

Vicinal Diketones and Precursors

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CONCLUSIONS

Eight collaborators participated in evaluating a procedure for determining the total of vicinal diketones (VDK) plus VDK precursors. The results indicate large amounts of within-laboratory and between-laboratory error. The difficulties encountered with the tested method appear to be fundamental. It is unlikely that the Subcommittee would succeed with further testing of this procedure.

TABLE I
Vicinal Diketones plus VDK Precursors in Beer ($\mu\text{g/l.}$)

Collaborator	Sample A	Sample B
1	76.8	75.0
2	10.8	1.8
3	553.0*	313.0*
4	39.3	39.3
5	64.8	37.8
6	42.3	59.3
7	67.3	84.8
8	80.3	108.0
9	83.8	61.3
Average	54.5	58.0

*Excluded from average and statistical treatment.

RECOMMENDATIONS

The Subcommittee work should be continued for another year. The modified broad spectrum VDK method and gas chromatographic VDK determination, both as described by Gales (3), should be tested collaboratively.

Much previous work on the analysis of diacetyl and related compounds has been done, leading to the adoption of four official procedures in the ASBC *Methods of Analysis* (1). A recent survey conducted by the Subcommittee on Coordination of New and Alternate Methods of Analysis indicated that, while the existing methods appear to be satisfactory for diacetyl itself and for diacetyl plus 2,3-pentanedione, there was also interest in measuring VDK precursors and, especially, the total of VDK plus VDK precursors (2).

A two-step procedure can be used to determine the total of VDK and VDK precursors (4). Heating the sample in the presence of air converts VDK precursors into VDK. Any reliable method for estimating VDK concentration can then be used. The result represents the total of the VDK originally present as such plus the VDK precursors which were converted into VDK by the forcing step.

EXPERIMENTAL

A pair of beer samples, designated A and B, was analyzed and the data were treated by the unit block design of Youden (6).

The forcing procedure used was that described by Inoue and Yamamoto (4), while the determination of VDK was done by the

TABLE II
Statistical Summary of Results for VDK plus Precursors ($\mu\text{g/l.}$)

No. of Labs	Grand Mean (X + Y)/2	Within-Lab	Between-Lab	Total Error (S _t)	Calc. F Ratio (S _t /S _i)	Critical F (95%)	Coefficients of Variation (%)		
		Error (S _i)	Error (S _b)				S _i	S _t	S _b
8	58.3	15.1	37.3	54.9	13.23	3.79	94.2	25.9	64.0

UV-micro method of Rice *et al.* (5). In this method, the VDK is swept from the sample with nitrogen and trapped from the gas stream.

RESULTS AND DISCUSSION

Eight collaborators submitted results (Table I). Of these, one set of results was classified as an outlier and excluded from the calculations. The remaining results are quite scattered. This is reflected in the coefficients of variation in Table II. The total laboratory error coefficient of variation was 94%, which is far from acceptable. Within-laboratory performance was somewhat better than the between-laboratory results.

In an effort to identify the problems, some supporting data were examined. No collaborator encountered difficulty in preparing a standard curve and all were in reasonably good agreement. Two problem areas were noted. One collaborator reported widely differing values when four separate blank samples were prepared and analyzed. Considerable variation (53–90%) between labs was noted in the efficiency with which added diacetyl was swept from

the beer into the trap. This has a large influence on the magnitude of the results.

Acknowledgment

The Subcommittee wishes to thank J. F. Rice of the Miller Brewing Company (Milwaukee) who made many helpful suggestions and personally fabricated gas distribution manifolds for all members of the Subcommittee.

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