c]Max Planck Institute of Colloids and Interfaces, 14424 Potsdam, Germany, [d]BESSY, Berlin, Germany, [e]Fritz-Haber-Institut of the MPG, 14195 Berlin, Germany

Abstract. Our study is dedicated to the development of the methodology for an accurate characterization of the support and the inks of the Dead Sea Scrolls. To that aim we use optical and electron microscopy. micro-XRF, 3D-SY-XRF, different IR methods including synchrotron radiation based reflectance spectroscopy. Simulation experiments to identify different water sources and binding agents in the carbon inks are presented.

Keywords. Dead Sea Scrolls, parchment, carbon ink, XRF, FTIR, SEM

Introduction

Material study of the Dead Sea Scrolls has accompanied the work on the texts since the first days after the discovery of the scrolls. Questions on the identification of the support material and the first attempts of establishing its age date to the late fifties and early sixties [1-5]. J. Poole and R. Reed's comprehensive work revealed that the parchment production process was different from that known since the Middle Age, namely they discovered that tannins were applied to the parchment surface at the stage of finishing. In the same study the authors pointed out that the distribution of the elements in the composition of the parchment was highly inhomogeneous and, thus, incompatible with a single place of production. They also investigated the shrinkage temperature, a property reflecting the gelatinization degree of collagenous material. If gelatinization is the main effect of aging, the shrinking

temperature is a measure of the age of a material. Indeed, the shrinkage temperature for the majority of the samples they analyzed fell within a narrow band. In his groundbreaking study in 1980, S. Weiner employed X-ray diffraction to determine the ratio of collagen to gelatin in a number of scrolls. His results confirmed the assumption of Poole and Reed [6]. Moreover, the use X-ray diffraction has allowed a better understanding of the degradation process in parchment and has become a routine application today [7]. Both methods are also well suited for a quick determination of forgery.

Radiocarbon dating of the scrolls conducted in the 90's corroborated largely the dating of the scrolls based on the paleography [8,9]. However, some reservations towards the results obtained by this method were voiced recently. It is known that some of the scrolls were treated with castor oil to enhance text legibility. Usual sample cleaning procedure does not remove castor oil completely, resulting in a younger age for the material tested [10]. A new cleaning procedure has been developed to solve this problem [11]. Another solution could be the use of a sensitive and non-destructive method able to identify foreign organic materials, to identify the samples suitable for radiocarbon dating [12].

Further historical queries that might be solved with the help of the material study include the question of the origin of the scrolls [13], of their archaeological provenance [14] and of the correct attribution of the fragments to one scroll [15]. In this contribution we summarize the main principles of our approach and illustrate it with examples.

Principles of the approach

Our approach is based on the recognition of various traces accumulated on a fragment in the course of its history. Spatially resolved measurements of elemental composition with subsequent chemical analysis by means of vibration spectroscopy allows determination of characteristic patterns, the so-called fingerprints. The latter can be then correlated with a distinct event in the life span of the fragment. The cross section sketch in figure 33 presents schematically the main periods of discernible trace accumulation as spatially separated layers: production of parchment, inscription, use, storage in the caves, post discovery treatments. In reality the layers are not separated. However, intermixing layers present a problem only in the rare cases of com-

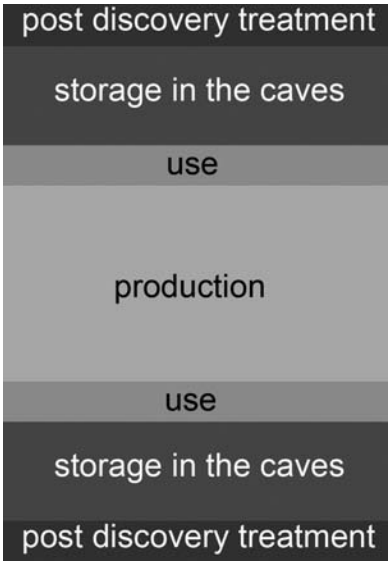

Figure 33. Cross-sectional scheme of a fragment with traces from different periods in its history.

plete gelatinization of parchment accompanied by full dispersion of the ink in the material.

From the outer, more recent layers, one encounters traces of post discovery events, like humidification, treatment with oils, adhesives, consolidants. Storage in the caves is characterized by deposits rich in minerals. A prolonged use of the scrolls in Khirbet Qumran is associated with a characteristic air composition of this area. The water used during the processing of the hides might have a fingerprint of the source.

Both parchment and ink preparation involve handling with water [4, 16]. After drying and finishing stages parchment has a homogeneous distribution of the elements that were present as impurities in the water. Carbon ink produced from soot is handled with water twice. First, during the production of the dry inks, soot is mixed with a binder dissolved in a small amount of water. The mixture is dried and pressed into pellets or bars. Then, the the liquid ink is prepared directly before writing adding a bigger quantity of water. Thus, the resulting distribution of the impurities will be largely representative of the water used at the location of writing. Spring water of the coastal region of the

Dead Sea has a very specific composition similar to that of the Dead Sea itself [17,18], with a strikingly low ratio of chlorine to bromine as compared to any other water source throughout the country. The composition did not change significantly since the first measurements by Gmelin in 1827 [19]. Based on the chemical analysis of the aquifer and of the water sources in the area of ancient Israel conducted in the last 70 years, we can safely assume that also in the antiquity the composition of the waters of the coastal area differed greatly from that of the rest of the country [20,21].

Methods

Optical microscopy was performed with Ascania SMT4 which allows 8—100 times magnification. It is equipped with an x-y stage used for fragment mapping and the determination of the areas of interest.

X-ray Fluorescence (XRF)

Profile scans were carried out with the mobile energy dispersive micro-X-ray spectrometer ArtTAX® (Bruker AXS Microanalysis GmbH, formerly Röntec-GmbH, Berlin, Germany), which consists of an air-cooled low-power X-ray tube, poly-capillary X-ray optics (measuring spot size 70 μm diameter), an electro-thermally cooled Xflash detector, and a CCD camera for sample positioning. Furthermore, open helium purging in the excitation and detection paths allowed for detection of light elements ($z \geq 11$). All measurements were made using a 30 W low-power Mo tube, operated at 50 kV and 600 μA, and with an acquisition time of 70 s (live time).

The quantification of the XRF-results is based on the fundamental parameter method. This procedure requires an accurate knowledge of the excitation spectrum. The tube spectra were calculated with the expressions of Ebel [22]; the transmission of the polycapillary lens was determined performing an extended calibration procedure with thin, one element standards. For the determination of the total mass deposition of the samples we used the Compton scattering peak of the characteristic Mo-K-alpha-line of the tube.

Fourier-Transform Infra Red microscope (FTIR)

For acquiring transmission FT-IR spectra, micro samples were prepared in a Diamond micro compression cell and measured at a room temperature. A 15x IR objective with an aperture of 50 μm × 50 μm was used to focus the beam on the sample. The measurements were performed using infrared synchrotron radiation of the BESSY storage ring (IRIS line) and a FT-IR microscope (Nicolet) equipped with a liquid nitrogen cooled MCT detector. A total of 256 scans were co-added per sample spectrum (wave number range: 4,000–700 cm^{-1}).

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM): FEI Quanta 200 FEG, EDX system: EDAX Genesis 4000 with Si(Li) detector, type Sapphire. The measurements were carried out at accelerating voltage of 20kV, pressure of 1.1kPa in H_2O vapor, Secondary Electron (SE) mode plus Back Scattered Electrons.

1. *Results and discussion*

Characterization of parchment

Historical parchment is highly inhomogeneous because collagen degradation can be limited to extremely small spots neighboring with the regions of intact material. Dead Sea Scroll parchment exceeds in this respect any other known historical parchment and can be described as a "patch work" of different traces. To reliably identify and attribute the traces we employ methods with an interaction window of 10 to 70 μm. To avoid unrepresentative sampling, a usual problem associated with "micro" methods, we are developing a methodology to obtain a representative chemical map of a scroll fragment. Characterization of the parchment starts with a visual examination and a study of the surface to determine the areas of a similarity. In this respect hyper-spectral imaging has shown the most promising results for it allows a fairly high-resolution measurement of the spectral response of areas as large as A4 sheet [23]. Once the areas of similarity are established, we generate an x,y maps with the help of the optical microcopy to allow accessing the same point by different methods such as micro X-ray fluorescence, scanning electron microscopy and various techniques of vibration spectroscopy. An example of the

border region between the area of the mineral deposits and collagenous material is shown in figure 34a. The uninscribed fragment under study belongs to the collection of Ronald Reed. It has been reported to contain more calcium than any other in the collection of approximately 100 pieces [3]. Rich mineral deposits have been found on the surface of this fragment from Cave 4 of Qumran. Fortunately, the deposits are localized at the surface: the study of the cross sections conducted with micro X-ray diffraction has shown no penetration of the minerals from the surface into the bulk region [24]. This is corroborated by our study of calcium profile along the cross sections of the fragments from Cave 4 [25]. Element profiles across the border region measured with micro XRF are displayed in figure 34b. Concentration of the elements Si, Ca, K, Fe and Sr change at the border whereas bromine remains constant throughout the whole region. The variation of the chlorine concentration does not correlate with the crossover. The EDX spectra of both regions complement the micro-XRF analysis: in the spectrum on the left side of figure 34c the ratio of carbon and oxygen as well as the large Si and Ca peaks reflect the dominating presence of calcite and soil (earth) on the surface. On the right side of the same figure, the fibrous structure of parchment is clearly seen and the ratio of carbon to oxygen in the EDX spectrum point to the presence of a protein. Constant concentration of bromine throughout the scan (Figure 34b) is due to the fact that bromine salts do not crystallize and, therefore, are not found among the mineral deposits. Since it is very unlikely that fragments could survive a prolonged contact with water after parchment has been inscribed, the hypothesis of a homogeneous bromine distribution originating from the storage in the caves can be safely ruled out [26]. Thus, bromine originates either from the water used to process the skin or due to the penetration from the air. In the case of chlorine, mineral deposits cannot be excluded from the possible sources; therefore its variation must be studied in greater detail for a correlation with past events. In other words, despite the fact that in the example below no chlorine salts were found within the region sampled, the variation of the chlorine concentration suggests more than one source. It is clear that a single sample delivers insufficient information with respect to the distribution of the chlorine in the fragment material.

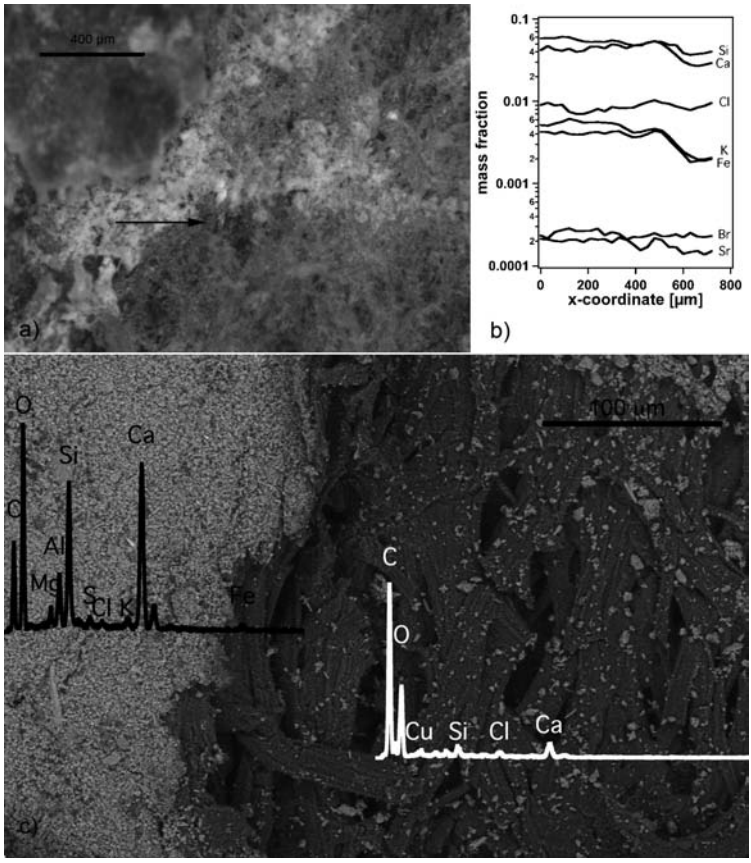

Figure 34. Surface characterization of a Dead Sea Scrolls fragment from Cave 4: a) optical microscopic image of the transition from an area rich with mineral deposits to the area where fibers are exposed; b) element profiles across this border measured by X-ray fluorescence; c) scanning electron micrograph of the border regions and the corresponding EDX spectra (color photo in Figure IX)

Characterization of ink

To evaluate our ability to differentiate between the composition of water in the ink and parchment we conducted simulation experiments with various inks and water from different sources. Parchment used in our experiments ranged from a modern goat parchment with a low calcium concentration to the Dead Sea Scroll parchment from the collection of Manchester University Library [27]. The carbon inks were either prepared according to the basic recipe (soot + binding agent

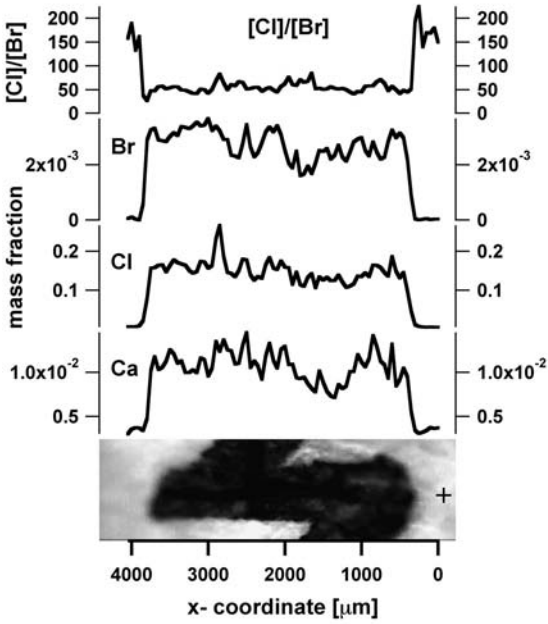

Figure 35. Characterization of the inscription by elemental composition. [Cl]/[Br] ratio and concentration profiles of the elements Br, Cl and Ca are recorded along x coordinate shown at the bottom together with the inscription. The position of the first point is indicated by the black cross.

dissolved in water, then pressed into pellets and dried) or purchased and their XRF and FTIR spectra measured.

Dry inks were mixed with a small amount of water of known composition to produce liquid inks. After the inscribing them, parchment samples were allowed to dry before the next set of measurements was made. Samples prepared with inks mixed with distilled water in the first stage, second stage and both stages were used as a reference.

In the example shown in figure 36, a piece of non-limed goat skin parchment was inscribed with commercial dry Chinese ink dissolved in water of known chemical composition.

The concentration of bromine, chlorine and calcium rise steeply at the border of the inscribed line. Their concentration remains constant throughout the inscribed region and falls steeply again once blank parchment is reached. The average [Cl]/[Br] ratio in the inked area amounts to 54 (upper curve in figure 35) as compared to the value of 52 of the water used to produce liquid ink from the dry one. This result

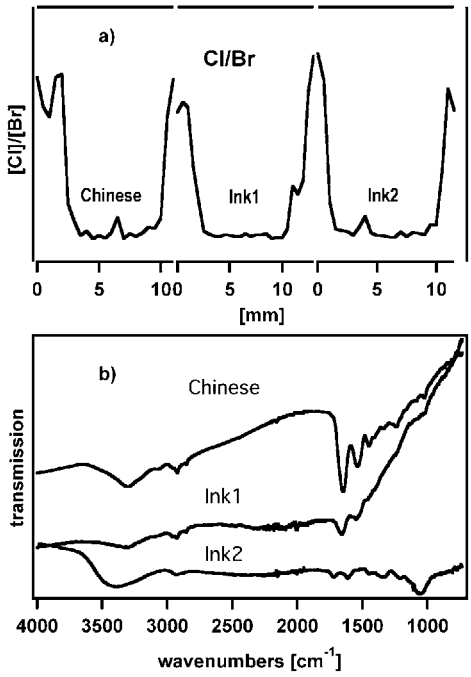

Figure 36. a) Comparison of the Cl/Br ratio measured in the inscriptions made with different type of dry inks. The inks were mixed with water from the same source prior to writing; b) FTIR spectra of the inks in the inscriptions.

shows that in this idealized case measurement of the chlorine and bromine concentration allows to characterize the water source for the inks. The difference between the chemical composition of a usual water source and the ones along the Dead Sea coast is so large that we found no difficulties in differentiating between them. Figure 36a shows comparison of the [Cl]/[Br] for three different dry inks mixed with the water from the same source. In this experiment in addition to a commercial Chinese ink of unknown composition we used self made dry inks. To prepare the ink designated Ink1 we have taken a gum from the local acacia Raddiana, known to belong to the flora of the Dead Sea region in the antiquity. The properties of its water-soluble gum are similar to that of the more popular brands, Acacia Senegal and Acacia Seyal, commonly knows as gum Arabic [28,29]. Gum of acacia Raddiana is still used by Bedouins as a food and medicine additive, similarly to the way gum Arabic is commonly used. Ink2 contains commercial gum Arabic as a binding agent and an extract of oak galls

as recommended in one of the recipes. The FTIR spectra of the inks are shown in figure 36b. In the fingerprint region between 1000 and 2000 cm^{-1} each spectrum has features characteristic of the organic binder present in the ink, e.g. gum Arabic in the Ink2 can be easily recognized by its prominent broad peak at 1043 cm^{-1}.

Hydrolyzable tannins from the oak gall in the same ink manifest themselves through the series of the peaks between C—O stretch 1190 cm^{-1} and C==O stretch at 1715 cm^{-1}[30-32]. Bands corresponding to the condensed tannins of acacia Raddiana (around 1000 cm-1) couldn't be resolved in this sample (middle curve I figure 36b). The binding agent of the Chinese ink (upper curve of figure 36b) could be clearly identified as animal glue due to the characteristic pattern of the amide bands of the collagen.

One of the most important results of our study of inks with the help of "dummy" samples is the determination of the most reliable spectroscopic method for identification of the binders used in the inks. A detailed comparison exceeds the scope of this contribution. However, we would like to mention that a combination of Raman spectroscopy and micro-ATR seems most promising in terms of the maximal identification power and minimal danger to the object.

Conclusions

Provenance study of the Dead Sea Scrolls is based on the analysis of parchment and reconstruction of its history. For this purpose we are developing a methodology for an accurate characterization of this highly inhomogeneous material. Furthermore, we presented simulation experiments for the identification of the binder in carbon inks as well as the sources of water used for preparation of the liquid ink from its dry precursor.

Acknowledgements

We are grateful to M. Broshi, G. Brooke, E. Tov, M. Binetti, L. Halicz, I. Gavrieli, P. Fratzl for discussions and suggestions of the relevant literature, to B. Gillo for the help with the studies of Acacia Raddiana and to R. Franke for his help in preparation of the samples. The research project is funded by the Stiftung Preussischer Kulturbesitz.

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CHAPTER TEN

ON THE AGE OF JAR-35

Kaare L. RASMUSSEN[a], Jan GUNNEWEG[b],
Johannes van der PLICHT[c,d], and Marta BALLA[e]
[a]Institute of Physics and Chemistry, University of Southern Denmark, Campusvej 55, DK-5230 Odense M, Denmark, [b]Hebrew University, Jerusalem, Israel, [c]Centrum voor Isotopen Onderzoek, Nijenborgh, 9747 AG Groningen, the Netherlands, [d]also at the Faculty of Archaeology, Leiden University, Leiden, the Netherlands, [e]Institute of Nuclear Techniques, Budapest University, H-1521 Budapest, Hungary

Abstract. A unique find in 2004 of an intact and sealed storage jar on the plateau south of the Qumran settlement provided a rare opportunity to make archaeometric analyzes that would help reveal the time and last use of the jar. In 2006 Buti *et al.* reported results showing the identification of tartaric acid, which indicated the past presence of wine or vinegar in the jar. In the present work we present an overview of our attempts to date samples of charcoal in the deposit by radiocarbon dating and the ceramic fabric of the jar by thermoluminescence dating.

Keywords. Radiocarbon dating, Thermoluminescence dating, Jar-35, Qumran.

Introduction

On the Southern plateau of Qumran no trace of buildings or other habitation has been identified (Figure 37 and the updated Map 2). It has been suggested that in the main period of habitation the plateau was used for agriculture (R.R. Cargill 2007).

On 2nd of August 2004 Randall Price and Oren Gutfeld unearthed a completely intact ovoid jar with two loop handles. Nearby was found two small hoards of animal bones and ceramic shards. The intact jar, named Jar-35, was intact and sealed with an overturned bowl (Figure 38). When the lid was lifted and a camera lowered into the interior, a deposit was discovered lining the bottom. In several places the deposit was mixed with ceramic flakes from the interior surface of the jar

(Figure 39). The report on the excavation is, unfortunately, still unpublished.

Hindsight is of course an easy position, particularly when an excavation is done by humanists and the post treatment and analyzes are performed by scientists. This caveat in mind, the focus of this workshop is *a holistic view on Qumran*, and in this light it would have been optimal if the excavation team had stopped immediately when they had excavated Jar-35, and not removed the lid in the field. A headspace GC analysis of the original air inside the jar would have been very interesting and potentially rewarding. Also, had the jar been opened in a laboratory clean room the ancient inventory of dust could have been secured, later to be studied by SEM imaging and various other forms of focused beam analyzes, *e.g.* SEM-EDX for major elements, ion beam analysis for isotopic ratios, and synchrotron beam analysis for structure. Such studies could potentially have provided important and unique information about the habitation in Qumran and dust studies could have provided basis for interpretation of the Dead Sea Scrolls themselves (see *e.g.* Rasmussen 2008). Of course we can still extract ancient particles from the jar even though it was opened in the field, but a serious suspicion now lingers whether a particular particle extracted from the jar's interior is indeed ancient or if it could be modern.

It must be emphasized that coordination between the team of excavators and the team of scientists who are to perform the scientific analyzes is not very common. One such coordination was practised with considerable success before, during and after the excavation of an 11th century site at Viborg Søndersø in Denmark (Iversen *et al.* 2005). This excavation was also done as a *context-excavation*, where each archaeological context was dug individually and an appreciable amount of the soil was stored in a warehouse in 10 L plastic buckets, which were later accessible to the scientists if they needed extra material, adjacent soil samples *etc*. However, one could argue that excavations at important sites of World Heritage status such as Qumran would be important enough for such coordination to be mandatory. If a holistic view on Qumran is truly to be obtained, it would be highly advantageous that future excavations are coordinated between the excavators and scientists of all conceivable fields which could have an impact on the interpretation of the events that took place in biblical times—both before, during, and after the excavation.

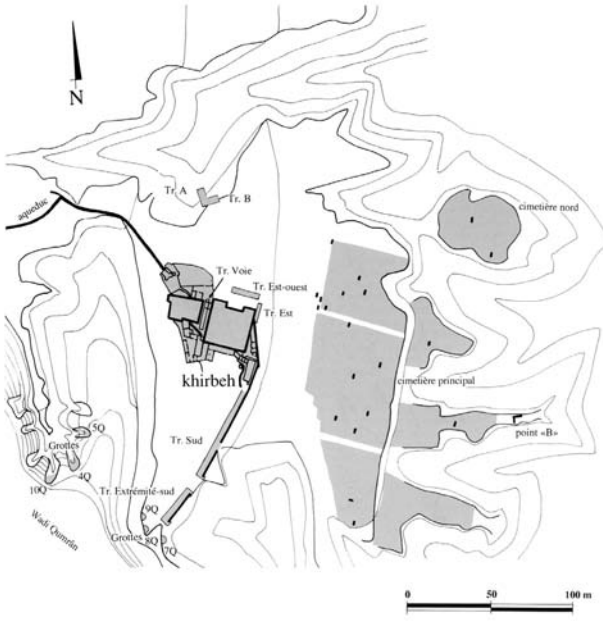

Figure 37. Situation plan of the Qumran settlement (from Figure II, Humbert-Gunneweg 2003). The position of the find of Jar-35 is indicated with an arrow (see Price/Gutfeld in Map 2).

Still, the finding of an intact and sealed storage jar is an extremely rare event, and even if the lid was removed in the field, it is possible to make analyzes pertinent to its last content and an estimate of the age of the jar. In this way one may retrieve rare insight about the use of an individual jar. Together with the rather odd position in which the jar was found, on the middle of the Southern plateau, this fact calls for a thorough scientific investigation. In 2006, Buti *et al.* reported a study of the deposit using the techniques of capillary electrophoresis (CE) and high-pressure liquid chromatography (HPLC) showing that it contained a small but unquantified amount of tartaric acid, and they therefore concluded that the jar had contained wine. In a study under way (Rasmussen *et al.* submitted), the deposit were subject to an investigation where a different conclusion is reached. However, in the present presentation we will focus on the issue of the date of the jar.

Figure 38. Jar-35 during the excavation in 2004—here still *in situ* with the bowl removed.

Figure 39. Samples of the deposit found inside Jar-35. *In many instances small flakes of ceramic material of the jar's interior surface was found adhered to the deposit.*

1. *On the TL-dating of Jar-35 and its deposit*

Two dates are of interest in establishing a time frame for Jar-35. The first date is that of manufacture, which in the absence of the possibility of a severe disintegration of the jar in search of a straw or a macrofossil suited for radiocarbon dating can only be provided by luminescence dating. TL-dating is far less accurate than radiocarbon dating and the method is subject to many more potential sources of error. These include: 1) the occurrence of a nearby (< ca. 50 cm) unnoticed rock containing anomalously high uranium, thorium and potassium

(U, Th, and K). Such an occurrence could result in determination of an age artificially older than the true age; 2) the partial resetting of the TL-chronometers by sunlight, heating by a nearby campfire, or heating during use. Such events will make the determined age artificially younger than its true age; 3) finally there is the possibility that the trapped electrons are not quite stable in their traps in the forbidden energy gap between the valence electrons and the conduction band, which would lead to a slow but continuous loss of electrons and cause the determined age to be artificially too young. Although we have little control over these sources of error, luminescence dating remains our only possibility to acquire a date of the manufacture (or last re-heating event) of the jar.

Prior to TL-dating the ceramic sample was crushed, sieved, and finally placed in a sample holder in the TL-apparatus, in our case a DA15 TL-reader build by Risø National Laboratory in Denmark. Here it was heated from room temperature to 400°C. When the sample is heated, the electrons caught in the electron traps between the valence electrons and the conduction band are gradually released, and when an electron is released from its trap it recombines with a similar hole in the electronic structure of the crystal. The recombination results in the emission of a tiny flash of light (a photon), which is detected by the apparatus. The more photons that are emitted during heating, the more electrons were stored in the traps, and the more radiation must have passed through the crystal through time since the point in time when the ceramic material was heated in the potter's oven or last heated in a camp fire and in this way experienced a resetting its TL-clock. The amount of radiation that has passed through the crystal is a product of time and the dose speed rates of essentially three sources: U, Th, and K from the sample itself, the U, Th, and K from the surrounding soil lying immediately adjacent to the jar, and thirdly the cosmic radiation originating from the Sun and beyond. The last dose speed, the cosmic contribution, can be estimated as a fixed value (150 μGy/year), but the first two must be determined by measuring U, Th, and K in small samples of the jar and of the adjacent soil.

In the present study two samples were drilled out by J. Gunneweg in a darkened room, 9 and 14 cm from the bottom of Jar-35. Some soil was still attached to the handle, and from this a soil sample was procured. However, it is questionable, and at best only a rough estimate, if the radiation emitting from this soil sample is representative for the entire surroundings of the Jar in its long storage position.

Uranium, Th, and K occur in concentrations that are so low, that an especially sensitive analytical technique is required. Instrumental Neutron Activation Analysis (INAA) was carried out by lowering subsamples of ca. 100 mg of the jar and the soil into the nuclear research reactor in the University of Technology and Economics at Budapest, Hungary. Residing for 8 hours inside the running reactor exposed the samples to a neutron flux of 2×10^{12} neutrons cm^{-2} s^{-1}. This irradiation produced new isotopes from some of the U, Th, and K atoms present in the samples. These new isotopes are radioactive and the amounts of each one can upon retrieval be determined by counting the gamma-rays emitted from the unstable isotopes on a high purity Ge-detector. Based on this, the concentrations of U, Th, and K in the samples can be calculated (for a more detailed discussion see Rasmussen *et al.* 2009).

In this way, TL-dates were determined for the two samples taken at the handle. Each sample was sub-divided into 4 aliquots dated separately in the TL-apparatus in order to reduce noise. The average of the 4 independent TL-dates turned out to be AD 685 ± 41 (sample KLR-5672) and AD 680 ± 42 (sample KLR-5673). These dates were surprising young and from a time when there was no known habitation at Qumran. Therefore we immediately suspected that these late dates were due to complete or partial resetting of the TL-chronometers. The resetting could have been caused by exposure to sunlight in the 7th century AD or during the excavation in 2004.

If the scenario of resetting the TL-chronometers was indeed correct, it would be a viable strategy to secure a sample from the *interior* of Jar-35. This was carried out on small flakes of ceramics adhered to the deposit retrieved from the interior of the jar. The average TL-date of 6 independent determinations from the interior of the jar was: 105 ± 97 BC, which is in accordance with the main habitation period of the Qumran settlement.

The uncertainty interval (± 1 σ, covering 66 % of the probability distribution) is quite large: ± 97 years. Comparing this uncertainty with the results from the radiocarbon dating (see below) it can seen that TL-dating is inferior to radiocarbon dating not only in number of error sources, but also in terms of uncertainty.

Four ceramic samples were selected from the two adjoining hoards: Jar-2, a *large jar*, a *small jar* and a *cooking pot*. The results of these TL-dates were:

Sample	Lab-No.	TL-date
Jar-2	QUM-385, KLR-6996	AD 139 ± 140
Large jar	QUM-391, KLR-6999	AD 710 ± 97
Small jar	QUM-394, KLR-7000	No date
Cooking Pot	QUM-395, KLR-7001	AD 840 ± 92

These dates suggest that Jar-2 is also from the main habitation period in Qumran, whereas the TL-chronometers in the *large jar* and the *pot* have probably been reset in the same way as the exterior samples of Jar-35. We have not been able of determine what precisely caused this resetting.

2. *On radiocarbon dates of the content of Jar-35*

Radiocarbon is a much more precise and much more reliable dating method. However, this method requires the availability of organic material that can be connected directly to the event under investigation. In almost all archaeological excavations in the Mediterranean area, the major find group is ceramics, and organic material suited for radiocarbon dating has to be sought after. This was also the case here. In the case of Jar-35 only three small pieces of charcoal were indentified in the deposit found inside the jar. The radiocarbon method is described in detail elsewhere in this volume (see van der Plicht and Rasmussen, this volume p. xx). We will therefore confine ourselves here to report the result of the radiocarbon date.

Three samples of charcoal secured from the deposit inside Jar-35 were radiocarbon dated at the Groningen AMS facility in the Netherlands. The average date was 2035 ± 35 BP. The calibrated date using the INTCAL04 curves and the University of Groningen calibration program (WinCal25) was 90 BC–AD 18 (the ± 1 σ interval). The complete result of the calibration is shown in figure 40.

The radiocarbon date is in accordance with the TL-date of the interior sample from Jar-35. It is also in accordance with the main habitation period in Qumran.

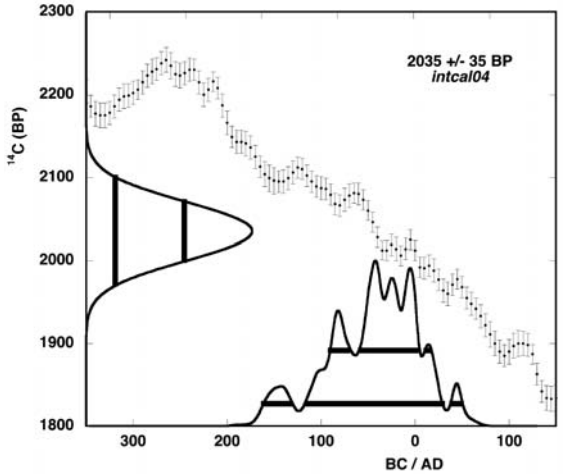

Figure 40. The calibrated date of the average of the three radiocarbon dates performed on small charcoal samples found in the deposit in Jar-35. The upper horizontal bars indicate the ± 1 σ range, and the lower horiziontal bars the ± 2 σ range. In the calibration the University of Groningen, WinCal25, is used together with the INTCAL04 curves.

3. *Summary*

We have radiocarbon-dated pieces of charcoal embedded in the deposit in side Jar-35 and found a calibrated date of 90 BC to AD 18 (± 1 σ). The exterior of Jar-35 has been TL-dated to AD 685 ± 41 and AD 680 ± 42. Two samples from the hoard of ceramics adjoining the Jar-35 have been TL-dated to similarly late dates (the *large jar* dated to AD 710 ± 97 and the *pot* to AD 840 ± 92). All of these late TL-dates are attributed to partial resetting of the TL-chronometers, possibly due to exposure to sunlight during the excavation in 2004. Another possibility is more or less complete resetting by exposure to sunlight or heating in a campfire, which should have taken place at a time near the TL-dates, *i.e.* between AD 680 and AD 840.

The interior of Jar-35 was TL-dated to 105 BC ± 97 and Jar-2 from the hoard to AD 139 ± 140. Both of these are statistically in accordance with the much more precise radiocarbon date of the deposit inside Jar-35. We conclude that Jar-35 was manufactured and used some time between 90 BC and AD 18 (± 1 σ).

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CHAPTER ELEVEN

ANALYZES OF A SAMPLE OF '*MASSE DE FER*' FROM QUMRAN LOCUS 104 EXCAVATED BY DE R. DE VAUX

Kaare L. RASMUSSEN[a], Andrew D. BOND[a], Gregory DOUDNA[b], and Jan GUNNEWEG[c]
[a]*Institute of Physics and Chemistry, University of Southern Denmark, Campusvej 55, DK-5230 Odense M, Denmark,* [b]*Columbia College, 13910 45th Avenue NE #802, Marysville, WA 98271 USA,* [c]*Hebrew University, Jerusalem, Israel*

Abstract. A sample described as *"masse de fer" (i.e.* 'mass of iron') was excavated in Qumran by Roland de Vaux. Our analyzes cannot support the description as a *"masse de fer"*. Rather we find that the sample consists mainly of quartz grains of uniform size with another small, unidentified crystalline component. In this paper we describe the situation of the find and report our analytical results, XRD, SEM-EDX and the determination of maximum firing temperature.

Keywords. XRD, SEM-EDX, de Vaux, Qumran

Introduction

The sample was excavated at locus 104 in Qumran by Roland Guérin de Vaux O.P. in 1955 and assigned the sample name KhQ-2114. The position of the find is shown in figure 41 (marked '104') and is depicted in figure 41b hereunder. Locus 104 together with loci 100 and 102 formed what de Vaux interpreted as a "miller's quarter", based on the finds of parts of millstones in loci 102 and 104, and a mill reconstructed by de Vaux in locus 100. However de Vaux interpreted the finds of locus 104, including the "*masse de fer*", not as related to the mill but rather as possibly clearing of fill from an adjacent cistern, locus 91.

Of three coins found in locus 104 mixed among other items found there, one was identified certainly as coined by Alexander Jannaeus (1st century BCE) and a second tentatively by Alexander Jannaeus, both in agreement with many similar coins found elsewhere at the site.

But the third coin found in locus 104 is identified as "Elgabalus, 218-222 CE" (Humbert and Chambon 2003), which is significantly later than the Qumran site is believed to have had human activity or dwellers.

Meanwhile in locus 91, which is the cistern whose removed fill de Vaux believed was dumped anciently into locus 104, out of a total of thirteen coins found by de Vaux in that locus, one was "4th cent. CE, 330-335 CE" and another was "Roman, 3rd cent. CE?". These coins were later than expected from the site, and they were found in the two adjacent loci, one of which was believed by de Vaux to consist of fill removed from the other. This raises the possibility that some of the items found with the coins—including the "*masse de fer*"—could similarly be from later than the believed end of habitation of the site in the 1st century CE and then, briefly, at the time of Bar Kochba early 2nd century CE.

On the other hand, it is also possible that the "*masse de fer*" could derive from 1st century BCE/1st century CE in common with the vast quantity of other items found at Qumran from those time periods.

Roland de Vaux does not seem to have discussed or offered an interpretation of these 3rd/4th century CE coins or conceivably associated finds. De Vaux did say he thought the millstones of loci 100 and 104 were evidence that "the people of Qumran cultivated corn or barley, for it would have been easier to buy flour than grain which they would then have had to grind themselves. Corn-growing of this kind is not possible on the shore of the Dead Sea, but it can be practiced in the plain of the Buqei'a which overlooks Khirbet Qumran to the west, and up to which, as we have said, an ancient pathway led" (de Vaux, 1977).

1. *Analytical methods and results*

Roland de Vaux labeled the sample of "*masse de fer*" KhQ-2114. It was designated sample number QUM-390 by Jan Gunneweg at Hebrew University (see Humbert and Gunneweg, 2003). After deliverance to Kaare Lund Rasmussen at University of Southern Denmark it was designated KLR-6998.

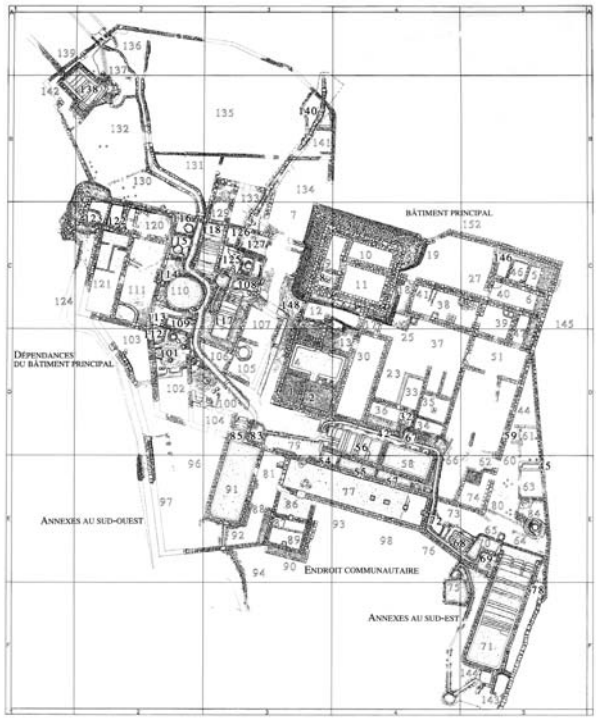

Figure 41a. Plan of the Qumran settlement during period II (from Figure IV, Humbert-Gunneweg 2003). The sample was excavated in locus 104.

Figure 41b. "Masse de fer" (KhQ2114) waster found by de Vaux.

Scanning Electron Microscopy—Energy Dispersive X-ray analysis (SEM-EDX)

A subsample was subjected to SEM and SEM-EDX analysis on a LEO 435VP equipped with a Rontec EDX-detector. A representative view of the sample can be seen on the micrograph in figure 42, which was obtained with an electric high tension of 20 kV. The EDX-spectrum of a *c.* 0.3 mm^2 area is shown in figure 43. In this spectrum are seen X-ray lines of the following elements: Na, K, Mg, Ba, Mn, Fe, Cu, Al, Si, and Cl. The ZAF-corrected semi-quantitative concentration data are listed in Table 1. It should be stressed that these numbers are only semi-quantitative. They are normalized to yield 55 wt% for the selected elements. No other elements could be seen in the spectrum; however there are most likely lighter elements present that are not detected by the EDX-detector.

The main element is Si (21 wt%) followed by Al (13 wt%) and Fe (11 wt%). This composition is more in accord with silicates than iron rich compounds such as iron oxides or iron hydroxides, although the latter cannot be excluded from the EDX-data alone. The large amount of Al (trivalent) would make a clay mineral a likely candidate. The rest of the elements detected are found in concentrations below 5 wt%: Na, K, Mg, Ba, Mn, Cu, and Cl. They could either be part of a silicate phase or they could be part of other minor mineral components.

X-ray Diffraction (XRD)

We then undertook a structural analysis by X-ray diffraction in order to identify the crystalline components of the sample. A *ca.* 100 mg sample of KLR-6998 was crushed with an agate pestle and mortar, and the powder X-ray diffraction pattern was measured at room temperature using a Siemens D5000 instrument, equipped with CuK**a** radiation (wavelength 1.5418 Å). The resulting profile is shown in figure 44. There is a very good match between the sample (black line) and the database entry for quartz, SiO_2 (red bars). Based on this analysis, it is evident that the major crystalline phase in the sample is quartz. Three peaks of very low intensity (at 2θ = 12.4, 20.0, and 25.0 degrees) suggest the presence of some minor crystalline phase, but this has not been identified. It should be stressed that the XRD-technique identifies only crystalline phases, and has an effective detection limit in the region of 2–3 volume %. Amorphous materials will not be detected,

and trace amounts of other crystalline phases, if present, might also escape detection.

Determination of the maximum firing temperature

At this stage it is rather clear that Father de Vaux's field notation of the sample being a *'masse de fer'* is not supported by our analyzes. One hypothesis is that the sample could be some sort of deposit or slag originating from the potter's kiln in the Qumran settlement.

In order to test this hypothesis we undertook to determine the maximum firing temperature (if any) that the sample had experienced. The methodology is described in Rasmussen (2003; 2008) and Rasmussen and Hjermind (2005). It is an empirical method that relies on the progressive phase transformation of clay minerals and other silicate minerals as the temperature is raised, probably involving the formation of magnetite. When a sample, which was previously heated to a certain maximum temperature, T_0, is stepwise heated in the laboratory and in between measured for magnetic susceptibility, a graph is produced, which shows a slowly varying susceptibility until the temperature T_0 is reached. At T_0 the susceptibility curve exhibits a sharp discontinuity, which can be even more acutely recorded by plotting the square of the first derivative of the curve. The square of the first derivative usually shows vanishing values until the maximum firing temperature T_0 is reached, at which point the curve raises abruptly. The method has been verified on a series of samples of natural clay, which were preheated to progressive higher temperatures.

The magnetic susceptibility as a function of stepwise heating temperature (black curve) as well as the square of the first derivative of the susceptibility (red curve) are shown in figure 45. From the plot it is clear that the sample has indeed been heated, and we estimate the maximum firing temperature in antiquity to be 730 ± 10 °C.

2. *Discussion*

There are at least two examples of decorated Pseudo Nabataean pottery of local Qumran manufacture, QUM-134 from Cave 7, No. 6, a locally made Qumran jar with two inscriptions ROMA in black and with a red-painted rim, and KhQ-621, a locally made Qumran jar with red script in Aramaic (Lemaire, 2003, *p.* 346-7) both shown in Figure 46. This is the only type of decoration found on pottery belonging to

the habitation period *c.* 40 BCE—68 CE. If the ink was fired in the kiln, which we do not know took place for sure, the presence of such inscriptions and decorations suggest that fired clay (or some other compound) was ground, mixed with a fluid and then used to write on ceramic vessels and ostraca. The Qumran ostraca have scripts in beige, brown, and sepia, and if the writing had also been fired, these inks could not have been manufactured using black carbon ink or any other organic paint, because those color pigments would have been destroyed in the high temperatures of the potter's kiln. The red collar of the ROMA jar, however, was applied before firing.

Investigating Qumran jar QUM-235 Jan Gunneweg found a major clay contribution with a FT-IR spectral similarity to burnt umber, a brown color pigment derived from clay (this volume p. xx). The same signal was found in the powder sample of QUM-222, which is a black substance from an inkwell. On black scraping from jar QUM-223 was found a FT-IR match with bentonite. It could therefore be asked if the *'masse de fer'* sample was a piece of burnt bentonite intended for the production of brown/umbra color.

In the strict sense of the word 'bentonite' is a clay mineral, an aluminum phyllosilicate, generally impure, and mostly consisting of montmorillonite. There are several types of bentonites depended on the dominant elements, K, Na, Ca, and Al. Bentonite usually forms from weathering of volcanic ash, often in the presence of water. However, the term bentonite can also be used more loosely for clay deposits of uncertain origin. In consequence of this, it is somewhat uncertain what the FT-IR reference to bentonite really covers. However, based on the XRD results there is clear evidence that the major phase of the sample KLR-6998 is quartz and not bentonite. We did, however, show the presence of a so far unidentified minor crystalline phase (probably less than 2-3 wt%), which could possibly be bentonite or some other clay mineral. Imaging in the SEM shows well-rounded grains of uniform sizes in accordance with the identification of quartz, and the SEM-EDX analyzes show consistently that the sample contains large amounts of Si. The presence of Al, Fe, Mg, Na, and K is not in contradiction with this. These elements could be part of a doped quartz phase, or more likely they could belong to one or more minor mineral phases. One likely candidate for such a minor phase would be a clay mineral.

The sample KLR-6998 was indeed fired, and the maximum firing temperature was determined to be 730 ± 10°C. This is in very fine

agreement with other firing temperatures determined on ceramic samples from Qumran, as Rasmussen (2003) showed that the general range of firing temperatures for ceramics samples fired in the oven at Qumran was between 710 and 860ºC. It is therefore a quite plausible hypothesis that the *'masse de fer'* sample was fired in the kiln at Qumran.

3. *Concluding remarks*

The so-called *'masse de fer'* sample is neither iron nor an umbra colored pigment consisting of burnt bentonite. Its major crystalline component is quartz. However, the sample was fired at 730ºC, which would be expected if the inhabitants of the Qumran settlement thought it was bentonite and intended to process it for the production of umbra colored pigment. It has a brown color, a content of Si, Al, Fe, Mg, and K, and an undetermined minor crystalline phase. These facts put together could sustain the possibility that the sample had been mistaken for bentonite by the inhabitants of Qumran, heated and then discarded, when it failed to have the desired qualities to be used as a color pigment.

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Table 1. Results of an SEM –EDX analysis of a ca. 1 mm^2 area. The concentrations are ZAF-corrected and the sum yields a total of 55.3 % for the selected elements. The numbers are only semi-quantitative. The relative abundances are more reliable than the absolute values. No other elements could be seen in the spectrum.

	Concentration (wt %)
Ba	1.6 ± 0.2
Fe	10.8 ± 1.1
Cu	0.2 ± 0.1
Na	4.9 ± 0.4
Mg	2.4 ± 0.4
Al	13.3 ± 1.5
Si	21.4 ± 2.3
Cl	1.9 ± 0.3
K	1.7 ± 0.2
Mn	0.6 ± 0.2
Sum	55.3

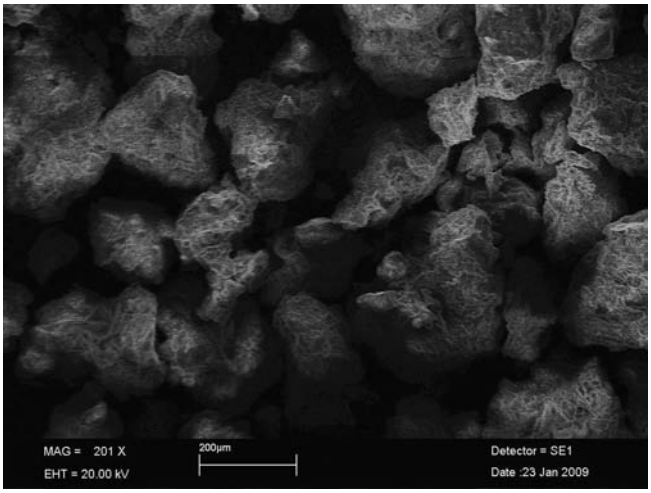

Figure 42. SEM micrograph of a representative part of KLR-6998, which consists of well-sorted partly rounded grains, seemingly coated with an at least partly crystalline compound.

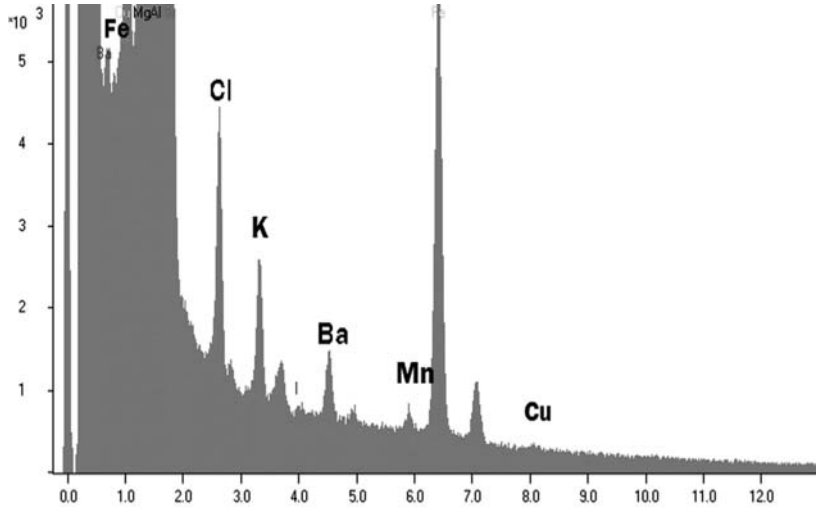

Figure 43. EDX-spectrum of an area of ca. 1 mm^2. Sample KLR-6998. X-ray intensity in counts as a function of X-ray energy in keV. The following elements are seen: K, Mg, Ba, Mn, Fe, Cu, Al, Si, (I), and Cl.

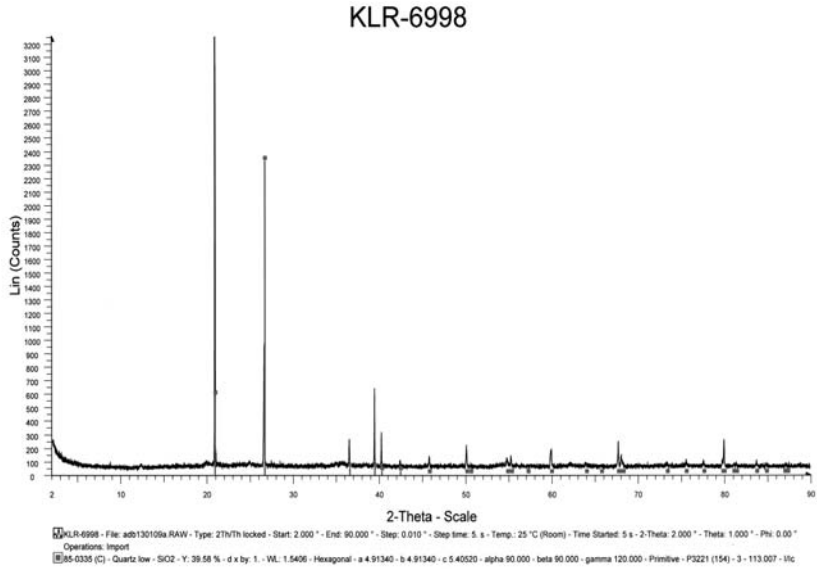

Figure 44. Powder X-ray diffraction pattern of KLR-6998. The black line is that measured for the sample. The red bars correspond to the database entry for quartz (SiO_2).

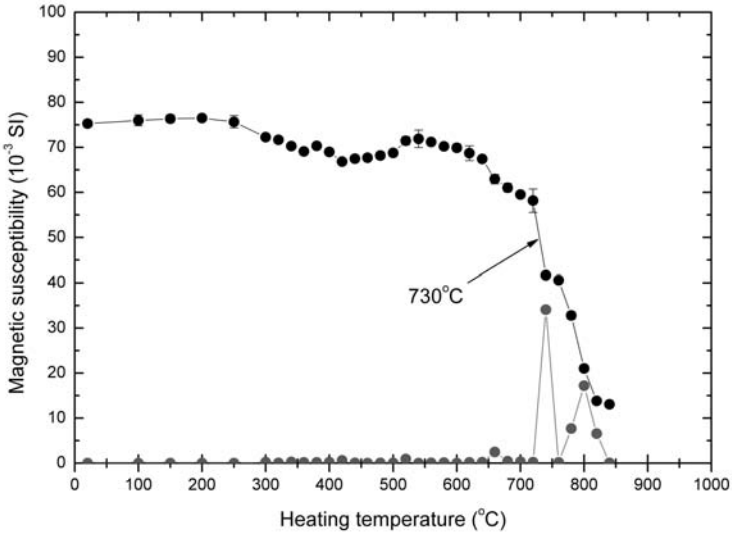

Figure 45. Magnetic susceptibility measured of a c. 40 mg sub-sample of KLR-6998 after stepwise heating in the laboratory (upper curve). The square of the first derivative of the susceptibility curve is shown below on an arbitrary scale (lower curve). The discontinuity seen between 720 and 740 °C indicates the maximum firing temperature.

CHAPTER TWELVE

THE SCIENCES AND THE RECONSTRUCTION OF THE ANCIENT SCROLLS POSSIBILITIES AND IMPOSSIBILITIES (SUMMARY)[1]

Emanuel TOV
Department of Bible, The Hebrew University of Jerusalem

Abstract. Over the past five decades, the sciences have come to our aid in examining several material aspects of the scroll fragments, their coverings and stitching material. The sciences can help us in determining the date of the scrolls, whether fragments derive of the same sheet by DNA, C14, and follicle patterns in parchment and fibers in papyrus. Advanced photographic techniques can retrieve previously unreadable letters and identify fragments and determining the relation between fragments of scrolls with the aid of computer-assisted research.

Keywords. Parchment, papyrus, C14, exegesis, DNA, follicle pattern

The study of the Qumran scrolls is the study of fragments and sheets rather than that of complete scrolls (the Messianic Apocalypse text in figure 46 serves as an example). When dealing with a topic like the sciences and the ancient scrolls, scientists often forget that these fragments are parts, however minute, of once complete sheets, and that each medium-sized scroll consisted of a number of sheets. A fragment does not constitute an independent unit for a material investigation, since the information about fragments needs to be supplemented by that in other fragments deriving from the same sheet. Each sheet forms an independent unit, not necessarily of the same nature as the sheet that is now stitched to it. Therefore, in the material analysis of the fragments it is necessary to know more about each sheet or the scroll as a whole. The scroll is the overriding unit, but since many scrolls are

[1] For the full text of this study, see my contribution to *The Dead Sea Scrolls in Context: Integrating the Dead Sea Scrolls in the Study of Ancient Texts, Languages, and Cultures, Vienna, February 11–14* (Series No. not yet known) (ed. A. Lange et al.; Leiden: E. J. Brill, 2009).

Figure 46. An example of Hebrew script consisting of the Messianic Apocalypse text of the Dead Sea scroll 4Q521. This fragment was found in Cave 4 at Qumran. Photo Clara Amit, Courtesy of the Israel Antiquities Authority (Color photo in Plate X).

composed of different sheets, we have to base our remarks on these sheets. Single-column sheets like 4QTest (4Q175) and single-sheet scrolls are rare in Qumran. Most scrolls are composed of a number of sheets, seventeen in the case of the large Isaiah scroll.

Over the past five decades, the sciences have come to our aid in examining several material aspects of scroll fragments, their coverings, stitching material, etc. There are many ways in which the sciences helped or *could* help us gain a better understanding of the scroll fragments and aid us in their reconstruction. The main areas are: (1) determining the date of the scrolls (based on the age of the leather and ink [?]), (2) determining whether fragments derive from the same sheet (Carbon-14, DNA research, the chemical composition of the leather and ink; follicle patterns in leather, and fibers in papyrus), (3) retrieving previously unreadable letters with the aid of advanced photographic techniques, (4) and identifying fragments and determining the relation between fragments with the aid of computer-assisted research. At the same time, we should also be able to determine where these sciences are *unable* to help us.

This study refers solely to the scientific examination of the fragments, and not to the identification and reconstruction on the basis of content.

I. *Topics examined and results reached with the aid of the sciences*

Individual scholars as well as groups of scholars advanced the scientific investigation of the scrolls in individual and collective publications dealing with the sciences. Progress has been made in the following areas.

a. *Dating the scrolls: Carbon-14*

The first system used for dating scrolls was that of *paleography* (dating on the basis of the type of handwriting), and this is still our major resource for dating. At the same time, at an early stage in the study of the scrolls, C-14 examinations of the leather and papyrus fragments became instrumental in determining their dates, usually corroborating paleographical dating. These examinations have been applied only to a small number of scrolls.

Some scholars ascribe the deviating dates of some documents—either too early or too late according to the common view about Qumran—to the applying of castor oil to the leather in the 1950s in order to improve the clarity of the written text. On the other hand, according to others, the possible influence of such oil is negligible. The last word has not been said on this issue, and the presence of castor oil on the margins of the leather (from which samples were taken) as opposed to the inscribed surface itself, has yet to be proven.

b. *Relation between fragments*

When reconstructing scrolls there are many unknowns. The question of whether two or more fragments should be joined as adjacent fragments or designated as belonging to the same column or sheet, remains a major issue in scrolls research. Information about the content is usually insufficient in fragmentary scrolls. The analysis of script is often equally unsatisfying when analyzing small fragments. We would appreciate some help from the sciences in either linking fragments or excluding such a connection, but such help is still being developed. In short, we would like to have objective criteria for making a connection between any two fragments or excluding such a possibility. The first steps in exploring some possibilities have been made, but scholars are in need of a database incorporating alternative scientific data referring to a large number of fragments. The techniques that come to mind relating to the possible joining of fragments are DNA research, ink

research, research of leather follicles and papyrus fibers, and elemental composition analysis. However, it should be remembered that these examinations can only determine whether or not two fragments belong to the same sheet. A fragment is not a unit; the real unit is the sheet, because the information gathered by the aforementioned examinations pertains to the sheet as a whole. To the best of my knowledge, all these techniques would produce the same results for fragments taken from any part of the sheet (C-14, DNA, research of leather follicles, ink research), with the exception of the examination of fibers in papyri, a technique that is not yet developed.

In all these cases, the sciences may help us in determining whether a frg. a and frg. b derive from the same sheet or of the same animal, no more and no less. If they derived from the same sheet, the exact relation between these fragments cannot be determined with the aid of the sciences, since the fragments could be three columns apart, so that multiple possibilities should be envisaged. Furthermore, if two completely different compositions were written on skins deriving from the same animal, wrong conclusions could be drawn if we were to be guided solely by the scientific examinations.

1. DNA research of ancient texts is still in its infancy. The technique, applied to fragments of several scrolls, can (1) determine the species of animal from which the leather derived, (2) distinguish between the DNA signature of individual animals, (3) determine groups of animals (herds) from which the hides derived. Ideally, these herds should be linked with bones of individual animals or herds, ancient or modern, since the DNA signature has not changed from antiquity to modern times. However, these links between hides and herds have hardly been made, and researchers are still waiting for the construction of databases that link specific fragments and bones.

2. The study of the composition of ink could give us some clues regarding the relationship between scroll fragments. Rabin believes that a basic distinction can be made between ink prepared at Qumran and ink prepared elsewhere because of an analysis of the water component in ink. In particular she points out that the chlorine / bromium ratio is higher in places near the Dead Sea than in other localities. Studies like this could help us differentiate between groups of scrolls penned at different locations, even if the locations themselves cannot be named.

3. A study by Hahn et al. based on the contaminants present in the parchment and ink (elemental composition analysis) showed how two

fragments cannot have been belonged to the same sheet. The elemental composition analysis of the leather and ink executed by Hahn et al. determined that 4Q413 and 4Q413a could not have belonged to the same sheet. This type of analysis may well be better suited for negative than positive conclusions.

4. Sheets in parchment scrolls were joined with different stitching materials. DNA and C-14 analysis of the stitching materials may aid us in understanding the background of the different scrolls. So far, one such examination has been carried out.

c. *Retrieving previously illegible letters with the aid of advanced photographic techniques*

For their time, the black/white infrared photographs taken by Najib Anton Albina, the photographer at the Palestine Archeological Museum (PAM) in the 1950s and 1960s were extraordinarily good. In later years, with the advancement of technology, better photographs were taken, revealing additional parts of letters, complete letters, and in rare cases complete words.

d. *Identifying fragments and determining the relation between fragments,*

Especially with research of hair follicles in leather and fibers in papyri. The analysis of hair follicles and papyrus fibers could indicate that two or more scroll fragments derived from either the same or a different sheet. The research of leather and papyrus sheets is promising, but at this stage it is unclear whether the various parameters identified in the fragments are distinctive enough in order to identify and differentiate between individual sheets. Research needs to proceed from the features of known sheets of complete scrolls to fragmentary texts, and such studies have not yet been written.

II. *Aid from the sciences for the reconstruction of ancient scrolls: possibilities and impossibilities*

In previous examinations, the reconstruction of the missing parts of the ancient scrolls was based mainly on content. In the case of biblical scrolls or other known compositions, content is our main guide, but even in these compositions small fragments with partial or frequently

occurring words cannot be identified easily. In other cases, with fragmentary contents and the fertile minds of scholars, there are many possibilities and therefore it would be good to be aided by additional methods. Such aid may come from an exact or almost exact physical join, but such joins are rare. Some fragments of similar shape reflect subsequent layers or revolutions of a scroll, but such cases are also rare. In many cases, we would like to look to the sciences for help. Our main interest would be in proving or disproving a link already made between two fragments or in searching for a scroll to which a given fragment may have belonged. In such cases, we would like to resort to the sciences for objective criteria. The sciences have been invoked often, with high expectations, so it is time to be a little realistic.

It would not be feasible to send all the fragments to C-14 analysis only in order to know if their C-14 dates match. Ink analysis, if advanced sufficiently, would be easier and may be very relevant. In my view, the so-called elemental composition analysis sounds promising, and it is non-destructive, but we wait for the verdict of scientists. DNA will provide some answers, as will the follicle research on leather, and fiber research on papyri. It should be remembered that the maximum results we would receive refer to the identity of the complete sheet(s) from which the fragments derived, and not to the placing of individual fragments. These sheets were 21 to 90 cm long at Qumran, mostly 30–40 cm, and the placing of a fragment in such a large space would leave many options open. Most animals would not yield more than one hide of 90 x 60 cm. For details relating to these and other technical aspects of the scrolls, see my monograph *Scribal Practices and Approaches Reflected in the Texts Found in the Judean Desert* (STDJ 54; Leiden/Boston: E. J. Brill, 2004).

On the other hand, in the descriptions of the DNA method, especially that of Woodward, the expectations from DNA have been very high. In a programmatic paper (1998)[2], he lists five questions for which DNA was supposed to provide answers.

1. "How many different manuscripts are represented in the collection of fragments at the Rockefeller and Israel Museums? … Obtaining DNA signatures unique to each manuscript will make it possible to sort out the physical relationships of scroll fragments." However, at

[2] S. R. Woodward, in D. W. Parry, D. V. Arnold, D. G. Long, S. R. Woodward, "New Technological Advances: DNA, Databases, Imaging Radar," in *The Dead Sea Scrolls after Fifty Years: A Comprehensive Assessment* (ed. P. W. Flint and J. C. VanderKam; Leiden/Boston/Cologne: E. J. Brill, 1998) 1.496–515.

most, we would be able to list the individual animals, from whose skins the hides were derived. When naming these animals "animal 1," "animal 2" we would have an important summary list, but that list would provide only a few clues for researchers. Thus, if two different compositions were written on the hide of animal 1, DNA alone would not suffice to distinguish between them. Further, multi-sheet compositions required more than one animal, sometimes as many as ten or more, so that DNA signatures alone would not be able to distinguish between Qumran manuscripts.

2. "Which pieces can be grouped together as originating from the same scroll because they are from identical or related manuscripts? … This should assist both in the reconstruction of manuscripts and in the verification of assemblies that were previously already made." It seems to me that all these are idle hopes as explained in my reply to item 1.

3. "Did more than one scribe work on a single document, or did different scribes use parchment that originated from the same source for different manuscripts?" In my view, neither question can be answered with DNA.

4. "Is the parchment for the patch from the same herd as the original manuscript? Does the patch represent a herd from a different region, reflecting mobility of either the original scroll or the herd?" These suggestions are helpful, but impractical. Most importantly, the number of patches in the scrolls can be counted on one hand.

5. "Does the collection represent a library from a single locality, or is it a collection representing contributions from a wide region?" In general it is true that DNA analysis will help us to know more about the provenance of the hides, if only the connections between hides and bones can be made.

Summarizing the various types of expectations for scroll research, we note that they may help us with regard to some issues.

a. C-14 examinations should be continued as a useful tool for dating in spite of the uncertainty regarding the contamination of castor oil.

b. If performed on a large scale, C-14 examinations could also help us understand the relationship between many individual fragments. For example, two or more fragments assigned to the same column or sheet should not have different C-14 dates.

c. Ink research, research of leather follicles and papyrus fibers, and elemental composition analysis such as the chlorium/bromium ratio should be encouraged as non-destructive examinations that may help us understand the relation between individual fragments. Scientists

should review the merits of these examinations, since we humanists lack the means to review the methods used.

d. The infrared color photographing of all the fragments with new techniques should be encouraged.

At the same time, expectations from these techniques should be realistic, taking into consideration the realia of scroll production such as described above, in particular the fact that the sheet and not the fragment is the unit of reference.

In an ideal world, we would have access to a database providing information of all the types described above about all the scroll fragments. Undoubtedly, this information would help us to solve some questions that face researchers. For example, by examining the technical data about the scrolls, we may be able to create clusters of scrolls of a certain nature, such as Qumran scrolls as opposed to non-Qumran scrolls (based on elemental composition analysis). We may be able to find that scrolls written on a specific type of leather (DNA analysis) or with a specific type of ink have something in common, or that the Hebrew scrolls somehow differ from those written in Aramaic.

In the analysis of individual fragments, this database would help especially in negative aspects, namely the suggestion that two fragments that were joined in the past should not be ascribed to the same manuscript, as in the case of 4Q413 and 4Q413a discussed above.

In an ideal world we should have access to a database like this, but we are also realistic enough to realize that the keepers of the scrolls would have to agree to all these procedures, some of which are destructive.

CHAPTER THIRTEEN

INTRODUCTION TO "SOAP AT QUMRAN"

Jan GUNNEWEG
Archaeometry, Hebrew University of Jerusalem

Abstract. This is the Introduction to what triggered the soap manufacture possibility at Qumran, before the Honour Class of Leiden-Delft tried to execute the proposed recipe in reality.

Keywords. Soap production, Qumran, lye, *Salsola Kali*, date palm

In April 2007, a discussion took place for a collaboration project between Joris Dik of the Technical University of Delft and myself of the Hebrew University of Jerusalem whereby the Honours Class with a program entitled "The merging of science, arts and archaeology" 2007-2008 of Leiden-Delft Universities in Archaeometry would be involved.

The project we discussed focused on an aspect of Qumran in Israel. The research project had to be feasible with positive results to be shown during the combined Lorentz Center Physics and NIAS Workshop of April 2008 where some members of the Honours Class would present their first Archaeometry work as an oral presentation and as a paper in the subsequent Workshop Proceedings. I proposed a hypothetical topic that seemed feasible, also because I had done some homework in Israel in 2005. The questions were whether 1) the Qumranites could have made soap 2000 years ago in their settlement on the shores of the Dead Sea, whether 2) they had the ingredients to make soap and 3) what they would have done with it. 4) Last but not least, what would be the impact on the history of Qumran in the light of the fact that many see the site as a sectarian settlement with strict purity laws, where the use of soap would be logical item having.

The reason that we wanted to focus on the soap manufacture was foremost the extreme puritan character of the Essenes that was described by the ancient writer Flavius Josephus, a Jewish army com-

mander converted into a history writer for the Romans in the first century after Christ. He described a sect called the "Essenes", a group of dissident Jews during 2nd century BC—1st century AD. If his account and that of the Dead Sea manuscripts apply to the same people that lived at Qumran 2000 years ago and by many thought as one of the Essene sectarian groups, the use of soap and the description of purity in the Dead Sea scrolls would indeed apply to the site of Qumran. If the Qumranites were that pure, we reckoned, they must have had access to a detergent in order to wash their linen cloths in which Flavius Josephus depicted them.

The second reason for the use of soap came from the excavator of Qumran in the 1950s, Roland de Vaux, who had found two low basins that he interpreted as a laundry facility in rooms 52-53 within the settlement that also had enough water nearby needed for doing laundry.

In order to make soft soap—the solid bar of soap is a recent French invention of two centuries ago—the Qumranites needed water, animal fat (goat tallow) or plant (olive oil) fatty substances, a fire and lye, also known as caustic soda. Without lye, one cannot make soap. What I intended to show is that the Qumranites could have had the knowledge how to produce the necessary lye and probably did based on material finds that will become clear in the next paragraphs.

Lye can be made from trees or plants that contain either sodium (Na) or potassium (K), both major elements because they occur in Nature in the percentage range. Especially plants that grow near the sea contain more sodium and potassium than more inland growing plants. At the Dead Sea we have two items of vegetation that grow all-year round near the Dead Sea: The Salsola Kali brush, better known by the name of Whortleweed and the date palm tree (*Phoenix dactylifera*).

How the process of making soap was invented is a mere speculation, but quite understandable if one considers that when the ancients grilled meat on a spit, fat would drip on the ashes of the bonfire and once water seeped through these ashes, one would obtain a soapy material. When this is mixed with sand and water by rubbing a cloth against itself or against another cloth, foam is produced that cleans a cloth from its greasy dirt. Dirt that does not contain fatty substances can usually also be cleaned by the use of salt water and rubbing. In both cases, the principle of washing is primarily a rubbing motion.

When this idea is applied to Qumran, the Qumranites had to be able to make lye in order to fabricate soap. For that reason, I collected a small bunch of date palm branches and an entire bush of Whortleweed (*Salsola Kali*) from the direct Qumran environment. Both items were separately burnt on a cleaned barbeque grill. 47 shows the cindering of *Salsola Kali*.

The cold ashes were put in a small plastic bottle of which the bottom was removed, whereas the cap was pierced by three tiny holes. The small bottle was placed upside down into a bigger plastic bottle of which the upper part was cut out to let the smaller bottle lean into it. On top of the ashes in the small bottle, water was poured which in 3-4 hours seeped through the ash and dripped into the lower bottle as shown in the figure 48. Water leached the sodium and the potassium. In order to show the deposit that was formed, the water was made to

Figure 47. In the upper left corner a date palm tree and the *Salsola Kali* bush on the right. Down, the cindering of *Salsola Kali* into ashes (photos J.G.).

Figure 48. From left up to right down: Two bottles of different size, the small one without a bottom, the larger one without neck and rim are placed into each other. The small bottle contains the ashes and water is poured on it that slowly seeps through it to the larger bottle. The obtained fluid collected in the lower bottle contains the lye (photos J.G.).

evaporate and a small cookie of sodium or potash carbonate became visible.

The Qumranites, however, did not have plastic bottles and just using jars that they had would result into a messy undertaking. This lead to the search for a vessel only developed at Qumran that might have served to contain ashes and that could be set into a jar and then filled with water that would seep through these ashes through a narrow opening. One of the first samples that were taken of Qumran pottery to establish its provenance was a jar of an unusual shape that might answer exactly to the type of a jar that was used for the making of lye. The jar in question as depicted in figure 49 has an opening that is wide enough to be filled from above. The jar has two handles to transport the jar wherever needed and a 1.5 cm diameter funnel opening at the bottom. The jar could be placed into any storage vessel that had an opening to contain the funnel-shaped jar. The same jar cannot be used for any other purpose, because pouring liquid such as oil or wine into another jar would not be functional because wider funnels

Figure 49. Photograph and drawing of the funnel jar found in Locus 45A. The code KhQ800 stands for Khirbet Qumran that was used by R. de Vaux (photo J.G.)

could be used, of which some have been unearthed at Qumran. Also in the eastern excavations of Qumran by Magen/Peleg a complete vessel that has exactly the same form and size has been found (Magen & Peleg, 2007, p.17, Pl. 2:3). Usually, one could pour a liquid from one jar into another without the need of a funnel. The small diameter of the intentionally shaped funnel served the purpose of letting water seep slowly through over a prolonged period of time, for example, overnight.

The third piece of evidence that points to the fabrication of soap at Qumran was for the first time shown during the Brown University meeting in 2001. Yuval Peleg presented, among others, an ostracon that was found just to the East of the potter's workshop at Qumran on the refuge dump between the settlement and the cemetery. The in ink inscribed words read: " [El'azar] Bar Yeshu'a Haborit" (Magen & Peleg 2007, p. 21, Fig.26). The Hebrew word *borit* is used in the Old Testament and means soda, lye.

The modern name for soap in colloquial Hebrew is '*sabon*' that probably derives through Arabic from the 1000 AD legend centered on Monte Sapo in Rome, a name that lives on in the western European

Figure 50. Hebrew Ostracon found at the eastern dump at Qumran with enhanced letter tracing of the original writing. The ostracon reads: *"[Eleazar?] bar Yeshu'a haborit"* (tracing of photo by J.G. from Magen/Peleg 2007, p. 21, Fig.26).

languages as French *savon*, English soap, Italian s*apone*, German *Seife* and Dutch *zeep*.

However, the ancient Hebrew language has another word for soap, *BORIT*, that is used in the Old Testament meaning soap but that probably refers to the way soap is made, i.e. a (caustic) soda or lye in general. *Borit* occurs in the Bible in Mal 3:2 and Jer. 2:22 and is derived from the verb *barar*, which means to purify. The substantive b*or* means "lye" and can be found in Is. 1:25 and Job 9:30.

It is rather interesting that just that name, *haborit* (figure 50), occurs in written form on one of the ostraca that have been found at Qumran and serves well our hypothesis that soap or lye was produced at Qumran.

A second use for lye could have been for the dehairing of animal skins that were prepared for making parchment. Although it is common knowledge that tanning is not needed for the making of parchment, the use of lye would have greatly facilitated the process of dehairing a hide. On the other hand, also remains of sandals have been found at Qumran. Leather used for making sandals would have needed

a tanning of the hide for which the present type of lye would have been welcome. In the near future, we will analyze a sample of a sandal that will indicate what sort of lye the Qumranites used to tan leather.

In the light of the above, I hope to have shown convincingly that, at least theoretically, a fluid, soft soap was indeed made at Qumran. The next step was to make actually soap from the data palm wood and Salsola Kali ashes, a project that started on the 17th of March 2007 when all involved met at the Chemistry Department of the Technical University of Delft, Netherland.

The results of these five days of work—through March-April—were shown by members of the Honour Class 2007-08 at the Lorentz Center and their contribution will appear in Chapter 14, hereunder.

What will be needed now is to sample the obtained soap that was produced by members of the Honour Class at Delft/Leiden and submit it to any technique of spectroscopy (perhaps INAA) in order to obtain a chemical fingerprint with specific trace element abundances that are not only characteristic to Qumran, but of which also traces have to be found in the still existing remains of the laundry facility in Rooms 52 and 53 at Qumran in order to compare the data. This, however, remains as yet a *desideratum*.

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CHAPTER FOURTEEN

MAKING SOAP AS THE QUMRANITES DID

Sasja van der VAART, Alexandra van den BROEK, Laura KLERKX and Nanda de VREE
Honour Class students in Archaeology/Archaeometry at Leiden University

Introduction

This year the Honours Class "The merging of Science, Arts and Archaeology" gave four students from the Leiden University the opportunity to transform themselves into archaeochemists! At Jan Gunneweg's request, the main research goal of this project was to attempt to figure out whether it would have been possible for people living in ancient Qumran to make soap. In order to tackle this problem help was recruited from the TU Delft. They were instrumental in performing the experiments needed to answer the key question of this project. The results of the experiments were presented at the transdisciplinary workshop 'A holistic view on Qumran and the Dead Sea Scrolls', and what follows is the written version of the results.

Background information

Qumran is located in Israel near the western shore of the Dead Sea, and the Dead Sea Scrolls were found in caves nearby the site. The theory is that a Jewish sectarian group, probably the Essenes, were the residents of Qumran. Ancient authors such as Flavius Josephus, Pliny the Elder and Philo of Alexandria mention these people in their writings. They describe the Essenes as a people who had to have a high corporal hygiene. It was very important to them that they be clean in body and in clothing. The Qumran site seems to support this notion. The archaeologist R. de Vaux found several basins that could have been used for doing all the laundry that would have been needed to keep the Essenes in clean clothes. The question, of course, is what did they use to wash the clothes with? Well, the obvious answer is fairly simple, soap.

But could the Essenes have made soap in ancient Qumran? To answer this question experiments using authentic local , substances would be needed.

1. *Methods, techniques & results*

For making soap, one needs water, oil or animal fat, lye and heat. It has been proven that the Essenes had access to fire, water, olive oil and animal fat. The missing ingredient is lye. But could the Essenes have made lye in ancient Qumran 2000 years ago? In the field, lye can be obtained in the form of wood ash lye. One can quite simply burn plants or wood from trees, mix the ashes with water and thus produce a lye solution. The problem is that not every tree or plant is equally suitable.

Certain wood ashes contain either potassium carbonate or sodium carbonate. And when these carbonates are dissolved in water, a chemical reaction takes place that results in the production of hydroxide ions (OH^-). It is the hydroxide ions that make the solution alkaline. Two good candidates for the region would have been the date palm *(Phoenix dactylifera)* and the Whortleweed *(Salsola kali)*.[3] Ashes from both plants were made available for the experiments.

Before experimenting with authentic substances, calculations were performed to establish how much sodium or potassium carbonate needs to be dissolved in a liter of water to make solutions with specific pH values. This would be needed to get a rough indication of the amount of carbonate contained in the ashes used in later experiments.

$$K_2CO_3 \xrightarrow{H_2O} 2K^+ + CO_3^{2-}$$
$$CO_3^{2-} + H_2O \leftrightarrow HCO_3^- + OH^-$$

$$Na_2CO_3 \xrightarrow{H_2O} 2Na^+ + CO_3^{2-}$$
$$CO_3^{2-} + H_2O \leftrightarrow HCO_3^- + OH^-$$

A formula was created for determining how many grams K_2CO_3 or Na_2CO_3 are needed to make solutions with certain pH values. Using this formula it was calculated how to make solutions with a pH of 9.5/10.5 & 11.5 for both sodium and potassium carbonate. The solu-

[3] *Personal communication* Bob Ursem, head of the Hortus Botanicus, Delft. April 2008.

tions were made according to the calculations using pure sodium and potassium carbonate. In order to gain some more experience in the lab, further experiments were done using ash from the university barbecue to see if it was possible to make an alkaline solution with them. The solutions had a pH of about 11, qualifying them as lye.

Having established this, the next step was to make lye using the real date palm and Whortleweed ash. 1 Gram of each of the ashes was dissolved in water and a precise reading of the pH was taken. The date palm ash produced a solution with a pH of 11.09, for the Whortleweed ash this was 11.55. So, it is in fact possible to make lye using the ashes of Whortleweed and the date palm. It was now time to make soap the way the ancient Qumranites might have.

More lye was made to make the soap, using up the remainder of the ashes. Based on previous experiments a pH of around 11.5 was expected. A quick check was done using pH-indicator paper. This showed the pH of the lye to be between 10 and 11, as expected.

The lye then had to be mixed with "animal fat", unfortunately real animal fat was unavailable. Stearic acid is an artificial version of animal fat, and would suffice in this experiment. The stearic acid was melted in an oil bath. Because it was unclear in what proportions the lye and stearic acids needed to be mixed, a small amount of stearic acid was used and lye was gradually added to it while stirring constantly. This method was used because it did not matter if too much lye was added, but it would have been a problem if the lye ran out before soap had formed. The available ash had been used up in making the lye and no more could be made. After adding enough lye, soap did indeed form! The beaker was iced to cool the mixture down. The mixture separated into two layers with the soap floating on the surface, this was scooped off and put in a container.

While the soap experiments were prepared and performed, Joris Dik was busy performing tests on the Whortleweed and date palm ashes used in making the soap. He performed X-ray diffraction analysis on the samples to determine the chemical composition of the ashes. X-ray diffraction is used to determine the crystalline structure of minerals, from the angle at which X-rays are reflected. The results showed that the date palm ash consisted of approximately 80% potassium chloride. The Whortleweed ash consisted of around 40% potassium chloride and 40% of sodium chloride.

2. *Future work*

The above proves that it was possible to make soap in ancient Qumran, but there is still more work to be done. The TU Delft will be performing spectrographic analysis on the soap made with the date palm and Whortleweed ash and compare this with modern soap as it is made today in the Netherlands. The next step will be to take samples from the remains of the laundry basins in Qumran. This step is of course contingent on whether there still are residues in the basins that can be sampled. It is hoped that it will be possible to extract some from the pores of the walls of the very poor remains of the basins. These samples will then be submitted to High Performance Liquid Chromatography. If trace elements are found both in the soap and in the basins, it might be possible to match them. Thus it would be possible to link the two and prove that the people who lived in Qumran made soap in the manner described above. And this would then be one extra little indication supporting the theory that a sectarian group, perhaps the Essenes, was the residents of Qumran.

Acknowledgements

We are grateful to Jan Gunneweg for giving us the opportunity to work on this project. We also want to thank the wonderful people at the TU Delft: Ger Koper, Ruben Abellon and particularly Richard Bosma, for having the patience to help us work out the chemistry part. We want to thank Joris Dik for performing the tests on the ashes and sharing the results with us and Bob Ursem for his knowledge about plants. And last but not least Patrick van Rijn who helped us work out the practical aspects of making soap.

CHAPTER FIFTEEN

THE DEAD SEA, THE NEAREST NEIGHBOR OF QUMRAN AND THE DEAD SEA MANUSCRIPTS WHAT SEM, XRD AND INSTRUMENTAL NEUTRON ACTIVATION MAY SHOW CONCERNING DEAD SEA MUD

Jan GUNNEWEG
The Hebrew University of Jerusalem

Abstract. Dead Sea mud, famous for its therapeutic properties to the human skin is also an important ingredient in the understanding of the Dead Sea make-up and its ecological system. Some questions will be answered concerning the possibility to make the mud into a ceramic and what the Dead Sea may contribute to the degradation of the Dead Sea scrolls found in its proximity.

Keywords. XRF, XRD, SEM, Dead Sea mud

Introduction

One of the questions that have intrigued me for years concerns the possibility of making pottery from Dead Sea mud. The latter is a substance of various tinted layers that vary in color from black to gray and white that has been deposited on the floor of the Dead Sea for millennia. The material consists of rain flood-matter, organic as well as inorganic, that rushes from the Judean Hills, primarily a Dolomite-laden desert, into the lowest natural lake on earth, presently 418 meter below sea level.

The scope of this paper is twofold: 1) to study the question whether Dead Sea mud is suitable to make ceramic and 2) since the environment of the Dead Sea area is imperative to be studied because of its impact on the conservation of the Dead Sea scrolls that depends among others also on what the scrolls went through the past 2000 years. It will have also its impact in case Dead Sea water was used to prepare parchment locally.

Because of the proximity of the Dead Sea to the Qumran settlement where sectarian Jews once lived, I argued that if they were such a pure society as depicted in the writings of Flavius Josephus, Pliny the Elder and Philo of Alexandria and also described in the cache of Jewish manuscripts that have been found on and near the Qumran spur, it would have taken care to obtain pure materials to produce their daily household ware, mainly ceramic containers of all shape and size. The Dead Sea mud, being covered by salt water could have been a good candidate as prime material for potter's clay.

At present, this mud is smeared on tourist's faces and remaining parts of their body, whereas entrepreneurs use the stuff in a cleaned form as prime material for the fabrication of soap, gel, crèmes and bath salt, rich in minerals that are excellent for the skin.

The Dead Sea area is rich in oxygen because it is so low (at present minus 418 meters below sea level) and the air is clean because the minerals in the Dead Sea are not contaminated. Sun heat evaporates Dead Sea water and together with the high-pressure oxygen keeps the sun's ultraviolet rays out that are dangerous for one's skin. A haze hangs over the sea, whereas this sea water as well as its mud is laden with minerals and sulphur that are extremely beneficial for one's skin, blood circulation, arthritis and nervous system, to get rid of youth acnes and wrinkles, and of course it is the only real remedy against psoriasis. Combine all this with the "wonder" of being able to float on the Dead Sea without to drown, and one has THE source of health and wonder.

The primary constituents of Dead Sea water and mud are the chlorides of magnesium (Mg), potassium (potash, K), sodium (Na), and ions of bromine (Br). Also the amount of calcium is high that derived from calcareous rock of the surrounding deserts, in Israel and Jordan.

Dead Sea mud obtains its black color from natural bitumen that derives from degenerated organic material that arrives at the Dead Sea together with various forms of silicates.

Although various firms of soaps and cleanser and creams have studied the products from the Dead Sea, such as its water and Dead Sea mud, most data have remained in the drawers of the companies' laboratories that deal with the Dead Sea.

My idea was to open the Dead Sea research to a broader audience. First, I intended to make a ceramic of the Dead Sea mud because for me it was possible that the Qumranites used it to have pure clay from the sea. In spite of the fact that serious scholars told me that it was

impossible to make a ceramic from Dead Sea mud, I boldly took mud and levigated it in a water bath. Heavy material was deposited on the bottom of a bowl whereas the lighter one, supposedly clay in suspension, was tapped off to another recipient where it was set to evaporate. After some days the clay/marl settled on the bottom. The latter was modeled into a small inkwell similar to real inkwells found in Qumran and into a circular brick that were fired to 720/730 degrees Celsius in an electric kiln (Chapter 4, figure 10 in this book). The result was a change of color from black to buff and it became ceramic, as least to the look and the feel of it.

A second part of the same lump of Dead Sea mud was also levigated, but remained unfired in order to detect what would be the difference in chemical composition between the fired and unfired mud.

1. *Nuclear data*

In Budapest at the Nuclear Reactor of the University of Technology and Economics, I convinced my colleague Marta Balla in 1998 to analyze two samples, one of unfired, the other of fired mud by instrumental neutron activation analysis (INAA). She accepted and the results are shown in Table 1. (for a description of what INAA is, I refer to Chapter 4 in this book).

Some interesting items can be extrapolated from these analyzes.

It was detected that the fired mud shows an increase in Ca of 30 percent. Nevertheless, all other elemental abundances in the fired mud are higher than in the unfired mud so that Ca cannot be explained by using a dilution factor, which would point to the opposite; namely that the values of the fired mud would be less in abundance than in the unfired one.

Barium in fired mud is high (800 ppm) and twice that of the unfired one. No explanation could be found for this phenomenon.

Bromine in the fired mud is half that of the unfired mud (1668). This can easily be explained because Br is a volatile element and by heating it, evaporates. This explains also the Br in the Dead Sea atmosphere.

A possible conclusion for the lower abundances of all elements in fired mud is that the latter, by just firing it, looses 30 percent of organic matter that unfired mud might contain.

Table 1. Elemental chemical composition of Unfired and Fired Dead Sea mud by INAA. All values are in the parts-per-million range if not indicated otherwise.

Unfired Mud		Fired Mud
Ba	400	800
Ca%	6.2	9.5
Ce	20	31
Co	5.5	8.9
Cr	62	101
Fe%	1.3	2.2
Hf	3.2	0.7
La	11.1	17.4
Lu	0.2	0.3
Na%	1.5	0.8
Rb	25	62
Sc	3.8	5.9
Sm	2.1	3.4
Th	2.6	3.4
U	3.5	4.8
Yb	1.3	0.1
Zn	85	82
Br	1650	830

The next step is to see whether also XRD corroborates this conclusion.

2. *From nuclear to synchrotron*

Knowing the "shortcomings" of INAA, such as not being able to analyze quartz (Si) and aluminum (Al), I asked the help of Wim Bras at the European Synchrotron Radiation Facility (ESRF) at Grenoble for getting an answer to the presence of the major elements, such as Al, Mg, P and S and Si in Dead Sea mud.

Wim Bras heads the DUBBLE CRG station at the ESRF in Grenoble, a station that encompasses especially scientists from Holland and Belgium, but not solely.

Bras suggested to apply first plain X-Ray Fluorescence to the mud samples and asked Irina Snigireva (ESRF) to do the analyzes. The following spectra are the results of the unfired and the fired Dead Sea mud.

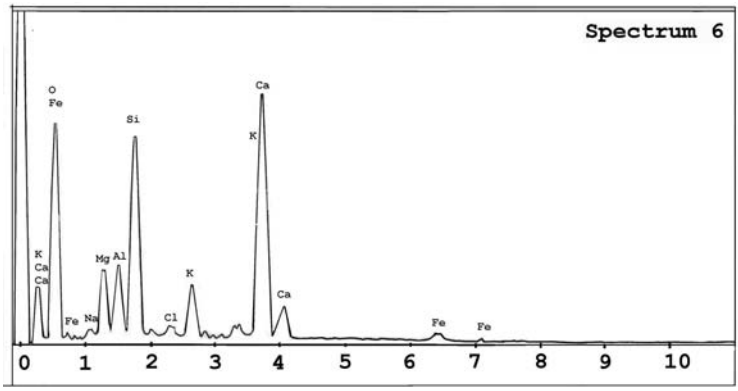

Figure 51. Spectrum 6—one of the six spectra taken—(with peaks of Cl, Ca, Fe, Na, Mg, Al, Si, S, Cl, K, Ca and Fe from left to right). Spectrum 6 shows data of Fired Dead Sea mud.

Unfired Dead Sea mud results

Three measurements were performed, first on Unfired Dead Sea mud (Spectra 1-3), and then three on Fired mud. In all, six spectra were obtained. The spectra are available for those who want to see them. Please, write me an email.

Hereunder, Spectrum 6 will be presented, since Spectrum 6 is the full scale of 1178 counts. One can see the various Ca peaks, Na, Cl, Mg and Si, as well as minor peaks of sulphur (S) and iron (Fe).

Figure 51. Spectrum 6—one of the six spectra taken—(with peaks of Cl, Ca, Fe, Na, Mg, Al, Si, S, Cl, K, Ca and Fe from left to right). Spectrum 6 shows data of Fired Dead Sea mud.

Wouter van Beek quantizied and corrected the data of Spectrum 6 by normalizing all analyzed elements. The elements will be expressed in atomic%.

First the normalized data of Spectrum 6 will be shown (full array of counts), and thereafter the normalized data of 3 spectra of unfired mud vis-à-vis those of 3 spectra of fired mud.

Spectrum 6 showed the following values (atomic%):

Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe
2.36	10.85	9.27	28.05	x	1.26	7.44	1.64	36.8	2.37

(x means not measured)

In the following paragraph is shown the individual average +/" standard deviation (M+/- S) for each three spectra of measurements of unfired and fired mud.

Unfired mud

Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe
(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M± S)	(M±S)	(M±S)
x	3.5±0.3	6.4±0.3	48.4±1.2	1.12	x	x	0.9±0.2	37.3±0.9	2.7±0.05

Fired mud

Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe
(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M±S)	(M± S)	(M±S)	(M±S)
1.43±0.03	10.9±0.1	7.2±0.3	26.8±1.1	x	1.4±0.1	7.7±0.3	1.7±0.2	39.1±2	2.8±0.4

It is seen that Spectrum 6 in figure 51 has a constant higher abundance for each element than what is expressed in the section dealing with data of Fired Mud.

Nota bene: When one wishes to compare the INAA elemental results with the same elements that were measured by XRF, one has to 'correct' the latter with a gravimetric factor from element to its oxide or visa versa. Each factor is different for each element.

When one compares the various spectra taken from the unfired as well as of the fired Dead Sea mud one notices that the abundances of magnesium (mg) is higher in fired than in unfired mud, which may mean that Mg increases when heated (3.5 versus 10.9%). A different phenomenon was observed in Br, which is volatile when heated

Si in Unfired mud is almost twice that in Fired mud (48% versus 26.8%)

Finally, by comparing Spectrum 6 (full counts) of Fired mud with the averaged values of 3 spectra of Fired mud above, it is seen that they are statistically similar within 2 standard deviations for the elements Mg, Al, Si, S, Cl, K, Ca and Fe.

3. *Conclusion*

The mineral content of the mud may be summarized as follows: Gilles Mertens, Ruben Snellings and Sam Hertsens identified the minerals in

the diffractogrammes (with phase identification). Due to the low resolution, no diffragtogramme has been depicted, but can be obtained by writing the author an email.

It was learned that Unfired Dead Sea mud consists primarily of calcite, quartz and opal CT (cristobalite-tridymite), with a small quantity of heulandites (zeolite).

On the contrary, the Fired Dead Sea mud consists primarily of quartz and calcite and various calcium aluminates ($Ca_3A_{l2}O_6$, $Ca1_2Al1_4O_{33}$)

The conclusion, therefore is, that the quantity of clay minerals is probably small as a result of which, the clays could not be identified. By the process of firing the Dead Sea mud in the kiln, the (semi-) amorphous silica (de opal CT) was converted into crystalline quartz. Calcite re-combined with the present Al- Al-containing phases to calcium aluminates, which will give the end product the look of a real ceramic, although it is not.

All this suggests that the analyzed Dead Sea mud is no "real clay" but rather a fine-grained chalkstone or marl (marl limestone), a conclusion that never could have been provided with INAA data alone.

The conclusions regarding the environment of the Dead Sea area will have to be studied thoroughly. In this paper, some indications are offered that are needed if one researches the Dead Sea scrolls that have been two millennia in the Dead Sea environment laden with all its minerals.

Acknowledgements

My sincere thanks to Wim Bras at he EDRF who was interested from the very start in the Dead Sea mud project and who pushed it through. I further thank the following scientists who all have worked together on a very short notice and in a short time on the analyzes: Gilles Mertens, Ruben Snellings, Sam Hertsens, Irina Snigireva and Wouter van Beek all at the ESRF in Grenoble, France. Last but not least, thanks are due to my colleague Marta Balla at the nuclear reactor facility at the University of Technology and Economics in Budapest for the INAA results on Dead Sea mud.

EPILOGUE

At the end of the Qumran Proceedings, there are two items to focus on:

1. Together with the present amount of pages, the total amount of scientific research applied to Qumran since 2003 by our team alone, reaches now more than thousand pages, divided over three volumes.

2. Scholars who have collaborated amount to 130 people. They are from 45 institutions covering 19 countries, 16 in Europe, and 3 in other continents, Israel, New Zealand and the United States.

It becomes clear that with such statistics, the idea of repetition of collaboration, as referred to in the preface and introduction, trans disciplinarity will become essential in humanistic and scientific circles. One knows the value of all artifacts treated in this volume, but there is an added value when they are properly studied. The added value is achieved by trans disciplinarity as shown throughout the book.

It is our wish and vision that this collaboration will stay alive and that the present authors as well as new ones will get the inspiration of collaborating with each other.

A similar approach as our Qumran study can also be executed on every other topic anywhere on earth. One has just to start the research.

It is this continuous handshake policy that keeps collaboration going. We are sure of that. The present Qumran research proves it.

PUBLICATIONS

List of publications of authors in the present Proceedings who have already contributed to the scientific Qumran research.

I. *Books*

J-B. Humbert and J. Gunneweg (eds.) *Khirbet Qumran et 'Ain Feshkha II ,Studies of Anthropology, Physics and Chemistry*, (Academic Press Fribourg; Vandenhoeck & Ruprecht, Göttingen (2003);

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INDEX

AAA-procedure to clean parchment and textile, 99, 106, 111-115
Accelerator Mass Spectroscopy (AMS), 99, 102, 107
Adriaens, Annemie, iii, 6, 11, 185
AMS. *See* Accelerator Mass Spectroscopy
amulet, 5
Animal bone heaps/piles, 39, 65-69
Archaeometry, 41, 57, 163

Balla Marta, 1, 7, 8, 39, 77, 135
bentonite, 92, 150, 151.
binder for ink, 77, 78, 80-82, 90, 91, 95, 125, 132
bone burials, 39, 65-69
borit, Hebrew for soda, 167-168

C14 dating, 5, 47, 63, 71, 99-118, 155
calibration of XRF, 126
calibration of C14, 99-100, 102-105, 108-112, 118, 141, 142
castor oil, 8, 99, 111-112, 115-118, 124, 157, 161
caves of Qumran,
  total 100, 40
  with pottery, 11
  with written materials, xvii, 3, 29, 32-33, 41, 67, 111-112, 124, 125, 128
cemetery of Qumran, xvii, 7, 30-32, 35, 64, 68, 114, 167
clay, 2, 7, 34, 40-46, 49, 51-58, 148-150, 177
coins, 11-19, 145-146
combs, 29, 32
contamination DNA, 70, 71
contamination in dating, 99, 100, 102, 106, 108, 111-118, 161
corrosion, 11, 12, 15-18, 22, 60
COST Action G8, xii, 19, 59, 143, 185
COST Action D-42, xii, 19
CT (computed tomography). *See* Tomography
dating, 63, 99-102, 105, 107-115, 117, 118, 124, 135, 138-141, 157, 161
De Vaux, Roland Guerin, 1, 2, 8, 24, 29, 30, 58, 63, 96, 145-147, 149, 164, 167, 171
Dead Sea, 65, 78, 79, 126, 131, 146, 158, 163, 164, 171, 175-177, 181
Dead sea scrolls, ix, xi, 2, 3-7, 9, 21-23, 35, 39, 41, 67, 68, 77, 79-82, 94, 96, 99, 104, 111, 112, 115-118, 123, 129, 132, 136, 164, 171, 175, 181
degradation of parchment, 21, 23, 25, 77-85, 87-93, 95, 124, 127, 175
Dendrochronology, 103, 104, 110
deterioration, 7, 23, 25, 77
Dik, Joris, xi, 21, 163, 173
DNA, 3, 7, 63-65

Electrophoresis, 69, 137
Engedi, 7, 9, 39, 44, 45, 54, 55, 58
epigraphy, ix
Epilogue, 183
ESRF (European Synchrotron Radiation Facility), 7, 23, 24, 84, 85, 178
European Synchrotron Radiation Facility (ESRF), 7, 23, 24, 84, 85, 178
Exegesis, x, 155

female presence, 3, 29, 32, 34, 35
flood, 40, 42, 54-55, 57
follicle, 25, 133, 155, 156, 158-161
Fourier Transform Infrared (FT-IR), 77, 81, 85, 127
FT-IR spectrography, 7, 77, 81, 85, 92-94, 123, 127, 130-132, 150

Galor, Katharina, 32
gender, 32
Gunneweg, Jan, iii, 1, 11, 21, 39, 63, 77, 135, 145, 163, 175

hairnet, 32
Hebrew University of Jerusalem, 41, 65.
Hebron, 40, 46, 56, 57

Honour Class Leiden/Delft 2008, 8, 163, 169

INAA. *See* Instrumental neutron activation analysis
Infrared microscopy, 77, 84-85, 93-95
ink, 3, 7, 22, 24, 40, 67, 77-91, 94-96, 123, 125, 129-132, 150, 156-162, 167
inkwell, 40-43, 48, 50-51, 55-57, 81, 83, 92, 150, 177
Instrumental neutron activation analysis (INAA), 39-56, 65, 140
Israel map, xvi

Jericho, 46, 51, 56, 58
Jerusalem, 1, 2, 5, 8, 41, 44, 46, 47, 49, 50, 55
jewelry, 31, 32, 36, 38

Kachila Bar-Gal, Gila, 65, 67
Ketef Hinnom, 5
Khirbet Qumran, 67, 68, 71, 74, 75, 112, 115, 125, 146, 167
kiln waster, 39-42, 53, 56-58

Laminography, 4, 7, 21
Law of repeated games, 1
lime, 78, 81, 91, 96
Lorentz Center, xi, 1-3, 42, 65, 163, 169
lye, 8, 163-169, 172-173

Magnetic Susceptibility, 149, 154
maps, xvi, xvii, xviii, 137, 147
marl (type of clay), 2, 7, 39-43, 48-51, 54-57, 177, 181
Masada,7, 9, 39, 45, 46, 49, 55, 59, 72
Masoretes, 5
Micro-X-ray tomography, 4, 7, 21 24. *See* also Laminography
Middle East Map, xvi
mitochondrial DNA, 67, 69-71
Monotheism, x, 4
Motsa Clay Formation, 46, 50, 51, 55-57
mud of Dead Sea, 8, 32-33, 40, 43, 53-54, 175-181
Murphy, Bridget, 7, 77

Netherland Institute of Advanced Studies (NIAS), xi, 1, 3, 6, 42, 65, 163
Neutron tomography, ix, 3, 4, 7, 11, 15, 17-19
Nuclear Reactor, 1, 39, 40, 48, 177, 181

Oxalate, 84, 95

paint, 3, 22, 33, 92, 149, 150
Paleography, ix, 3, 4, 5, 124, 157
parchment, 3, 5-8, 21-23, 24-26, 67, 68, 77-96, 115-118, 123-130, 132, 155, 158-159, 161, 175
perfume, 29, 34, 35
plan Qumran, xvii, xviii, 137
pottery production, 39, 41
Preface, ix
Price-Gutfeld excavation, 43, 49, 74
provenance (provenience), 39, 49, 55, 59, 68, 72, 124, 132, 161, 166

Qumran map, xvii, 137, 147
Qumranites, 2, 40, 42, 51, 57, 58, 163, 165

Rabin, Ira, 123, 158
radiocarbon dating, 8, 63, 99, 112, 114, 124, 135, 138, 140-142
Rasmussen, Kaare, L., 42, 65, 99, 135, 145, 146
Reactor (Nuclear), 1, 39, 40, 48, 177, 181.

Scanning Electron microscopy (SEM), 8, 123, 127, 136, 145, 148, 150, 152, 153, 175
scribe, ix, 3, 4, 5, 161
scroll, 6, 21, 26, 29, 32, 77, 79-82, 111, 115, 129, 155-162
scroll jar, 41, 51, 56
SEM. *See* Scanning Electron microscopy
Settlement, 39-44, 55, 58, 63, 64, 68, 135, 137, 140, 147, 149, 151, 163, 167, 176
soap preparation, xi, 8, 163, 168, 169, 171-174, 176
South Plateau at Qumran, xvii, xviii, 2, 7, 39-40, 43-44, 49, 57, 63-64, 68, 71, 135, 137
Spectrography FT-IR, 7
Synchrotron Radiation (SR), ix, 7, 11-13, 21, 23, 25, 77, 81, 84-85, 95, 123, 127, 136, 178
Synchrotron-based micro-X-ray tomography, 77, 81

tartaric acid, 135, 137

textile, 3, 8, 33, 38, 78, 112-114, 186
Thermoluminescence dating (TL), 135
TL. *See* Thermoluminescence dating
Tomography, 3, 4, 7, 11, 15, 17-19, 21 24
Tov, Emanuel, 5, 8, 36, 155
trans-disciplinarity, ix-xi, 2-4, 185

umber, 92-93

Van der Plicht, Johannes, 8, 65, 71, 99, 109, 111, 118

waster (pottery), 39-42, 53, 56-58
wine jar 2, 35, 44-46, 49, 50, 135

X-Ray Diffraction, 8, 11, 12, 16, 18, 25, 145, 148, 150, 175, 178
X-Ray Fluorescence, 8, 25, 77, 78, 80, 91, 93, 94, 123, 126, 128, 130, 175, 178, 180
XRD. *See* X-Ray Diffraction
XRF. *See* X-Ray Fluorescence

COLOR PLATES

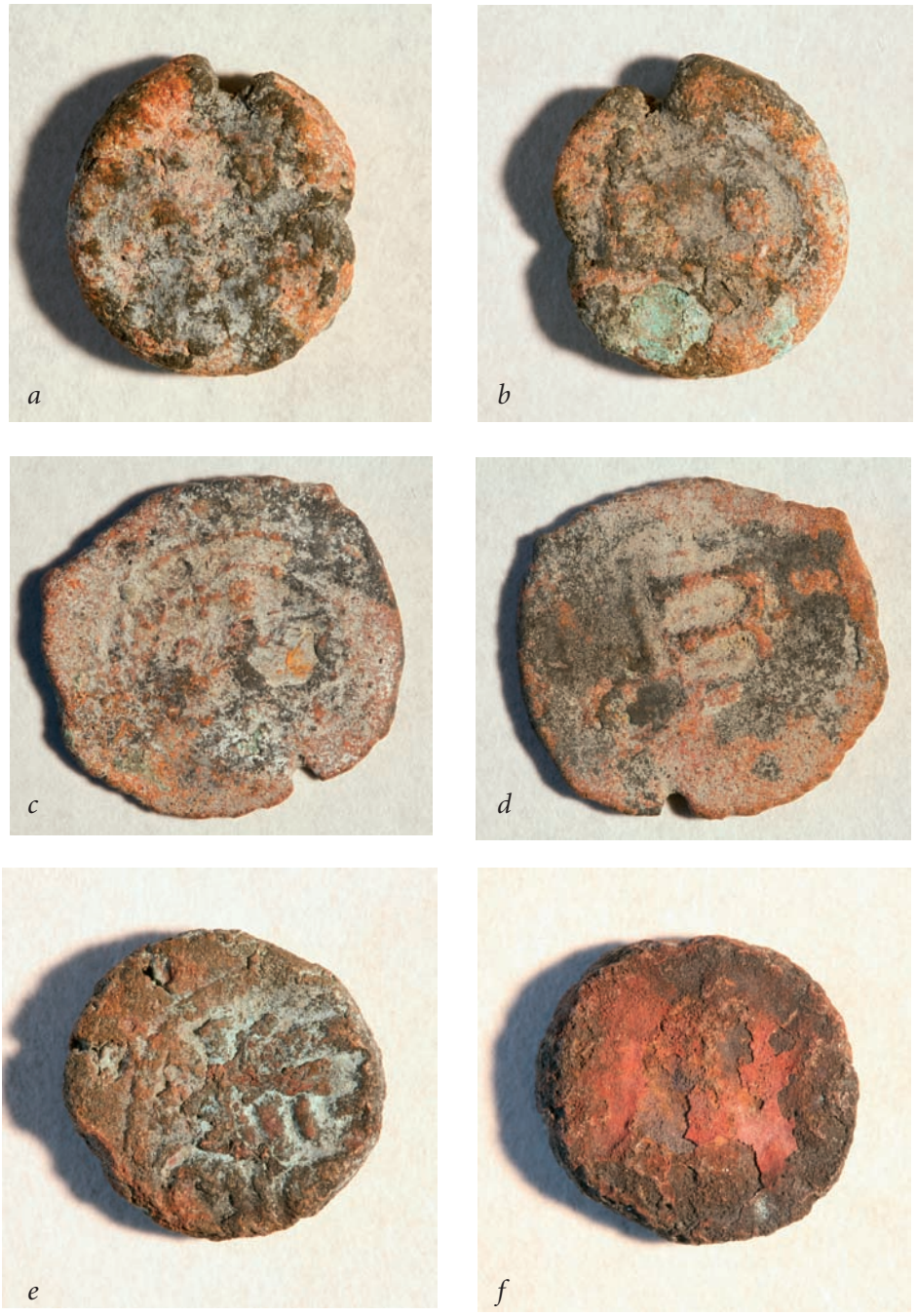

Figure I: figure 1. Photographs of the obverse (a, c and e) and reverse (b, d and f) of respectively Coins B, A and C.

Figure II: figure 4. Top row: photographs and neutron tomography images of the surface of obverse (left) and reverse (right) of Coin B. Middle row: idem for Coin A. Bottom row: idem for Coin C.

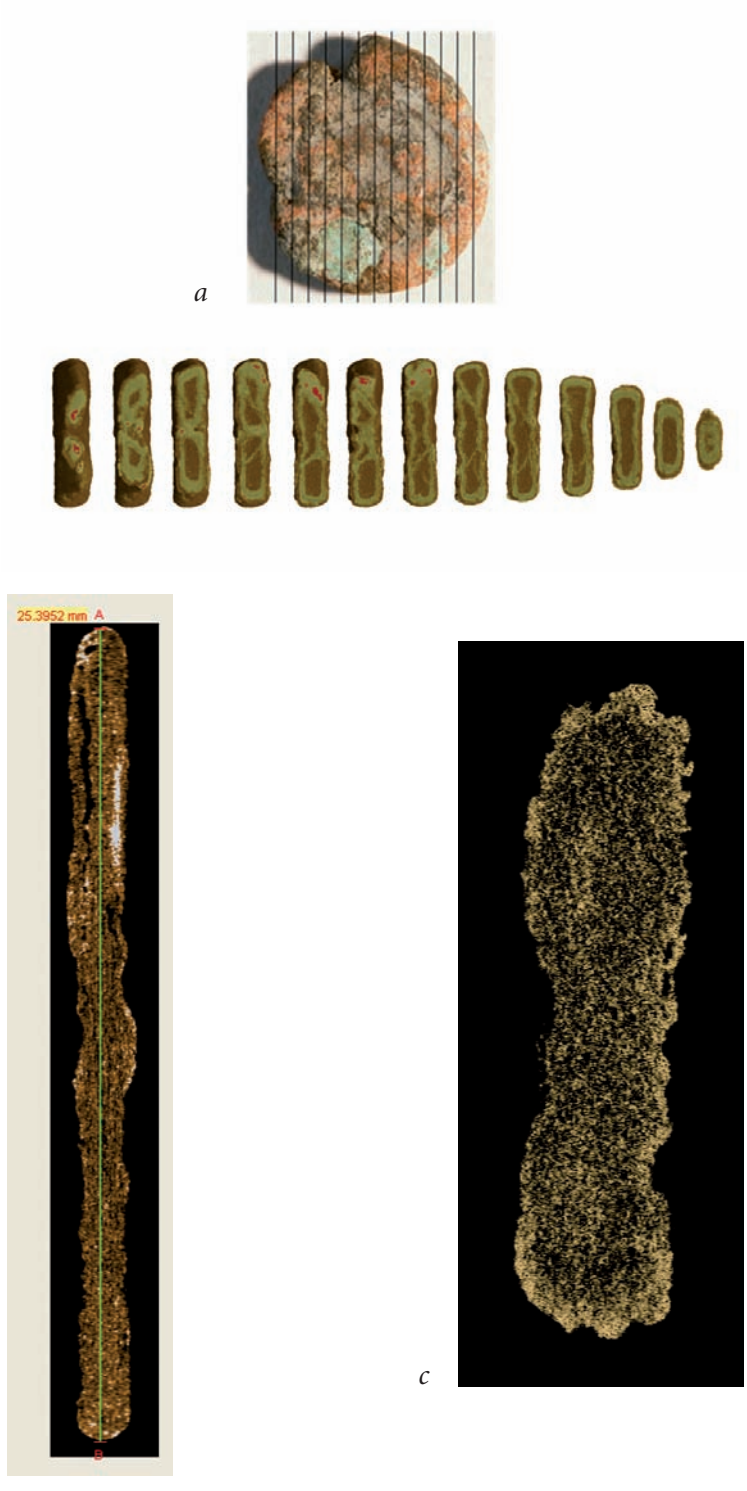

Figure III: figure 5. Neutron tomography images from inside the three coins.

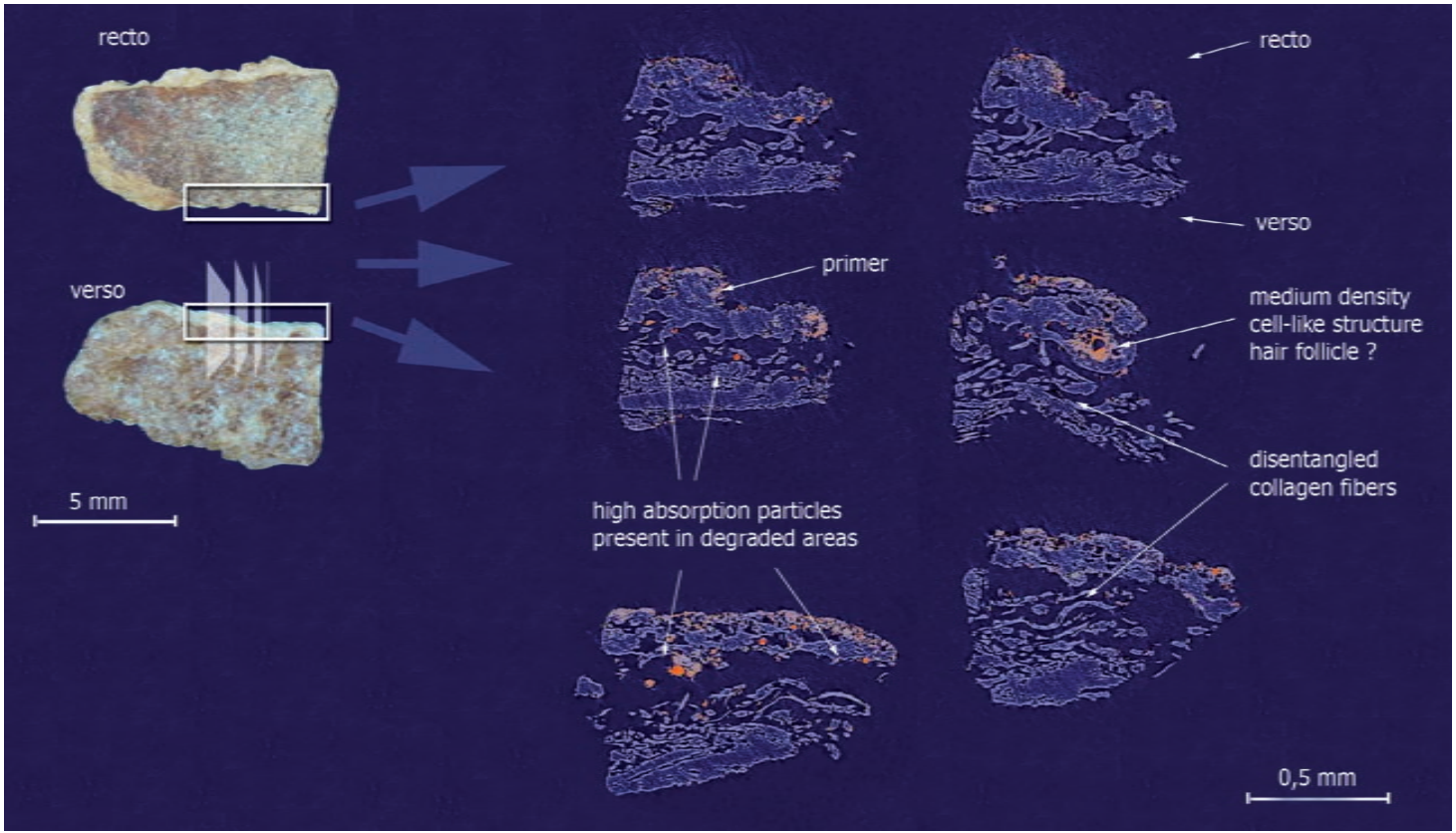

Figure IV: figure 6. On the left stereomicroscopic images of the Dead Sea scroll fragment photographed on both sides, showing a reddish primer on the recto side. The white frame indicates the area that was imaged with micro-CT. On the right several reconstructed slices. Their plane of view is indicated in transparence on the left.

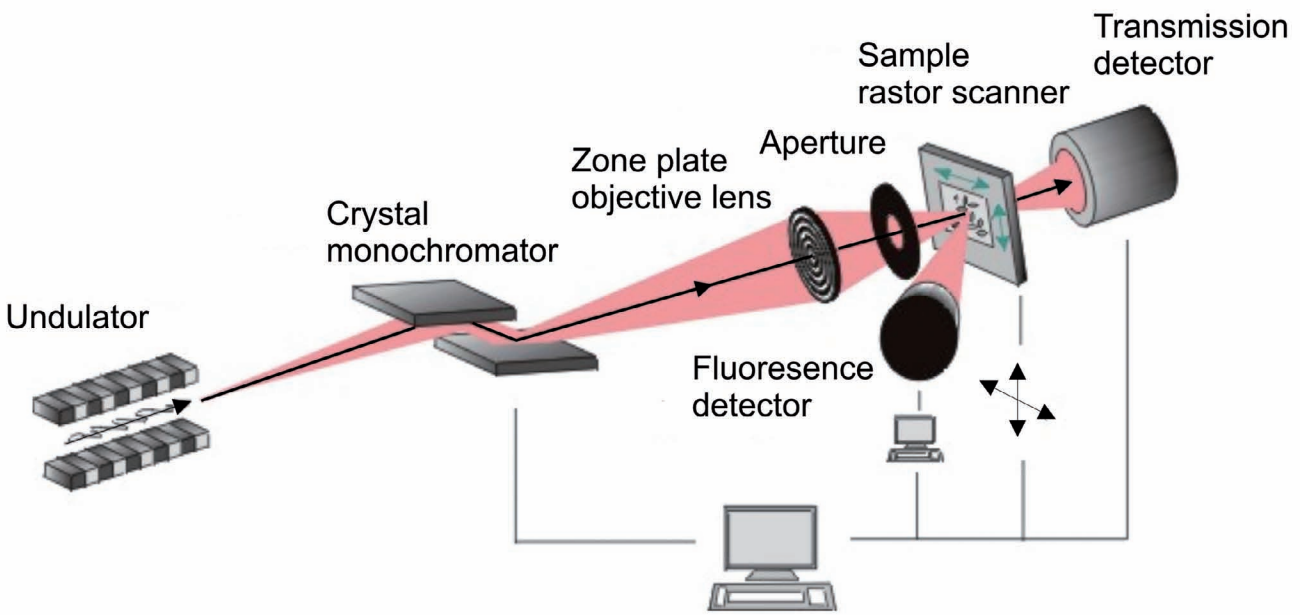

Figure V: figure 18. The experimental set up at the scanning X-ray microscope on ID21 at the ESRF.

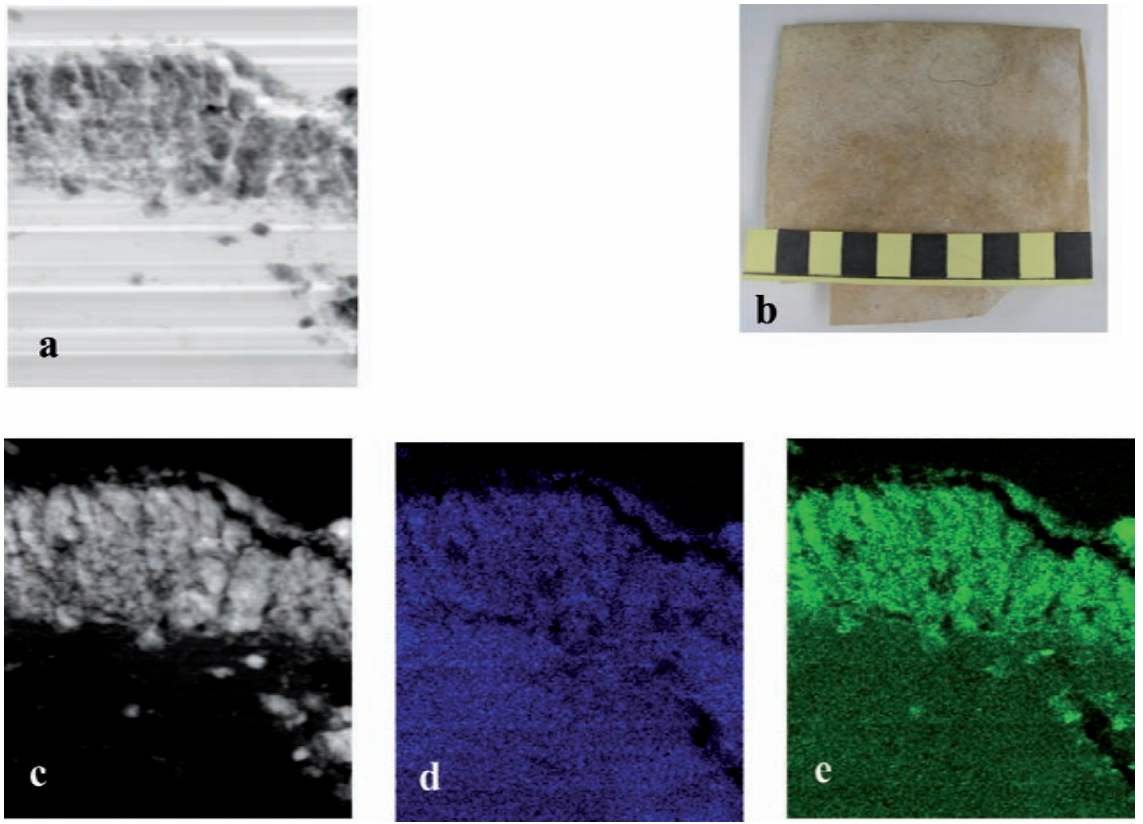

Figure VI a: figure 19. Absorption and fluorescence maps of modern goat parchment (QUM 1; 90 x 100 μm^2): (a) absorption profile (b) photograph of the fresh parchment sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map. It is possible to see the embedding resin surrounding the section of parchment in the absorption profile. Color photos in Figures VI a through c.

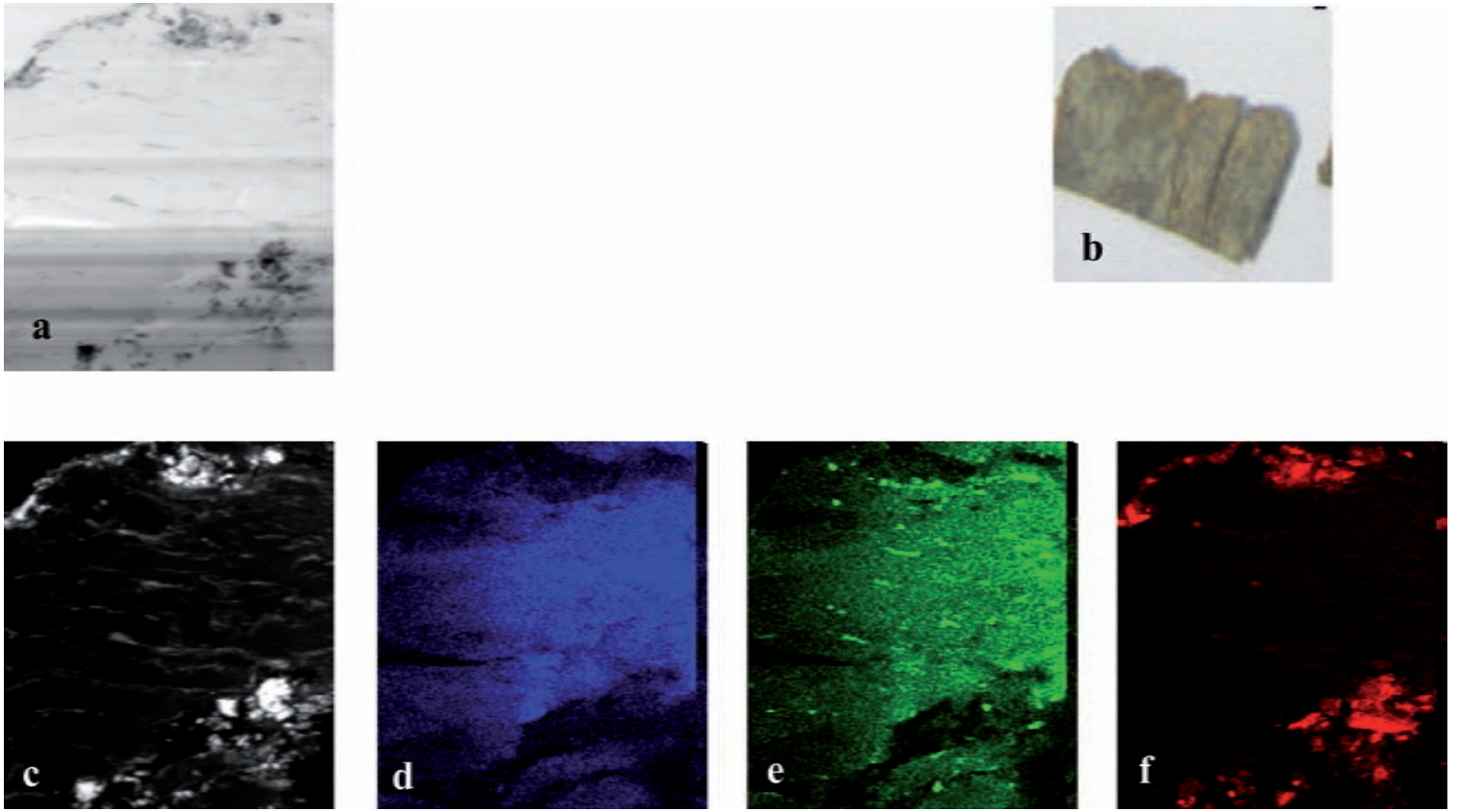

Figure VI b: figure 20. Absorption and fluorescence maps of Dead Sea parchment (QUM 3; 90 x 100 μm^2): (a) absorption profile (b) photograph of the sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map and (f) potassium.

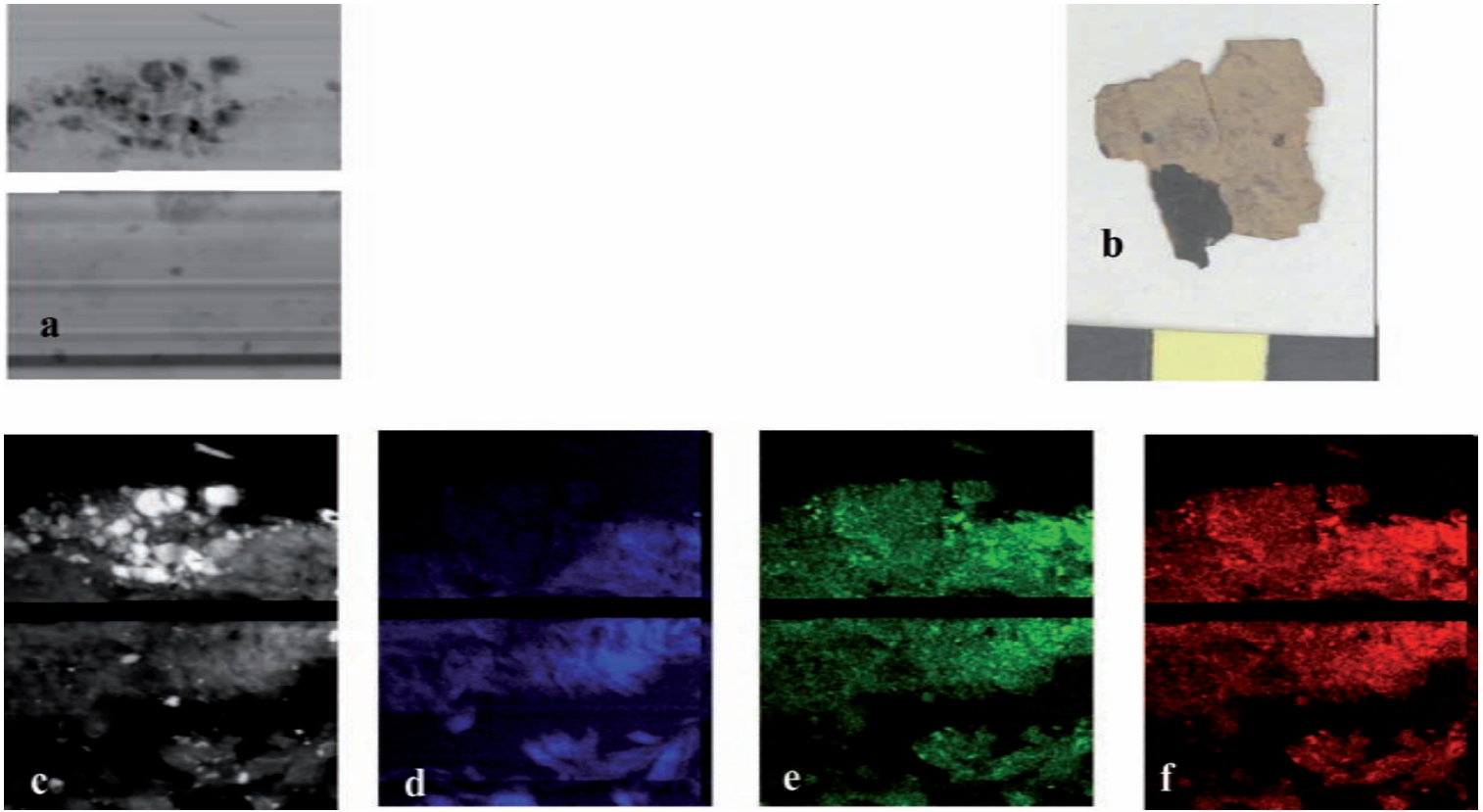

Figure VI c: figure 21. Absorption and fluorescence maps of an ink dot on Dead Sea parchment (QUM 4; 90 x 100 μm^2): (a) absorption profile (b) photograph of the sample that the cross-section was obtained from. (c) Ca map, (d) Cl map; (e) S map and (f) potassium. The white/black stripe running through all images is due to a loss of the X- ray beam during the measurements.

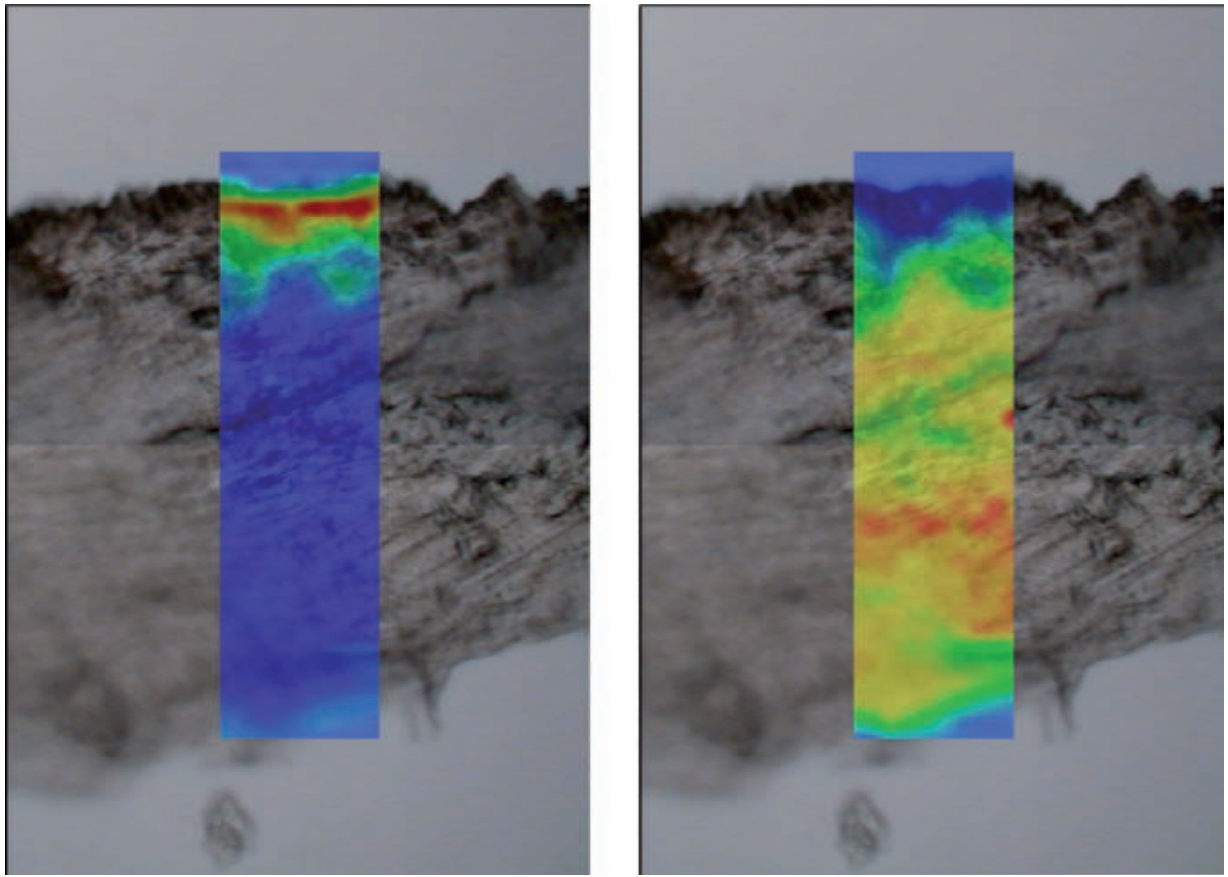

Figure VII: figure 26. Microscope images of a thin section of fresh parchment (QUM 1) with an overlay of FT-IR maps of carbonate band intensity (left) and amide I band intensity (right). FT-IR map size is $80 \times 272\ \mu m^2$ with 8 μm step size in the two directions.

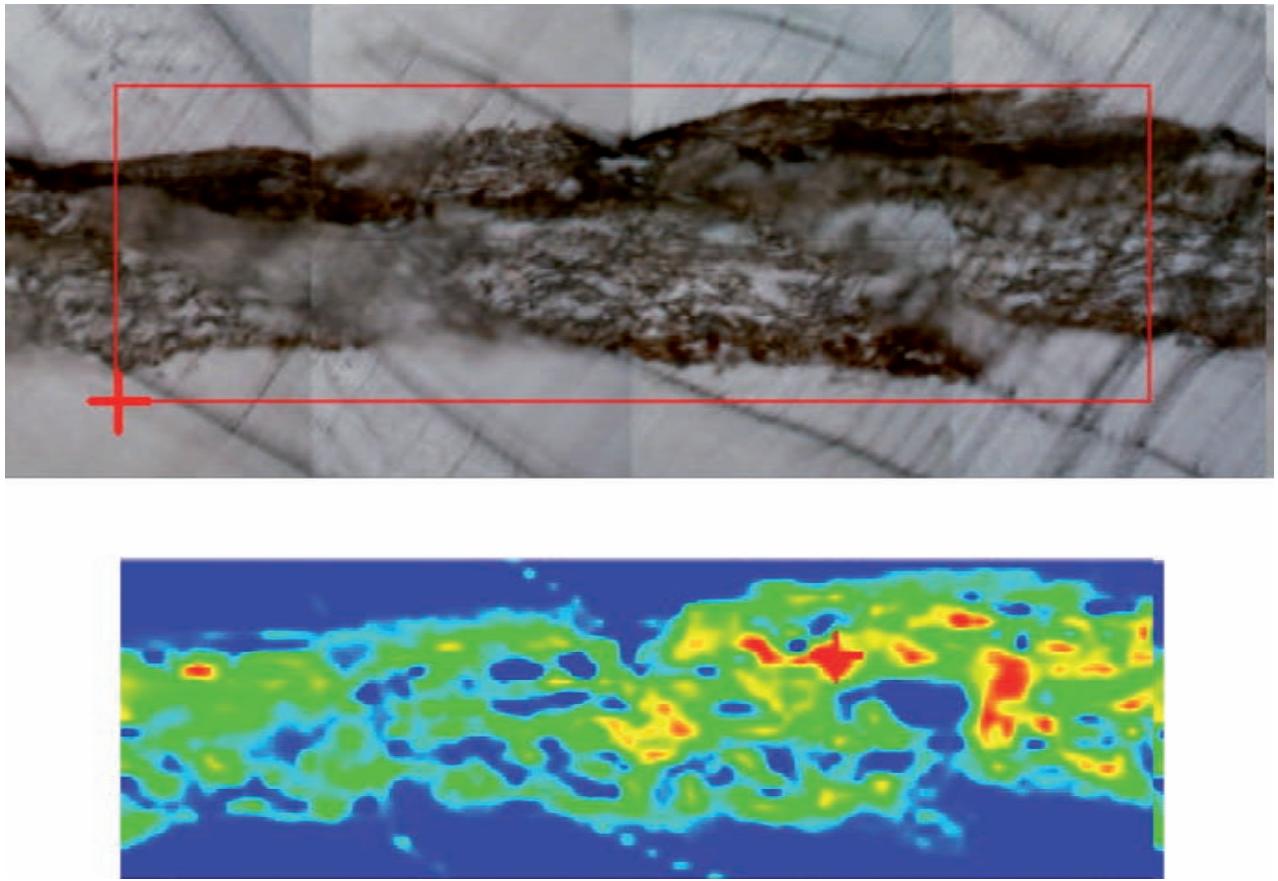

Figure VIII: figure 28. Microscope image of a thin section of QUM 7 (top) and amide II band intensity (bottom). FT-IR map size is $860 \times 270\ \mu m^2$ with 10 μm step size in the two directions. The ink dot is located on top of the section in the center (horizontal position: 6100 μm) with a hole well visible underneath the ink area.

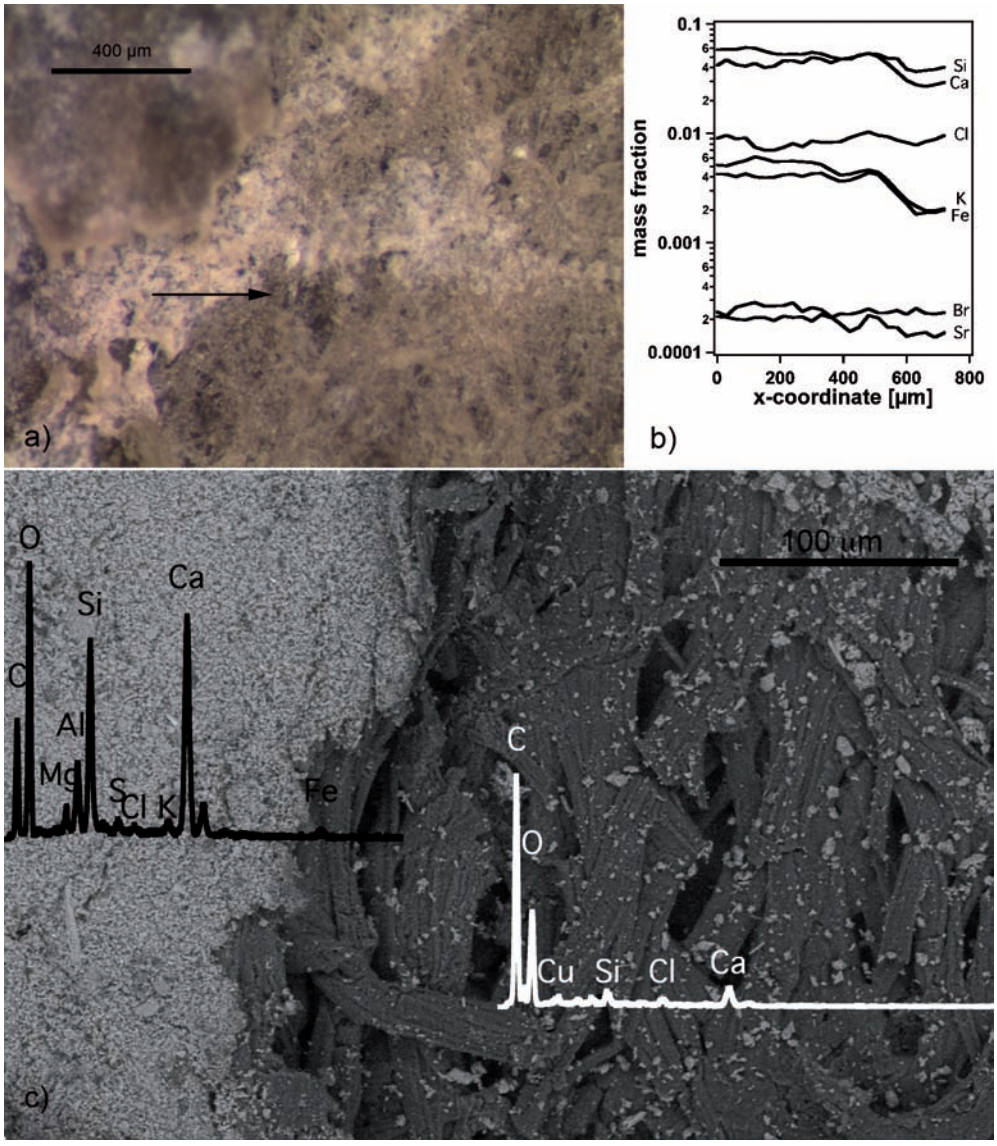

Figure IX: figure 34. Surface characterization of a Dead Sea Scrolls fragment from Cave 4: a) optical microscopic image of the transition from an area rich with mineral deposits to the area where fibers are exposed; b) element profiles across this border measured by X-ray.

Figure X: figure 46. An example of Hebrew script consisting of the Messianic Apocalypse text of the Dead Sea scroll 4Q521. This fragment was found in Cave 4 at Qumran. Photo Clara Amit, Courtesy of the Israel Antiquities Authority.

Figure XI. Wadi Qumran with caves 4 and 5 and remains of caves 7-9.

Figure XII. Qumran settlement with Tower seen from the S-W.