

Gas Chromatography

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CONCLUSIONS

1. Proper execution of the collaborative work on the gas chromatographic analysis of solvent residues in can coatings was hampered by the lack of uniformity in the test cans obtained from the can plants, and an unusual degree of control will be needed to overcome the problem.

RECOMMENDATION

It may be desirable to test the gas chromatographic part of the method for its accuracy and reproducibility. The same subcommittee or a new subcommittee may be recharged with this assignment using glass bottles containing known concentrations of added solvents.

This Subcommittee was formed in 1973 to evaluate by collaborative testing a method for the analysis of solvent residues in can coatings. The method used was the one described by Colberg and Shapiro (3) with modifications based on the paper by Garza

and Bavisotto (4). This report concerns the attempt of this Subcommittee to achieve the above objective. Work carried out in past years pointed to some obvious advantages in one sampling technique over the other (1) (1974) and the results of more recent work (1) (1975) indicated that the sampling system using the Altek piercing device gave the most reproducible results. This Subcommittee's work in 1975-1976 indicated that nonuniformity of the test cans contributed to the inconclusive results.

In order to overcome the difficulties from nonuniform can liners, this year's work was to include a set of unlined cans to which standard mixtures of solvents were added and analyzed along with the production cans.

PROCEDURE

Two-piece cans from a regular production run, representing two slightly different curing temperatures, served as the Youden block pair. In addition, a set of unlined cans served as a second sample pair. Standard mixtures of solvents were to be added to the unlined cans.

Both sets of cans as obtained were analyzed in the laboratory of the Chairman of the Subcommittee in order to check the uniformity of the production cans and to determine if the unlined cans could be used for preparing standard samples.

TABLE I
Analysis of Replicate Samples of Cans Baked at
Low and Regular Temperature^a

Peak No.	Solvent	Temperature	1	2	3	4	5	6	Mean	Std. Dev.
1	MIBK	low	0.95	0.96	1.04	0.88	1.25	1.50	1.10	0.24
		reg.	0.77	0.70	1.33	0.90	0.78	1.74	1.04	0.41
2	Diacetone alcohol	low	0.52	0.57	0.58	0.56	0.61	0.85	0.62	0.12
		reg.	0.86	0.42	0.55	0.84	1.99	0.73	0.90	0.56
3	Toluene	low	11.68	11.68	12.07	10.16	9.95	11.64	11.20	0.90
		reg.	5.69	4.91	7.50	6.13	5.91	8.39	6.42	1.28
4	n-Butyl alcohol	low	531	456	467	403	419	449	454	44.62
		reg.	217	196	313	243	229	358	259	62.62

^aNumbers represent ratio of solvent peak area to that of internal standard (2,6-dimethyl-3-heptanone).

TABLE II
Analysis of Replicate Samples of Unlined Cans^a

Peak No.	Solvent	1	2	3	4	5	6	Mean	Std. Dev.
1	MIBK	0.17	0.19	0.20	0.18	0.16	0.18	0.18	0.01
2	Diacetone alcohol	0.56	0.63	0.53	0.48	0.54	0.65	0.58	0.07
3	Toluene	0.38	0.28	0.36	0.38	0.47	0.53	0.40	0.09
4	<i>n</i> -Butyl alcohol	11.22	9.15	8.45	9.08	13.01	11.17	10.35	1.74

^aNumbers represent ratio of solvent peak area to that of internal standard (2,6-dimethyl-3-heptanone).

RESULTS

Results of the examination of the production cans once again indicated that they were so lacking in uniformity that any further collaborative work was of questionable value. The unlined cans also showed variable low levels of the same organic solvents found in the production cans. This was probably derived from the atmosphere of the can-making plant. Typical results for lined and unlined cans are given in Tables I and II, respectively.

These results indicated that, unless special precautions are taken, a normal production cannot supply cans of such uniformity as is needed by a collaborative test of the nature attempted by this Subcommittee.

Literature Cited

1. AMERICAN SOCIETY OF BREWING CHEMISTS. Report of subcommittee on application of gas-liquid chromatography to analytical problems in brewing chemistry. *Proc.* 1974, p. 28; *Proc.* 33(3): 79 (1975).
2. AMERICAN SOCIETY OF BREWING CHEMISTS. Report of subcommittee on gas chromatography. *Proc.* 34(3): 98 (1976).
3. COLBERG, K. J., and SHAPIRO, R. *Amer. Soc. Brew. Chem., Proc.* 1969, p. 149.
4. GARZA, A. C., and BAVISOTTO, V. S. *Amer. Soc. Brew. Chem., Proc.* 1972, p. 10.