Technical Committee and Subcommittee Reports

2017–2018 Report of the Technical Committee

Committee Members: M. Eurich, *Chair*; S. Brendecke; L. Barr; R. Jennings; E. Jorgenson; F. Fromuth; K. Lakenburges; A Mac-Leod; L. Nagle; C. Pachello; J. Palausky; A. Porter; N. Rettberg; L. Bech (EBC); and B. Foster (*senior advisor*).

The ASBC Technical Committee and Subcommittee chairs conducted a number of method evaluations through collaborative study, and coordinated a range of additional activities during 2017/2018. For this year, four new methods are recommended for inclusion in the ASBC *Methods of Analysis* (MOA):

- Beer Method 25B- Diacetyl, chaired by Robert Fulwiler (Fremont Brewing Co.).
- Sensory Analysis 16: Hop Grind Sensory Evaluation, chaired by V. Algazzali (John I. Haas).
- Sensory Analysis 17: Sensory Production Release, chaired by I. McLaughlin (Craft Brew Alliance).
- Sensory Evaluation 18: Tetrad Test, chaired by D. Bissmeyer (Wolf Group), Tara Teras (Wolf Group), and C. Lakenburges (AB/In-Bev).

In addition, the following methods will continue for another year of collaborative study in 2018/2019:

- FastOrangeTM Brett and Yeast Agar Detection, chaired by Guy Stewart (New Belgium Brewing Co.).
- Hop aroma compound analysis by GCMS, international method, chaired by Nils Rettberg (VLB-Berlin).

The ASBC Technical Committee regularly reviews each section of MOA. In 2017/18 review of one section of the ASBC Methods of Analysis was completed:

• Beer, chaired by Karl Lakenburges (Anheuser-Busch InBev) and Mark Eurich (New Belgium Brewing Co.)

In order to gather information on the requirements of the ASBC membership, the Innovative Methods Subcommittee organized roundtable discussions at the annual meeting in San Diego, CA. Aaron Porter (subcommittee chair) worked closely with the Technical Committee chairs to collect feedback for these breakout sessions and input from these roundtable discussions.

In addition, the following topic will undergo preliminary analysis and ruggedness testing prior with the possibility of collaborative study in 2018/2019:

 Beta Glucan in Wort by Automated Discreet Analysis, chaired by Aaron MacLeod (Hartwick College).

doi:10.1094/ASBCJ-2018-4916-01 © 2018 American Society of Brewing Chemists, Inc. • Free Amino Nitrogen in Wort by Automated Discreet Analysis, chaired by Aaron MacLeod (Hartwick College).

As in previous years, the following standing subcommittees continue:

- Innovative Methods, chaired by Aaron Porter (Sierra Nevada Brewing Co.).
- International Methods, chaired by Mark Eurich (New Belgium Brewing Co.)
- Craft Brew, chaired by Eric Jorgenson (Victory Brewing Co.).
- Sensory Science, chaired by Lindsay Barr (New Belgium Brewing Co.).
- International Hop Standards Committee, chaired by Bob Foster (MillerCoors).
- Packaging Methods, chaired by Scott Brendecke (MicroStar Logistics).
- Microbiological Methods in Brewing, chaired by Caroline Pachello (MillerCoors).
- Soluble Starch, chaired by Rebecca Jennings (Origin Malt).
- Lab Proficiency Program, chaired by Rebecca Jennings (Origin Malt), Aaron MacLeod (Hartwick College) and Carol Ericson (ASBC- Scientific Societies).

In 2017/18 the Technical Committee collaborated with the Brewers Association in video production to provide additional content to MOA. These videos were completed and published this year: Videos

- Calibration and Use of a Hydrometer
- Calibration and Use of a Density Meter
- Wort and Beer Sample Filtration
- Setting up a Microscope
- Yeast Cell Counting

No student grant evaluations were submitted in 2017/18.

The Technical Committee would like to thank the current subcommittee chairs for their hard work and dedication in conducting their respective collaborative studies during the past year. Furthermore we would like to formally acknowledge the many subcommittee members who have participated over the past year.

I would also like to recognize the dedication and hard work put forth by all members of the Technical Committee over the previous year. The continual enthusiasm and commitment demonstrated by the team is sincerely appreciated and I firmly believe is key to ensuring that the ASBC Methods of Analysis remains contemporary, relevant, and of exceptional practical value to the brewing community.

Innovative Methods

(Aaron Porter, aporter@sierranevada.com)

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

This is a standing subcommittee whose function is to collect, from various sources including polling members, new and alternate methods of analysis that may be useful for the industries our Society serves. These methods are reviewed to establish their merit and utility prior to evaluation.

International Methods

(Mark Eurich, <u>meurich@newbelgium.com</u>)

The function of this standing subcommittee is to encourage collaboration between ASBC and international brewing organizations. The primary focus is shared method collaboration with both BCOJ and EBC.

Craft Brew

(Eric Jorgenson, ericj@victorybeer.com)

The mandate of this subcommittee is to engage the craft brewing members of ASBC and explore opportunities to make the Society more relevant to these individuals. Additionally, the subcommittee aims to explore opportunities and pursue strategies to bring craft brewers who are not members of the Society into the ASBC.

Sensory Science

(Lindsay Barr, lBarr@newbelgium.com)

This is a standing subcommittee. It was formed on the recommendation of the Technical Committee to bring more focus to sensory science in ASBC and provide a forum for sensory scientists in the brewing industry to share and discuss current methodologies and propose new methodologies for collaborative testing.

International Hop Standards Committee

(Bob Foster, robert.foster@millercoors.com)

This subcommittee was formed in 1996 between the ASBC and EBC and is a standing Committee whose goal is to produce, purify, and verify isomerized and un-isomerized hop standards for the brewing, hops, and related industries.

Packaging Methods

(Scott Brendecke, sbrendecke@microstarkegs.com)

This is a standing subcommittee. It was formed to evaluate packaging methodology, review packaging methods within the MOA, and act as a liaison between ASBC and other packaging related organizations.

Microbiological Methods in Brewing

(Caroline Pachello, caroline.pachello@millercoors.com)

This subcommittee aims to evaluate novel methods for analysis of microbiological samples in brewing, including yeast and bacteria related assays. Individuals interested in contributing and/or participating in collaborative work are encouraged to contact Caroline Pachello directly.

Soluble Starch

(*Rebecca Jennings, rebecca@originmalt.com*)

This is a standing subcommittee whose goal is to coordinate a testing program for soluble starch that will ensure a consistent supply of quality soluble starch for the Society. To further this goal, the subcommittee monitors process methodology utilized in the production of starch, investigates improved methods for starch quality testing, and evaluates potential new suppliers of starch.

Lab Proficiency Program

(Rebecca Jennings, <u>rebecca@originmalt.com</u>, Aaron MacLeod, <u>macleoda@hartwick.edu</u>, and Carol Ericson, cericson@scisoc.org)

This is a standing subcommittee to ensure value and relevancy of the ASBC Check Sample Service This service provides subscribing members an opportunity to evaluate method accuracy and precision and instrument performance on a scheduled, regular basis. By comparing internal laboratory data to results from other laboratories around the world, a critical assessment of the analytical data generated by subscriber labs can be made and identification of areas for method improvement can be identified.

Hop Aroma Analysis by GCMS

(Nils Rettberg, nrettberg@vlb-berlin.org)

This subcommittee aims to develop methods for the analysis of hop aroma compounds using GCMS. Full details of this subcommittee will be confirmed in due course as well as international collaboration with the European Brewing Convention Analytical Committee.

MOA Review: Statistical Analysis of Samples

(Aaron MacLeod, macleoda@hartwick.edu)

This subcommittee has been initiated to provide guidelines for the statistical analysis of data related to brewery samples. The subcommittee will focus on comparison and validation of analytical methods through single and multi-laboratory studies. It will address topics such as identifying the appropriate statistical test to apply, dealing with outliers, and interpreting results. The primary goal is to prepare a set of methods and guidelines to assist the nonexpert in correctly analyzing data.

MOA 2.0

(Katie Fromuth, <u>katie.fromuth@colostate.edu</u> and Elizabeth Nagle <u>liz.nagle@cbrands.com</u>)

This is a new subcommittee. The subcommittee was formed to create supplemental content which will be associated with the most utilized Methods of Analysis (MOA) but in a separate format, which is under development. The purpose of the content is to be used to create method-specific training and troubleshooting tools that will enhance the methods currently in the MOA.



Report of SubCommittee

FastOrange[™] Brett and FastOrange[™] Yeast Agar Ring Study IM

Subcommittee Members: G. Stewart, *Chair;* M. Daniel; A. Garlit; R. Herndon; E. Jorgenson; K. Pawlowsky (EBC); C. Pull (EBC); C. Raleigh (EBC); A. Reilly (EBC); K. Syring; K. Taylor; P. Zeegers (EBC); and C. Pachello (*ex officio*).

Keywords: Beer Spoiling, Brewers Yeast, Brettanomyces, Contamination, Dekkera, Microbiology, Wild Yeast, Yeast

CONCLUSION

- 1. The sensitivity for correct detection of all strains tested for *Brettanomyces/Dekkera* yeast utilizing growth, color change, and microscopic evaluation for detection on FastOrangeTM Brett media was 100% by utilizing either a 5 or 7 day incubation at 25°C and was judged acceptable (Table 8).
- 2. The specificity for inhibition of all brewers yeast strains and *L. brevis* tested was 100% by utilizing either a 5 or 7 day incubation in conjunction with microscopic evaluation at 25°C and on FastOrangeTM Brett Media and was judged acceptable (Table 7). The *E. cloacae* strain tested resulted in 55% specificity for inhibition utilizing either a 5 or 7 day incubation (Table 7). This was judged acceptable with an understanding that some bacterial species will be resistant to inhibitors. Therefore, final written method must require that microscopic examination always be performed as part of the test method to differentiate growth as bacteria or *Brettanomyces/Dekkera* yeast.
- 3. FastOrangeTM Yeast recovered growth of all strains tested for *Brettanomyces/Dekkera* yeast and *Saccharomyces cerevisiae/pastorianus* brewer's yeast. The sensitivity for correct detection and microscopic identification of the tested sample cultures was 100% by utilizing either 5 or 7 days of incubation on FastOrangeTM Yeast and was judged acceptable (Table 10).
- 4. The specificity for inhibition of bacteria was 100% for *Lactobacillus brevis* by utilizing either a 5 or 7 day incubation in conjunction with microscopic evaluation at 25°C and is considered acceptable for FastOrangeTM Yeast Media and was judged acceptable (Table 9). The *E. cloacae* strain resulted in 64% specificity for inhibition utilizing either a 5 or 7 day incubation (Table 9). This was judged acceptable with an understanding that some bacterial species will be resistant to inhibitors. Therefore, final written method must require that microscopic examination always be performed as part of the test method to differentiate growth as bacteria or *Brettanomyces/Dekkera* yeast.

RECOMMENDATIONS

1. Result for the strain set tested for both FastOrange[™] Brett and FastOrange[™] Yeast are acceptable with 5 days of incubation. Additional yeast strains should be evaluated in a second round of testing for both medias for a smaller set of data to incorporate *Saccharomyces cerevisiae diastaticus*, *Pichia anaomola*, and *Saccharomyces cerevisiae boulardii* along with controls.

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

doi:10.1094/ASBCJ-2018-4922-01

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- 2. Controls will not be included in samples to be evaluated, separate samples will be sent and labelled as controls.
- **3.** Bacteria cultures will not be retested. The data from this ring study for bacteria will be referenced in the final report.
- **4.** Incubation time will be extended to 10 days per manufacturer instructions to observe any additional acid production or growth to enable additional information to be included in published test method.

This was the first year of the subcommittee's existence. The objective of this ring study was to analyze the effectiveness of PIKA FastOrange Brett and Yeast Agar media types. FastOrangeTM Brett Agar is designed to selectively culture and detect *Brettanomyces/Dekkera* yeast, while suppressing the growth of brewer's yeast and bacteria. FastOrangeTM Yeast Agar is designed to detect contamination of brewery products by wild yeast or mold.

The procedure is not reported, because it is under consideration for the Methods of Analysis.



Report of SubCommittee

LATERAL FLOW ASSAY FOR DEOXYNIVALENOL IN BARLEY

Subcommittee Members: M. Douglass, *Chair;* J. Brown; E. Cummings; E.Evink; J. Gillespie, C.Kapp,; A. Parks; A. Pieper; B. Roberts; , M. Rodriguez; K. Sich; M. Tess, R. Truland; and A. MacLeod (*exofficio*)

Need Key Words

CONCLUSIONS

1. Repeatability and reproducibility coefficients of variation for deoxynivalenol in barley by lateral flow assay ranged from 9.7 to 18.7% and 13.3 to 32.9%, respectively, and were judged acceptable.

RECOMMENDATIONS

- 1. The subcommittee recommends that the method for deoxynivalenol in barley by lateral flow assay be included in *Methods of Analysis*.
- 2. Discharge the subcommittee.

This is the subcommittee's first year of existence, started on the recommendation of the Innovative Methods subcommittee (reference). Quantitative lateral flow immunoreceptor assays (LFA) are a commonly used rapid method for measurement of mycotoxins in grain. Deoxynivalenol (DON), also known as vomitoxin, is extracted from ground grain using water. DON interacts with colored beads in the lateral flow test strip and the color intensity in the test zone is measured using a strip reader and interpreted as parts per million (ppm) DON. A collaborative test was required to determine repeatability

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

doi:10.1094/ASBCJ-2018-4934-01

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and reproducibility coefficients of variation for the new method prior to inclusion in the ASBC *Methods* of Analysis.

The full report appeared in the Journal of the American Society of Brewing Chemists.

https://doi.org/10.1080/03610470.2018.1562148

APPENDIX

DEOXYNIVALENOL CONTENT IN BARLEY BY LATERAL FLOW ASSAY

The specifics of this method are not included here, because it was published in the *Methods of Analysis* as Barley-11C.



Report of SubCommittee

BETA GLUCAN IN WORT BY AUTOMATED DISCRETE ANALYSIS

Subcommittee Members: A. MacLeod, *Chair*; C. Bains; R. Bond; S. Flager; J, Jones; E. Kraus; H Turner; and R. Jennings (*ex officio*).

RECOMMENDATIONS FROM THE PREVIOUS YEAR

This is the subcommittee's first year of existence, started on the recommendation of the subcommittee for *Innovative Methods*. Automation is employed in most malt testing facilities. Polling of the membership indicated sufficient interest in evaluating a method for determination of beta-glucan in wort using automated discrete analysis. Previous research has indicated that there is a strong correlation between results obtained using the Thermo Galley discrete analyzer and current reference method (1). A collaborative test is required to determine repeatability and reproducibility coefficients of variation for the method prior to inclusion in the ASBC Official Methods.

PRELIMINARY CONSIDERATION OF METHODOLOGY

A total of six malt samples representing three sample pairs (similar but distinct) and with a range of wort beta glucan levels will be sent to each collaborator. For each sample, the collaborator will prepare a congress mash according the method ASBC Malt-4 and determine wort beta glucan according to the attached method using their own discrete analyzer.

The procedure is not reported, because it is under consideration for the Methods of Analysis.

doi:10.1094/ASBCJ-2018-4947-01

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

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Report of SubCommittee¹

FREE AMINO NITROGEN IN WORT BY AUTOMATED DISCRETE ANALYSIS

Subcommittee Members: A. MacLeod, *Chair*; C. Bains; R. Bond; S. Flager; J. Jones; E. Kraus; H. Turner; and R. Jennings (*ex officio*).

RECOMMENDATIONS FROM THE PREVIOUS YEAR

This is the subcommittee's first year of existence, started on the recommendation of the subcommittee for *Innovative Methods*. Automation is employed in most malt testing facilities. Polling of the membership indicated sufficient interest in evaluating a method for determination of free amino nitrogen using automated discrete analysis. Previous research has indicated that there is a strong correlation between results obtained using the Thermo Galley discrete analyzer and current reference method (1). A collaborative test is required to determine repeatability and reproducibility coefficients of variation for the method prior to inclusion in the ASBC Official Methods.

PRELIMINARY CONSIDERATION OF METHODOLOGY

A total of six malt samples representing three sample pairs (similar but distinct) and with a range of wort beta glucan levels will be sent to each collaborator. For each sample, the collaborator will prepare a congress mash according the method ASBC Malt-4 and determine wort free amino nitrogen content according to the attached method using their own discrete analyzer.

The procedure is not reported, because it is under consideration for the Methods of Analysis.

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

doi:10.1094/ASBCJ-2018-4956-01

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Report of SubCommittee

SENSORY PRODUCTION RELEASE

Subcommittee Members: I. McLaughlin, *Chair*; S. Bennett; M. Peltz; A. Schultz; L. Barr (*ex officio*)

Keywords: Production release, go/no-go, ttb/not ttb

CONCLUSIONS

1. TTB/not TTB sensory assessments are the most straightforward way to routinely evaluate a large number of fresh samples in a production brewery. It is often the last, and most comprehensive, quality check performed on products before they begin their journey to the consumer. The use of a standardized methodology and adequately trained panelists is critical to consistent success. The method outlined below represents the scientific brewing community's most up-to-date approach to this crucial Quality Control assessment which should be considered a foundational aspect of a sensory lab in a production environment

RECOMMENDATIONS

- 1. The subcommittee recommends that the method for sensory production release be included in *Methods of Analysis*.
- 2. Discharge the subcommittee.

Sensory Production Release was added to the ASBC Methods of Analysis as Sensory Analysis 17.

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

doi:10.1094/ASBCJ-2018-4963-01

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Report of Subcommittee

HOP GRIND SENSORY EVALUATION

Subcommittee Members: V. Algazzali, *Chair;* S. Bennett; A. Benson; J. Beard; D. Bissmeyer; K. Fromuth; S. Gebhardt; H. Goodwin; T. Kostelecky; K. Payne; T. Pitra; C. Poirier; K. Nasiatka; T. Self; M. Zunkel; and L. Barr (*ex officio*).

Keywords: Hop Aroma, Rapid and Standardized Hop Preparation

CONCLUSIONS

The Hop Grind method was tested for sensitivity using the same hop material in both pellets and cones. In pellets and cones respectively, the Hop Grind method was found to have at least low sensitivity by 55% and 90% of the collaborators, moderate sensitivity by 9% and 75% of the collaborators, and high sensitivity by 9% and 45% of the collaborators.

RECOMMENDATIONS

- 1. The subcommittee recommends that the Hop Grind method be included in the *Methods of Analysis*, where it may be utilized as a rapid and standardized hop preparation method for the sensory evaluation of hop aroma.
- 2. Discharge the subcommittee.

The full report will appear in the Journal of the American Society of Brewing Chemists.

The specifics of this method are not included here, because it was published in the *Methods of Analysis* as Sensory Analysis 16.

doi:10.1094/ASBCJ-2018-4971-01

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

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Report of Subcommittee¹

VALIDATING THE SENSITIVITY OF THE TETRAD TEST AS COMPARED TO THE TRIANGLE TEST FOLLOW UP STUDY

Diane Bissmeyer, Tara Teras, Cindy-Lou Lakenburges, Co-Chairs

Subcommittee Members: Wolf Group's Trained Panel

Keywords: Tetrad Test, Triangle Test, Discrimination Tests

CONCLUSIONS

The Tetrad Test is a suitable replacement for the Triangle Test. Previous testing by this committee indicated that the Tetrad was more sensitive than the Triangle for samples with larger d' values. This study was designed to validate the sensitivity of the Tetrad for smaller d' values. For samples with smaller d' values, the Tetrad resulted in a 15% reduction in d' as compared to the Triangle test. This is less than the theoretical 33% reduction, indicating that the tetrad is more sensitive. The Tetrad also resulted in a higher proportion of correct responses, though the difference was not significant at the 95% confidence interval.

RECOMMENDATIONS

- 1. The subcommittee recommends that the Tetrad Test Method be published as a suitable alternative to the Triangle Test.
- 2. Samples with strong or lingering flavors may have too much carryover for the Tetrad to be effective. This was not a part of the current study, but should be a consideration in test selection.

This was the second year of the subcommittee's existence. The subcommittee was formed based on the recommendation of the ASBC Sensory Subcommittee to determine if the Tetrad Test is as sensitive as the Triangle Test.

doi:10.1094/ASBCJ-2018-4989-01

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

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The full report appeared in the Journal of the American Society of Brewing Chemists.

https://doi.org/10.1080/03610470.2019.1619323

The Tetrad Test was added to the ASBC Methods of Analysis as Sensory Analysis 18.



Report of Subcommittee

VALIDATING THE SENSITIVITY OF THE TETRAD TEST AS COMPARED TO THE TRIANGLE TEST

Diane Bissmeyer, Tara Teras, Cindy-Lou Lakenburges, Chairs

Subcommittee Members: Victor Algazzali, Patricia Aron, Samantha Bennett, Amanda Benson, Sami Hunt, Lindsay Kirchner, Kevin Payne, Meghan Peltz, Cassie Poirier, Hayley Potts, Anna Sauls, Kara Taylor

Keywords: Tetrad Test, Triangle Test, Discrimination Tests

CONCLUSIONS

The Tetrad Test is a suitable replacement for the Triangle Test. The Tetrad Test had a higher percentage of correct responses at more panel sites than did the Triangle Test.

At a confidence level of 95%, no significant differences were observed in the aggregate proportion of correct answers for Triangle Test versus Tetrad Test. The reduction of Effect Size (d') for the Tetrad was less than the theoretical reduction of 1/3, indicating that the Tetrad Test is slightly more powerful than the Triangle Test. The lower standard deviation of the Tetrad Effect Size indicates that it is more precise than that of the Triangle Test.

RECOMMENDATIONS

- 1. The subcommittee recommends that the Tetrad Test Method be published as a suitable alternative to the Triangle Test.
- 2. The subcommittee recommends that further testing be conducted to test the tolerances of the Effect Size (d').

This was the first year of the subcommittee's existence. The subcommittee was formed based on the recommendation of the ASBC Sensory Subcommittee to determine if the Tetrad Test is as sensitive as the Triangle Test.

doi:10.1094/ASBCJ-2018-4990-01

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited

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The full report appeared in the Journal of the American Society of Brewing Chemists.

https://doi.org./10.1080/03610470.2019.1619321

The Tetrad Test was added to the ASBC Methods of Analysis as Sensory Analysis 18.



Report of Sub Committee

BEER-25B UPDATE: SPECTROPHOTOMETRIC MEASUREMENT OF TOTAL VDK BY DISTILLATION AND COLORIMETRIC REACTION WITH α -NAPHTHOL AND CREATINE

Subcommittee Members: R. Fulwiler, *Chair;* S. Bruslind; C. Dwyer; K. Fromuth; C. Miller; K. Norman; J. Palausky, D. Russey; S. White; M. Wingert; and A. Porter (*ex officio*).

Keywords: 2-3-butanedione, 2-3-pentanedione, Diacetyl, Vicinal Diketones

CONCLUSIONS

Repeatability and reproducibility coefficients of variation for the determination of total Vicenal Diketones (VDK) by distillation and colorimetric reaction α -Naphthol and creatine ranged from 2.6 to 12.2% and 10.6 to 14.5%, respectively, and were judged acceptable.

RECOMMENDATIONS

- 1. The subcommittee recommends that the method for Spectrophotometric Measurement of Total VDK By Distillation and Colorimetric Reaction with α-Naphthol and Creatine (Beer-25B) be updated in *Methods of Analysis*.
- 2. Discharge the subcommittee.

This was the first year of the subcommittee's existence. Due to the amount of small craft brewery laboratories that do not have a Gas Chromatograph (GC), there has been an increased interest and usage in recent years of method Beer-25B Broad Spectrum Method for Vicenal Diketones (VDK) (1), originally published in the *Methods of Analysis* in 1964. ASBC membership was polled in 2016 (2), and it was determined by the ASBC Technical Subcommittee that there was enough interest to move forward with a collaborative to update the method with improvements and clarifications. The range of calibration standards was decreased to 25-250 µg/L to cover a more realistic range of fermentation samples. A filtration step to remove yeast was added to improve repeatability between samples. The method and apparatus sections were expanded to provide additional details that were not included in the original method: reagent preparation, dilution calculations, complete list of necessary apparatus. An additional section was added to explain calculations necessary for the method. This collaborative study was performed to determine the repeatability and reproducibility for the updated method.

doi:10.1094/ASBCJ-2018-4905-01

This report is published as submitted. The pages were numbered at the ASBC headquarters office, but the report was not edited.

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Report of Sub Committee

The full report will appear in the Journal of the American Society of Brewing Chemists.

The specifics of this method are not included here, because it will be published in the *Methods of Analysis* as Beer-25B.