



SELECTED TECHNICAL PAPERS

STP1618

Editor: Michael J. Brisson

Detection Limits in Air Quality and Environmental Measurements

ASTM STOCK #STP1618 DOI: 10.1520/STP1618-EB

Library of Congress Cataloging-in-Publication Data

Names: Micheal, Brisson J., editor. | ASTM International Committee D22 on

Air Quality, host institution.

Title: Detection limits in air quality and environmental measurements / editor, Brisson J. Micheal.

Description: West Conshohocken, PA: ASTM International, 2019. | Series:

Selected technical papers; STP1618 | Includes bibliographical

references. | Summary: "THIS COMPILATION OF Selected Technical Papers,

STP1618, Detection Limits in Air Quality and Environmental Measurements,

contains peer-reviewed papers that were presented at a symposium held

October 25-26, 2018, in San Diego, California, USA. The symposium was

jointly sponsored by ASTM International Committee D22 on Air Quality,

and Subcommittee D22.01 on Quality Control, in cooperation with

Committee E11 on Quality and Statistics"- Provided by publisher.

Identifiers: LCCN 2019030407 (print) | LCCN 2019030408 (ebook) | ISBN

9780803176829 (paperback) | ISBN 9780803176836 (ebook)

Subjects: LCSH: Pollution-Measurement-Congresses.

Air-Pollution-Measurement-Congresses. | Environmental

monitoring-Statistical methods-Congresses. | Observed confidence

levels (Statistics)-Congresses. | Sensitivity theory

(Mathematics)-Congresses.

Classification: LCC TD193 .D48 2019 (print) | LCC TD193 (ebook) | DDC

628.5-dc23

LC record available at https://lccn.loc.gov/2019030407

LC ebook record available at https://lccn.loc.gov/2019030408

ISBN: 978-0-8031-7682-9

Copyright © 2019 ASTM INTERNATIONAL, West Conshohocken, PA. All rights reserved. This material may not be reproduced or copied, in whole or in part, in any printed, mechanical, electronic, film, or other distribution and storage media, without the written consent of the publisher.

Photocopy Rights

Authorization to photocopy items for internal, personal, or educational classroom use, or the internal, personal, or educational classroom use of specific clients, is granted by ASTM International provided that the appropriate fee is paid to the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/

ASTM International is not responsible, as a body, for the statements and opinions expressed in this publication. ASTM International does not endorse any products represented in this publication.

Peer Review Policy

Each paper published in this volume was evaluated by two peer reviewers and at least one editor. The authors addressed all of the reviewers' comments to the satisfaction of both the technical editor(s) and the ASTM International Committee on Publications.

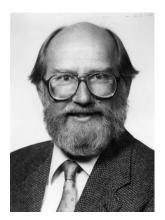
The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of the peer reviewers. In keeping with long-standing publication practices, ASTM International maintains the anonymity of the peer reviewers. The ASTM International Committee on Publications acknowledges with appreciation their dedication and contribution of time and effort on behalf of ASTM International.

Citation of Papers

When citing papers from this publication, the appropriate citation includes the paper authors, "paper title," in *STP title*, book editor(s) (West Conshohocken, PA: ASTM International, year), page range, paper doi, listed in the footnote of the paper. A citation is provided on page one of each paper.

Printed in Atlanta, GA November, 2019

Dedication



This publication, STP1618 on *Detection Limits in Air Quality and Environmental Measurements*, is dedicated to Dr. Harry L. Rook, our friend, colleague, Committee D22 "historian," and so much more. Harry passed away in March 2019 as we were close to finishing work on this STP. He joined ASTM International in April 1979 and served as Committee D22 Chairman on two occasions (1990–1995 and 2006–2007). He was passionate about family, nature, all things D22, and about the topic of this STP as author or coauthor for two of its papers.

Harry lives on in our hearts and our memories.

Foreword

THIS COMPILATION OF Selected Technical Papers, STP1618, *Detection Limits in Air Quality and Environmental Measurements*, contains peer-reviewed papers that were presented at a symposium held October 25–26, 2018, in Washington, DC, USA. The symposium was jointly sponsored by ASTM International Committee D22 on Air Quality and Subcommittee D22.01 on Quality Control, in cooperation with Committee E11 on Quality and Statistics.

Symposium Chair and STP Editor:

Michael J. Brisson Savannah River National Laboratory Aiken, SC, USA

Contents

Overview	ix
Detecting the Detection Limit	1
Thomas J. Bzik	
Fundamentals of the ASTM International Committee D22 Practice for	
Determination of the Detection Limit of a Well-Behaved Analytical Method	16
Michael J. Brisson and Harry L. Rook	
Limits of Detection and Quantification in Analytical Chemistry: A Brief Overview	
of the Currie Protocol	25
Harry L. Rook and Kevin Ashley	
ASTM E2677: International Standard on Limit of Detection for Trace	
Detectors—Explosives, Opioids, and Ozone	31
R. Michael Verkouteren, N. Alan Heckert, Stefan Leigh, Edward Sisco,	
James E. Norris, Jeffrey Lawrence, and Amber Burns	
Web-Based Calculator for ASTM E2677 Limits of Detection	49
N. Alan Heckert, R. Michael Verkouteren, and Keith Kwiatek	
Overview of Signal Detection Theory and Detection Limits	60
Thomas M. Semkow, Xin Li, and Liang T. Chu	
When Matrix Matters: A Case Study in Dirty Water and Clean Method	
Detection Limits	77
Richard Burrows, Nancy E. Grams, John Davis, and Louis P. Bull	
Impact of the Detection and Quantitation Limits on the Analytical Feasibility	
of Measuring the European Chemicals Agency Risk Assessment Committee's	
Recommendations for Occupational Exposure Limit Values for Nickel and	
Its Compounds in the Workplace	88
Steven Verpaele	

Handling Multiple Method Detection Limit Estimates: Which Statistical Estimate Should Be Reported? Thomas J. Bzik		
The Foundation for ASTM D6620, Standard Practice for Asbestos Detection Limit Based on Counts and Its Application as a Study Design Parameter Bertram Price	117	
Experimental Determination of Limits of Detection for Volatile Organic Compounds in Ambient Air Measured by Automatic Gas Chromatograph and Comparison with Reference Methods	134	
Sara Gutiérrez-Sama, Damien Bazin, Michel Robert, and Franck Amiet		
Detection Limit for Ra-228 in Drinking Water by Gamma Spectrometry Thomas M. Semkow, Abdul J. Khan, Traci A. Menia, Xin Li, Liang T. Chu, Miguel A. Torres, and Abdul Bari	146	
Censored Data and Statistics: How to Estimate Percentiles Anne Lotz, Justus F. Tulowietzki, Benjamin Kendzia, Tobias Weiß, Thomas Brüning, Thomas Behrens, and Dirk Taeger	161	
Treatment of Non-Detects Can Lead to Inaccurate Forensic Conclusions Melanie Edwards, Jaana Pietari, Linda Cook, and Paul Boehm	181	
Health Effects of Censored Elongated Mineral Particles: A Critical Review David Egilman, Joan E. Steffen, Triet Tran, Kate Clancy, Mark Rigler, and William Longo	192	

Overview

The concept of "detection limits," on the surface, would seem simple enough—what's the lowest value of "something" we can measure? The answer to that, at least in analytical chemistry, depends on the analytical method being used; for example, you can see more with a microscope than with the naked eye. It also depends on how *reliably* one can detect it. With the notion of reliability comes the need for statistics. For more than half a century, debates have taken place around what constitutes "detection," the degree of reliability with which that needs to be determined, and how to perform the necessary calculations.

In air quality and environmental measurements, these concepts can vary in meaning depending on technology, application, or stakeholder community. For example, stakeholders in the field of regulatory volatile organic compound measurements will define "detection limit" in a very different way from those interested in particulate, asbestos, or mold determinations. Until recently, a single U.S. federal agency had different meanings among different offices within the agency. In addition, navigating the concepts confronts stakeholders with a cornucopia of interrelated terms and abbreviations such as IDL, LCMRL, LDL, LOQ, MDL, PQL, RL, and more. These "limits," each of which means something different, have not served the overall science, or stakeholder communities (e.g., laboratories, their clients, or public policy makers), particularly well.

In 2015, ASTM International Committee D22 on Air Quality embarked on a mission to begin addressing these issues within its field, and in 2016 D22 hosted the first Conference on Detection Limits in Raleigh, NC, USA. That conference mainly served to identify the issues and seek consensus on how we might begin to address them. In addition to the topics described above, the conference also had presentations on a related topic known as "censored data." This refers to not reporting the numerical value obtained by an analysis if that value is below the detection limit and is a subject of controversy in its own right.

After the 2016 conference, D22 began developing a standard practice for determination of detection limits in air quality measurements, based on first principles and the need for sufficient statistical rigor while also being understandable to the laboratory personnel that would apply it. At the time of this writing, this practice remains a work in progress.

It was clear, though, that another symposium would be beneficial to advancing knowledge and continue the dialog that has been started. Thus, a Symposium on

Detection Limits was held on October 25–26, 2018, in Washington, DC. The symposium was sponsored by Committee D22 in cooperation with Committee E11 on Quality and Statistics. The primary goal of the symposium was to provide a forum to educate, discuss, and debate the various aspects of detection limits as applied to the various fields of environmental measurements and to determine where collaborative activity under the ASTM International umbrella can serve to simplify and coalesce some of the various terms. Publishing papers in this STP was also a key goal.

At this symposium, 21 presentations were made from experts from Belgium, Germany, France, and the USA. This STP comprises fifteen peer-reviewed papers from the symposium. They are arranged similarly to how they were presented in the symposium, as follows:

- Overview and Standards Development Activities
- Policy Issues and Mathematical Basis
- Applications and Recent Developments
- Censored Data

The symposium concluded with a panel discussion on censored data and limits of quantitation.

It is hoped that the papers in this STP will advance the science of determining detection capability by advancing understanding of the concepts as well as the controversies that remain, foster the dialog that is needed to obtain consensus that would benefit practitioners and policy makers, and establish the framework for development of new ASTM standards reflecting that consensus in Committees D22, E11, and others.

A heartfelt thanks to all presenters, manuscript authors, and peer reviewers for their contributions to this STP. Thanks also to ASTM staff for their efforts in keeping this project moving forward amidst many competing priorities.

> Michael J. Brisson Savannah River National Laboratory Aiken, SC, USA

ASTM INTERNATIONAL Helping our world work better

ISBN: 978-0-8031-7682-9

Stock #: STP1618 www.astm.org