

3-48322

Z. G. DeCapita	Avon Lake Technical Center	DATE THIS LETTER
R. S. Morgan	Avon Lake Technical Center	January 1, 1974 ASH-1-74

Residual Vinyl Chloride in Liquor * DE T A
(Ref: IOM dated 9-18-73 to Z. G. DeCapita from R. S. Morgan)

Introduction:

Samples of 50/50 by volume ethanol/water solutions contained known amounts of vinyl chloride. In this series, levels of approximately 7 and 400 ppb were prepared and analyzed by gas chromatography using a flame ionization detector. Data at higher concentrations were available from previous studies (IOM dated 5/24/73 to Z. G. DeCapita from C. A. Brunot and IOM dated 8/31/73 to Z. G. DeCapita from R. S. Morgan).

Conclusions:

1. The lower limit of detection is less than 5 ppb.
2. Test results on a sample containing 50 ppb will fall between 30 and 80 ppb. (Fig. 1)
3. Practically all of the vinyl chloride is recovered by test technique being employed. (Fig. 2)
4. No detectable loss of vinyl chloride occurred from the samples during three months storage.

Discussion:

The laboratory work involves a single operator using the same gas chromatograph and column. The column was removed and the chromatograph used for other purposes between sets.

The conclusion that the lower limit of detection is less than 5 parts per billion is based on fact that vinyl chloride was detected on all 42 samples, one of which was known to contain only 4 ppb and four of which were known to contain only 5 ppb; and none was detected on the nine samples that were not spiked.

Data from other studies were available from which the reproducibility at approximately 2000, 5000, 10000, and 20000 ppb could be calculated. The upper and lower limits for 95% confidence are plotted in Figure 2. Interpolation of this data indicates that 95% of the test results on a sample containing 50 ppb vinyl chloride would fall between about 30 and 80 ppb. This, of course, applies only for this operator and on this particular equipment.

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Figure 2 plots the percent of the known vinyl chloride content that was recovered (i.e. detected) by the gas chromatograph. For the solutions containing over 200 ppb vinyl chloride there is little difficulty in recovering over 90% which is a criteria mentioned by the FDA for an acceptable test procedure. In fact, 98% recovery was averaged for these samples. For the solutions containing less than 10 ppb the variability in percent recovery is quite poor, but the average recovery is satisfactory. The high variability in recovery is probably caused by the weighing errors inherent in adding the vinyl chloride to the ethanol-water solution. The fact that test results on samples containing 50 ppb can be expected to range between 30 and 80 ppb means that multiple sampling and testing will be required when test results are near a specification limit of 50 ppb maximum.

The first set of samples were retained and retested after one month and after three months. Based on the peak areas and the calculated ppb vinyl chloride, it appears no significant change in the vinyl chloride content occurred during shelf storage. Hence, the same known sample for instrument calibration can be retained and reused for up to three months.



R. S. Morgan

ch

cc: R. L. Bowles
L. B. Crider
J. R. Goots
F. E. Krause
R. M. Kreager
A. F. Metzger
J. J. Nive
A. L. Schultz
E. G. Schwagerle
B. M. G. Zwicker
C.T.F. ✓
File/RSM
H. H. Marty
Attachments (3)

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95% of Subsequent Analyses on
Same Sample Will Fall Between
Solid Lines.

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K SOLUTIONS (5-8 ppb)

$\Delta = 46.4\%$

$\bar{x} = 113\%$ Recovered

'D' SOLUTIONS (240-420 ppb)

$\Delta = 4.0\%$

$\bar{x} = 98.4\%$ Recovered

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PPB VINYL (NORRIS)

SHEET NO.

"D" SAMPLES

"E" SAMPLES

	KNOWN	FOUND	DATE	KNOWN	FOUND	K.
SET 1 (9-20-73)	380	362	(9-18)	7	3.7	
	381	359		8	6.6	
	399	367		7	7.2	
	359	369		7	8.3	
	374	359		8	16.5	
SET 2 (9-25-73)	379	364	(9-25)	7	7.9	
	389	372		7	8.2	
	378	374		7	7.0	
SET 3 (10-9-73)	418	398	(10-9)	8	7.9	
	417	406		7	14.7	
SET 4 (10-24-73)	403	409	(10-24)	8	5.3	
	407	357		8	13.5	
SET 5 (10-25-73)	375	357	(10-25)	7	5.1	
	369	370		7	6.3	
SET 6 (10-27-73)	309	317	(10-27)	6	1.1	
	315	316				
SET 7 (10-30-73)	239	250	(10-29)	4	2.8	
	240	241				
SET 8 (12-12-73)	292	286	(12-12)	5	5.6	
	296	322		5	7.3	
SET 9 (1-7-74)	314	296	(1-7-74)	5	5.6	
	305	295		5	5.0	
SET 1 (10-1-73)	380	364	(10-1-73)	7	10.8	
	381	373		8	9.0	
	399	386		7	15.2	
	359	369		7	6.5	
	374	366		8	8.0	
SET 1 (1-8-74)	380	360	(1-8-74)	7	7.0	
	381	319		8	9.9	
	399	383		7	6.6	
	359	370		7	6.6	
	374	378		8	12.2	
ST. dev. in Differences of Los pro		.0156			.1552	

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