

Determination of Oxalate in Beer

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CONCLUSIONS

Although the collaborative results of the oxalate analyses on beer not spiked with oxalate were very promising, the recovery of added oxalate was quite poor. The overall improvement in the results was due mainly to the use of a single source of PAR reagent distributed by the Chairman. The method appears to require some work outside the scope of the Subcommittee.

RECOMMENDATIONS

It is recommended that the studies be temporarily suspended.

Oxalate in beer is normally determined by slow and inaccurate methods based on precipitation of the oxalate with calcium (2,4) or thorium (3). A gas chromatographic method (5) exists which determines oxalate along with other organic acids, but the same criticism applies. The Subcommittee on Calcium and Oxalate in Beer examined the method of Koch and Strong (4) in 1970, 1971, and 1972 but found it too complex and variable for consideration as a standard procedure. In 1973, Bernstein and Khan (1) proposed a

TABLE I
Recovery of Added Oxalate

Collaborator	Sample No.				% Recovery of Added Oxalate
	A1	B1	C1	D1	
1	16.0	16.0	20.0	20.2	41
	16.0	16.0	20.0	20.2	41
2					
3	16.5	16.6	19.0	19.5	28
4	15.6	15.4	19.0	19.5	38
	15.4	15.6	19.0	17.8	
5	16.0	16.0	20.0	21.0	45
6	20.0	20.0	23.0	23.0	30
7	19.6	20.0	25.0	24.0	47
	19.6	22.0	23.4	23.4	26
8	28.4	27.9	29.0	30.6	16
9	25.0	25.0	27.0	28.0	25
		23.0	29.0	26.0	45
10	38.0	36.0	37.0	35.0	0

rapid method based on spectrophotometric measurement of the decrease in absorbance caused by the oxalate ion on the red uranium (IV)-4-(2-pyridilazo)-resorcinol (PAR) complex.

PROCEDURE

At last year's results were again quite disappointing, the consensus before the Milwaukee annual meeting was that perhaps we should discontinue the studies.

However, at the closed seminar, after some discussion, it was decided that possibly the biggest source of error was in variations in the PAR reagent. It was suggested that the Chairman obtain a supply of PAR and distribute it to three or four collaborators along with some beer to be analyzed.

Eastman Chemicals was chosen as the source of the PAR reagent.

Each of the four collaborators was sent four bottles of beer, two labeled A and two labeled B. Sample B was a control and A had 10 mg/l. of oxalate added.

Three sets of results were obtained. Although the recovery of oxalate from the spiked samples was quite poor, the results on the unspiked beer were sufficiently encouraging to plan further studies. The possibility of error in the addition of oxalate was considered.

Accordingly, the whole committee was sent eight bottles of beer, two labeled A1, two labeled B1, two labeled C1, and two labeled D1. A1 and B1 were the same and C1 and D1 were the same. C1 and D1 were spiked with oxalate at a level of 10 mg/l. oxalate ion. Each collaborator was also sent a supply of PAR reagent obtained from Eastman Organic Chemicals. Sodium oxalate was used as the source of the added oxalate ion.

The collaborators were asked to analyze the samples by the Bernstein and Khan method (1) taking all the precautions that evolved from our previous studies.

RESULTS AND DISCUSSION

In the preliminary work carried out by three collaborators to investigate the PAR source, the results obtained are as follows:

	Sample A		Sample B		% Recovery
Collaborator 1	20.1	20.1	15.3	15.3	48.0
Collaborator 2	23.0	23.0	16.1	16.6	64.0
Collaborator 3		21.0		15.0	60.0

Nine sets of results were received on the work carried out by all of the committee and the results are shown in Table I.

The recovery rate of the added oxalate ion is quite poor. On examining the spiked beer after sitting for about a month, a fair amount of oxalate crystals was noted. Sodium oxalate should be quite soluble in beer. The Chairman ran some experiments in which the more soluble potassium oxalate was added but the recoveries, although better, were still quite poor, *i.e.*, when 2 mg/l. oxalate was added, the recovery was 50% and when 10 mg/l. was added, the recovery was 66%.

Literature Cited

1. BERNSTEIN, L., and KHAN, A. *Amer. Soc. Brew. Chem., Proc.* 1973, p. 20.
2. BURGER, M., and BECKER, K. *Amer. Soc. Brew. Chem., Proc.* 1949, p. 102.
3. GLENISTER, P. R., and ALAVI, Z. I. *Amer. Soc. Brew. Chem., Proc.* 1969, p. 175.
4. KOCH, G. H., and STRONG, F. M. *Anal. Chem.* 37: 1092 (1965).
5. MARINELLI, L., FEIL, M. F., and SCHAIT, A. *Amer. Soc. Brew. Chem., Proc.* 1968, p. 113.