

METHODS IN MOLECULAR BIOLOGY

Series Editor

John M. Walker

School of Life and Medical Sciences

University of Hertfordshire

Hatfield, Hertfordshire, AL10 9AB, UK

For further volumes:

<http://www.springer.com/series/7651>

Nitric Oxide

Methods and Protocols

Edited by

Alexander Mengel and Christian Lindermayr

*Helmholtz Zentrum München—German Research GmbH, Center for Environment Health,
Institute of Biochemical Plant Pathology, Ingolstädter Landstraße 1,
Munich-Neuherberg, Germany*

 Humana Press

Editors

Alexander Mengel
Helmholtz Zentrum München–German
Research GmbH
Center for Environment Health
Institute of Biochemical Plant Pathology
Ingolstädter Landstraße 1
Munich-Neuherberg, Germany

Christian Lindermayr
Helmholtz Zentrum München–German
Research GmbH
Center for Environment Health
Institute of Biochemical Plant Pathology
Ingolstädter Landstraße 1
Munich-Neuherberg, Germany

ISSN 1064-3745 ISSN 1940-6029 (electronic)
Methods in Molecular Biology
ISBN 978-1-4939-7694-2 ISBN 978-1-4939-7695-9 (eBook)
<https://doi.org/10.1007/978-1-4939-7695-9>

Library of Congress Control Number: 2018935390

© Springer Science+Business Media, LLC, part of Springer Nature 2018

This work is subject to copyright. All rights are reserved by the Publisher, whether the whole or part of the material is concerned, specifically the rights of translation, reprinting, reuse of illustrations, recitation, broadcasting, reproduction on microfilms or in any other physical way, and transmission or information storage and retrieval, electronic adaptation, computer software, or by similar or dissimilar methodology now known or hereafter developed.

The use of general descriptive names, registered names, trademarks, service marks, etc. in this publication does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

The publisher, the authors and the editors are safe to assume that the advice and information in this book are believed to be true and accurate at the date of publication. Neither the publisher nor the authors or the editors give a warranty, express or implied, with respect to the material contained herein or for any errors or omissions that may have been made. The publisher remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Printed on acid-free paper

This Humana Press imprint is published by the registered company Springer Science+Business Media, LLC part of Springer Nature.

The registered company address is: 233 Spring Street, New York, NY 10013, U.S.A.

Preface

Nitric oxide (NO) is an important signaling molecule in humans, animals, plants, fungi, and bacteria, which is involved in the regulation of growth and development as well as stress response reactions. On the cellular level, NO is sensed through redox modifications of proteins. The most important mode of action of NO is protein S-nitrosation, the covalent attachment of NO to the thiol group of protein cysteine residues. Other major types of NO-dependent modifications are metal nitrosation and tyrosine nitration. Together, these protein modifications can lead to alteration of gene expression and/or direct metabolic changes and finally result in defined physiological responses. The past decade has seen an explosion in the number of articles relating to both the physiological and pathological responses evoked by NO.

Investigation of NO signaling is challenging because many available methods suffer from the lack of specificity for the parameter to be tested. Additionally, S-nitrosation is a thermodynamically unstable modification, impeding its direct detection. Therefore, NO research requires a broad set of complementary methods, which together allow the accurate identification of the functions of NO. In this volume, we tried to describe new methods for NO research with improved specificity and sensitivity compared to traditional methods.

This volume considers three main aspects of NO research and we hope this book is a useful resource for anyone with interest in NO research. Part I includes a protocol for NO treatment using chemical NO donors and several methods for NO detection using a NO-sensitive electrode, electron spin resonance, quantum cascade laser-based spectroscopy, membrane inlet mass spectrometry, and fluorescence-based NO sensor proteins. Part II focuses primarily on techniques to detect and identify NO-dependent modifications. This includes modified protocols of the biotin switch assay for detection, identification, and quantification of S-nitrosated proteins. Moreover, mass spectrometry, surface plasmon resonance, and antibody-based techniques are described to analyze low and high molecular weight S-nitrosothiols or tyrosine nitrated proteins. Additionally, the detection of nitro-fatty acids is described. In Part III of this volume protocols that are designed to measure S-nitrosothiol homeostasis and denitrosation activities are presented.

A special feature of this book is that it includes methods used in human/animal and plant NO research bringing together the experience from both fields. In many cases a transfer of the protocol from one system to the other system is possible with minor modifications. For example, the biotin switch technology is successfully used in human/animal and plant NO research using almost identical protocols. Techniques, which are based on analysis of protein extracts or recombinant proteins, might also be adapted easily. Others such as the detection of NO via NO sensor proteins, which was developed for the human/animal system, are very promising techniques for the plant system, but the transfer might be more challenging.

Although visible progress has been made in developing methods for NO research, future efforts should aim to further increase the sensitivity and specificity of those methods. In addition, many of the described techniques require sophisticated equipment not afford-

able to all NO labs. Better networking and a more open discussion on the problems associated with NO research will potentially lead to more robust and reproducible results in this important research area in the future.

München, Germany

*Alexander Mengel
Christian Lindermayr*

Contents

<i>Preface</i>	<i>v</i>
<i>Contributors</i>	<i>ix</i>

PART I NO TREATMENT AND DETECTION

1 A Simple and Useful Method to Apply Exogenous NO Gas to Plant Systems: Bell Pepper Fruits as a Model	3
<i>José M. Palma, Carmelo Ruiz, and Francisco J. Corpas</i>	
2 Measurements of Intra-oocyte Nitric Oxide Concentration Using Nitric Oxide Selective Electrode	13
<i>Sana N. Khan, Roohi Jeelani, and Husam M. Abu-Soud</i>	
3 Real-Time Imaging of Nitric Oxide Signals in Individual Cells Using geNOPS	23
<i>Emrah Eroglu, Helmut Bischof, Suphachai Charoensin, Markus Waldeck-Weiermaier, Wolfgang F. Graier, and Roland Malli</i>	
4 Detection of Nitric Oxide by Membrane Inlet Mass Spectrometry	35
<i>John M. Goodwin, Carol A. Chrestensen, and Ellen W. Moomaw</i>	
5 Quantum Cascade Lasers-Based Detection of Nitric Oxide	49
<i>Gracia Montilla-Bascón, Julien Mandon, Frans J. M. Harren, Luis A. J. Mur, Simona M. Cristescu, and Elena Prats</i>	
6 Detection of Nitric Oxide via Electronic Paramagnetic Resonance in Mollusks.	59
<i>Paula Mariela González and Susana Puntarulo</i>	

PART II NO MODIFICATIONS

7 Identification of S-Nitrosylated and Reversibly Oxidized Proteins by Fluorescence Switch and Complementary Techniques	73
<i>Alicia Izquierdo-Álvarez, Daniel Tello, J. Daniel Cabrera-García, and Antonio Martínez-Ruiz</i>	
8 A Proteomics Workflow for Dual Labeling Biotin Switch Assay to Detect and Quantify Protein S-Nitroylation.	89
<i>Heaseung Sophia Chung, Christopher I. Murray, and Jennifer E. Van Eyk</i>	
9 Surface Plasmon Resonance Spectroscopy for Detection of S-Nitrosylated Proteins	103
<i>Lili Zhang, Pengpeng Shang, Changbao Chen, Jie Zhou, and Shuhua Zhu</i>	
10 Measurement of S-Nitrosoglutathione in Plasma by Liquid Chromatography–Tandem Mass Spectrometry.	113
<i>Dimitrios Tsikas and Erik Hanff</i>	

11	Analysis of Recombinant Protein S-Nitrosylation Using the Biotin-Switch Technique	131
	<i>Sébastien Aimé, Siham Hichami, David Wendehenne, and Olivier Lamotte</i>	
12	Direct Measurement of S-Nitrosothiols with an Orbitrap Fusion Mass Spectrometer: S-Nitrosogluthathione Reductase as a Model Protein	143
	<i>Damian Guerra, Ian Truebridge, Stephen J. Eyles, Patrick Treffon, and Elizabeth Vierling</i>	
13	Identification of Tyrosine and Nitrotyrosine with a Mixed-Mode Solid-Phase Extraction Cleanup Followed by Liquid Chromatography–Electrospray Time-of-Flight Mass Spectrometry in Plants.	161
	<i>Mounira Chaki, Beatriz Sánchez-Calvo, Alfonso Carreras, Raquel Valderrama, Juan C. Begara-Morales, Francisco J. Corpas, and Juan B. Barroso</i>	
14	Electrophoretic Detection and Confocal Microscopic Imaging of Tyrosine Nitrated Proteins in Plant Tissue	171
	<i>Dhara Arora, Neha Singh, and Satish C. Bhatla</i>	
15	Identification of NO-Sensitive Cysteine Residues Using Cysteine Mutants of Recombinant Proteins	183
	<i>Azam Shekariesfablan and Christian Lindermayr</i>	
16	Detection of S-Nitrosated Nuclear Proteins in Pathogen-Treated <i>Arabidopsis</i> Cell Cultures Using Biotin Switch Technique	205
	<i>Azam Shekariesfablan and Christian Lindermayr</i>	
17	Nitric Oxide Analyzer Quantification of Plant S-Nitrosothiols	223
	<i>Adil Hussain, Byung-Wook Yun, and Gary J. Loake</i>	
18	Nitro-Fatty Acid Detection in Plants by High-Pressure Liquid Chromatography Coupled to Triple Quadrupole Mass Spectrometry	231
	<i>Capilla Mata-Pérez, María N. Padilla, Beatriz Sánchez-Calvo, Juan C. Begara-Morales, Raquel Valderrama, Francisco J. Corpas, and Juan B. Barroso</i>	
19	Bioinformatic Prediction of S-Nitrosylation Sites in Large Protein Datasets	241
	<i>Rosario Carmona, M. Claros, and Juan de Alché</i>	

PART III NO DETOXIFICATION

20	Biotin Switch Processing and Mass Spectrometry Analysis of S-Nitrosated Thioredoxin and Its Transnitrosation Targets.	253
	<i>Changgong Wu, Tong Liu, Yan Wang, Lin Yan, Chuanlong Cui, Annie Beuve, and Hong Li</i>	
21	Immunodetection of S-Nitrosogluthathione Reductase Protein in Plant Samples	267
	<i>Tereza Tichá, Lenka Lubová, and Marek Petřivalský</i>	
22	Thioredoxin-Dependent Decomposition of Protein S-Nitrosothiols	281
	<i>Sophie Kneeshaw and Steven H. Spoel</i>	

<i>Index</i>	299
------------------------	-----

Contributors

- HUSAM M. ABU-SOUD · *Department of Obstetrics and Gynecology, C. S. Mott Center for Human Growth and Development, Wayne State University, Detroit, MI, USA; Department of Biochemistry and Molecular Biology, Wayne State University, Detroit, MI, USA*
- SÉBASTIEN AIMÉ · *UMR 1347 Agroécologie, AgroSup Dijon, INRA, Univ. Bourgogne-Franche Comté, Dijon Cedex, France; Pôle Mécanismes et Gestions des Interactions Plantes Microorganismes, CNRS, Dijon Cedex, France*
- JUAN DE ALCHÉ · *Plant Reproductive Biology Laboratory, Department of Biochemistry, Cellular and Molecular Biology of Plants, Estación Experimental del Zaidín (CSIC), Granada, Spain*
- DHARA ARORA · *Plant Physiology and Biochemistry Laboratory, Department of Botany, University of Delhi, Delhi, India*
- JUAN B. BARROSO · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- JUAN C. BEGARA-MORALES · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- ANNIE BEUVE · *Department of Pharmacology, Physiology and Neuroscience, Rutgers, New Jersey Medical School, Newark, NJ, USA*
- SATISH C. BHATLA · *Plant Physiology and Biochemistry Laboratory, Department of Botany, University of Delhi, Delhi, India*
- HELMUT BISCHOF · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- J. DANIEL CABRERA-GARCÍA · *Servicio de Inmunología, Hospital Universitario de La Princesa, Instituto de Investigación Sanitaria Princesa (IIS-IP), Madrid, Spain*
- ROSARIO CARMONA · *Plant Reproductive Biology Laboratory, Department of Biochemistry, Cellular and Molecular Biology of Plants, Estación Experimental del Zaidín (CSIC), Granada, Spain*
- ALFONSO CARRERAS · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- MOUNIRA CHAKI · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- SUPHACHAI CHAROENSIN · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- CHANGBAO CHEN · *College of Chemistry and Material Science, Shandong Agricultural University, Taian, China*

- CAROL A. CHRESTENSEN · *Department of Chemistry and Biochemistry, Kennesaw State University, Kennesaw, GA, USA*
- HEASEUNG SOPHIA CHUNG · *Medicine and Heart Institute, Cedars Sinai Medical Center, Los Angeles, CA, USA*
- M. GONZALO CLAROS · *Departamento de Biología Molecular y Bioquímica, Universidad de Málaga, Málaga, Spain*
- FRANCISCO J. CORPAS · *Group of Antioxidants, Free Radicals and Nitric Oxide in Biotechnology, Food and Agriculture, Department of Biochemistry, Cell and Molecular Biology of Plants, Estación Experimental del Zaidín, Consejo Superior de Investigaciones Científicas, Granada, Spain*
- SIMONA M. CRISTESCU · *Department of Molecular and Laser Physics, Radboud University, Nijmegen, The Netherlands*
- CHUANLONG CUI · *Center for Advanced Proteomics Research and Department of Microbiology, Biochemistry and Molecular Genetics, Rutgers, New Jersey Medical School, Newark, NJ, USA*
- EMRAH EROGLU · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- JENNIFER E. VAN EYK · *Medicine and Heart Institute, Cedars Sinai Medical Center, Los Angeles, CA, USA*
- STEPHEN J. EYLES · *Department of Biochemistry and Molecular Biology, University of Massachusetts, Amherst, MA, USA*
- PAULA MARIELA GONZÁLEZ · *Facultad de Farmacia y Bioquímica, Físicoquímica, Universidad de Buenos Aires, Buenos Aires, Argentina; Instituto de Bioquímica y Medicina Molecular (IBIMOL), CONICET-Universidad de Buenos Aires, Buenos Aires, Argentina*
- JOHN M. GOODWIN · *Department of Chemistry and Biochemistry, Kennesaw State University, Kennesaw, GA, USA*
- WOLFGANG F. GRAIER · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- DAMIAN GUERRA · *Department of Biochemistry and Molecular Biology, University of Massachusetts, Amherst, MA, USA; Department of Obstetrics and Gynecology, University of Colorado, Aurora, CO, USA*
- ERIK HANFF · *Institute of Toxicology, Core Unit Proteomics, Hannover Medical School, Hannover, Germany*
- FRANS J.M. HARREN · *Department of Molecular and Laser Physics, Radboud University, Nijmegen, The Netherlands*
- SIHAM HICHAMI · *UMR 1347 Agroécologie, AgroSup Dijon, INRA, Univ. Bourgogne-Franche Comté, Dijon Cedex, France; Pôle Mécanismes et Gestions des Interactions Plantes Microorganismes, CNRS, Dijon Cedex, France*
- ADIL HUSSAIN · *Department of Agriculture, Abdul Wali Khan University, Mardan, Pakistan*
- ALICIA IZQUIERDO-ÁLVAREZ · *Servicio de Inmunología, Hospital Universitario de La Princesa, Instituto de Investigación Sanitaria Princesa (IIS-IP), Madrid, Spain; Biomechanics Section, KU Leuven, Leuven, Belgium*
- ROOHI JEELANI · *Department of Obstetrics and Gynecology, C. S. Mott Center for Human Growth and Development, Wayne State University, Detroit, MI, USA*

- SANA N. KHAN · *Department of Obstetrics and Gynecology, C. S. Mott Center for Human Growth and Development, Wayne State University, Detroit, MI, USA*
- SOPHIE KNEESHAW · *Institute of Molecular Plant Sciences, School of Biological Sciences, University of Edinburgh, Edinburgh, UK*
- OLIVIER LAMOTTE · *UMR 1347 Agroécologie, AgroSup Dijon, INRA, Univ. Bourgogne-Franche Comté, Dijon Cedex, France; Pôle Mécanismes et Gestions des Interactions Plantes Microorganismes, CNRS, Dijon Cedex, France*
- HONG LI · *Center for Advanced Proteomics Research, Rutgers and Department of Microbiology, Biochemistry and Molecular Genetics, New Jersey Medical School, Newark, NJ, USA*
- CHRISTIAN LINDERMAYR · *Institute of Biochemical Plant Pathology, Helmholtz Zentrum München – German Research Center for Environmental Health, Neuherberg, Munich, Germany*
- TONG LIU · *Center for Advanced Proteomics Research, Rutgers and Department of Microbiology, Biochemistry and Molecular Genetics, New Jersey Medical School, Newark, NJ, USA*
- GARY J. LOAKE · *Institute of Molecular Plant Sciences, School of Biological Sciences, University of Edinburgh, Edinburgh, UK*
- LENKA LUHOVÁ · *Department of Biochemistry, Faculty of Science, Palacký University in Olomouc, Olomouc, Czech Republic*
- ROLAND MALLI · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- JULIEN MANDON · *Department of Molecular and Laser Physics, Radboud University, Nijmegen, The Netherlands*
- ANTONIO MARTÍNEZ-RUIZ · *Servicio de Inmunología, Hospital Universitario de La Princesa, Instituto de Investigación Sanitaria Princesa (IIS-IP), Madrid, Spain; Centro de Investigación Biomédica en Red de Enfermedades Cardiovasculares (CIBERCV), Madrid, Spain*
- CAPILLA MATA-PÉREZ · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- GRACIA MONTILLA-BASCÓN · *Institute for Sustainable Agriculture, Spanish Council for Scientific Research (CSIC), Córdoba, Spain*
- ELLEN W. MOOMAW · *Department of Chemistry and Biochemistry, Kennesaw State University, Kennesaw, GA, USA*
- LUIS A.J. MUR · *Institute of Biological, Environmental and Rural Sciences, University of Aberystwyth, Aberystwyth, UK*
- CHRISTOPHER I. MURRAY · *Medicine and Heart Institute, Cedars Sinai Medical Center, Los Angeles, CA, USA*
- MARÍA N. PADILLA · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- JOSÉ M. PALMA · *Group of Antioxidants, Free Radicals and Nitric Oxide in Biotechnology, Food and Agriculture, Department of Biochemistry, Cell and Molecular Biology of Plants, Estación Experimental del Zaidín, Consejo Superior de Investigaciones Científicas, Granada, Spain*

- MAREK PETŘIVALSKÝ · *Department of Biochemistry, Faculty of Science, Palacký University in Olomouc, Olomouc, Czech Republic*
- ELENA PRATS · *Institute for Sustainable Agriculture, Spanish Council for Scientific Research (CSIC), Córdoba, Spain*
- SUSANA PUNTARULO · *Facultad de Farmacia y Bioquímica, Físicoquímica, Universidad de Buenos Aires, Buenos Aires, Argentina; Instituto de Bioquímica y Medicina Molecular (IBIMOL), CONICET-Universidad de Buenos Aires, Buenos Aires, Argentina*
- CARMELO RUIZ · *Group of Antioxidants, Free Radicals and Nitric Oxide in Biotechnology, Food and Agriculture, Department of Biochemistry, Cell and Molecular Biology of Plants, Estación Experimental del Zaidín, Consejo Superior de Investigaciones Científicas, Granada, Spain*
- BEATRIZ SÁNCHEZ-CALVO · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- PENGPENG SHANG · *College of Chemistry and Material Science, Shandong Agricultural University, Taian, China*
- AZAM SHEKARIESFAHLAN · *Iranian Research Institute of Plant Protection, Agricultural Research, Education and Extension Organization (AREEO), Tehran, Iran*
- NEHA SINGH · *Plant Physiology and Biochemistry Laboratory, Department of Botany, University of Delhi, Delhi, India*
- STEVEN H. SPOEL · *Institute of Molecular Plant Sciences, School of Biological Sciences, University of Edinburgh, Edinburgh, UK*
- DANIEL TELLO · *Unidad de Investigación, Hospital Santa Cristina, Instituto de Investigación Sanitaria Princesa (IIS-IP), Madrid, Spain; Centro de Investigación Biomédica en Red de Enfermedades Cardiovasculares (CIBERCV), Madrid, Spain*
- TEREZA TICHÁ · *Department of Biochemistry, Faculty of Science, Palacký University in Olomouc, Olomouc, Czech Republic*
- PATRICK TREFFON · *Department of Biochemistry and Molecular Biology, University of Massachusetts, Amherst, MA, USA*
- IAN TRUEBRIDGE · *Department of Biochemistry and Molecular Biology, University of Massachusetts, Amherst, MA, USA*
- DIMITRIOS TSIKAS · *Institute of Toxicology, Core Unit Proteomics, Hannover Medical School, Hannover, Germany*
- RAQUEL VALDERRAMA · *Group of Biochemistry and Cell Signaling in Nitric Oxide, Department of Experimental Biology, Center for Advanced Studies in Olive Grove and Olive Oils, Faculty of Experimental Sciences, University of Jaén, Jaén, Spain*
- ELIZABETH VIERLING · *Department of Biochemistry and Molecular Biology, University of Massachusetts, Amherst, MA, USA*
- MARKUS WALDECK-WEIERMAIER · *Molecular Biology and Biochemistry, Gottfried Schatz Research Center, Medical University of Graz, Graz, Austria*
- YAN WANG · *School of Pharmacy, Shanghai Jiao Tong University, Shanghai, P. R. China*
- DAVID WENDEHENNE · *UMR 1347 Agroécologie, AgroSup Dijon, INRA, Univ. Bourgogne-Franche Comté, Dijon Cedex, France; Pôle Mécanismes et Gestions des Interactions Plantes Microorganismes, CNRS, Dijon Cedex, France*
- CHANGGONG WU · *Center for Advanced Proteomics Research and Department of Microbiology, Biochemistry and Molecular Genetics, Rutgers, New Jersey Medical School, Newark, NJ, USA*

LIN YAN · *Center for Advanced Proteomics Research, Rutgers and Department of Microbiology, Biochemistry and Molecular Genetics, New Jersey Medical School, Newark, NJ, USA*

BYUNG-WOOK YUN · *School of Applied Biosciences, Kyungpook National University, Daegu, Republic of Korea*

LILI ZHANG · *College of Chemistry and Material Science, Shandong Agricultural University, Taian, China*

JIE ZHOU · *College of Chemistry and Material Science, Shandong Agricultural University, Taian, China*

SHUHUA ZHU · *College of Chemistry and Material Science, Shandong Agricultural University, Taian, China*