Revised Methods of Diacetyl Analysis

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CONCLUSIONS

1. The specificity of the distillation method (Beer-25,A) could not be determined because of limited collaborator response.
2. The data generated by the broad-spectrum method (Beer-25,B) confirmed its lack of specificity for diacetyl.
3. Specificity of the micro method (Beer-25,C) could not be determined because of limited collaborator response.
4. Temperature, time, and pH were all identified as significant parameters that must be adequately controlled when performing the precursor conversion method.

RECOMMENDATIONS

1. Remove the distillation method (Beer-25,A) from Methods of Analysis and place it in the archives.
2. Encourage the membership to provide precursor conversion methods and sample handling procedures. Future subcommittee work could evaluate those methods for inclusion in Methods of Analysis.
3. Discharge the subcommittee.

This continuing subcommittee was charged (2) with two tasks: 1) performing ruggedness testing on one precursor conversion method selected for use during sample preparation and 2) conducting studies to determine the specificity of the distillation method (Beer-25,A), the broad-spectrum method (Beer-25,B), and the micro method (Beer-25,C) for the analysis of diacetyl (1).

PROCEDURE

Three samples pairs of packaged beer were prepared representing low, moderate, and high concentrations of diacetyl and pentanedione and were sent to each collaborator. The Youden unit block experimental design (3) was used.

Packaged commercial beer was spiked with standards of diacetyl and pentanedione. Collaborators were requested to use a standard precursor conversion that consisted of aeration by pouring the sample between two containers five times, placing it in a boiling water bath for 20 min, and equilibrating it to 20°C before analysis.

Collaborators were requested to analyze the samples using the distillation method (Beer-25,A), the broad-spectrum method (Beer-25,B), or the micro method (Beer-25,C), as published in Methods of Analysis. One of the cochairmen analyzed the samples using the gas chromatographic method (Beer-25,E) (1). Results generated by the collaborators were compared to the cochairman’s gas chromatographic data.

RESULTS AND DISCUSSION

Precursor Conversion Method

Ruggedness testing (1) was completed on the precursor conversion method for diacetyl, and the results are listed in Table I. Table II lists the main effects and the confidence intervals for each parameter evaluated.

Increasing the temperature from 92 to 100°C decreased the response by 0.018 mg/L. Raising the residence time in the water bath from 10 to 20 min increased the response by 0.025 mg/L.
The degree of aeration had no significant effect at the 95% confidence level.

Specificity Studies

Outlier at \( P \leq 0.01 \) based on totals and/or differences (1). Calculated excluding outliers.

**LITERATURE CITED**

