

INTER-OFFICE MEMO TENNECO CHEMICALS, INC.

TO P.A. Ketterer
FROM W.C. Champion
SUBJECT Analysis for VCM in Resin

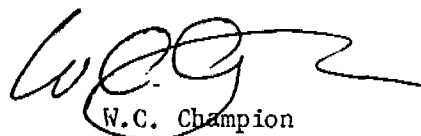
AT Flemington
AT Burlington

DATE November 21, 1974

COPY TO *P.L. Bogart
 H.B. Carr
 F.W. Kanzler
 R.A. Lojewski
 *Dr. M. Rosen
 /File

The attached B.F. Goodrich analytical method was obtained by Pete Bogart through the Vinyl Chloride Safety Association. Essentially, the method allows for determination of residual vinyl chloride in resin by analysis of the headspace of a vial of resin whose temperature has been raised to above the Tg to drive off the dissolved monomer.

If this method is as accurate and reproducible as claimed by Goodrich, considerable analysis time could be saved over our current dissolution method. We would request to you evaluate this method and comment upon it.


W.C. Champion

Attachment.

WCC/jst

*without attachment

→ G. D. SCHAFF

BFGoodrich

The B. F. Goodrich Company

RESEARCH AND DEVELOPMENT CENTER

9821 BRECKSVILLE ROAD • BRECKSVILLE, OHIO 44141 • PHONE: 216-526-4311

September 19, 1974

Dr. T. G. Fox
Mellon Institute
Pittsburgh, Pennsylvania 15213

Dear Dr. Fox:

Enclosed are three copies of the manuscript "Analysis for Vinyl Chloride in PVC Powders by Head-Space Gas Chromatography", by A. R. Berens, L. B. Crider, C. M. Tomanek, and J. M. Whitney. I hope you will consider this short paper for early publication in Polymer Letters Edition, Journal of Polymer Science.

We believe that this note warrants speedy publication in view of the current concern about the possible toxicity of vinyl chloride. The rapid, simple, and reliable analytical procedure we report should quickly be noted and applied by the many producers and fabricators of PVC around the world. Furthermore, the general method -- analysis of vapor in equilibrium with a polymer above Tg -- may well prove useful in other polymer/volatile-contaminant systems, which are of increasing concern throughout the polymer industry.

As I will be out of the country until November 4, would you please address your reply and any requests for clarification or revision to my co-author:

L. B. Crider
B. F. Goodrich Chemical Co.
Technical Center
Avon Lake, Ohio 44012

Thank you very much for your prompt consideration of this paper.

Sincerely yours,



Alan R. Berens
Research Fellow

bak
enclosures (3)

COLORITE 013271

Analysis for Vinyl Chloride in PVC Powders by
Head-Space Gas Chromatography

by: A. R. Berens, Corporate Research, The B. F. Goodrich Co.,
Brecksville, Ohio.

L. B. Crider and C. M. Tomanek, Technical Center, B. F. Goodrich
Chemical Co., Avon Lake, Ohio and

J. M. Whitney, General Chemical Plant, B. F. Goodrich Chemical Co.,
Avon Lake, Ohio.

Recent concern about the possible health hazard of exposure to vinyl chloride monomer, VCM (1) has focused attention on the measurement of residual monomer contents of commercial PVC products. We report here a simple, rapid analysis for VCM in PVC through gas chromatography of the vapor phase over PVC powder samples.

The basis of our method is the observation (2) that at a temperature above the glass-transition point of PVC, the solubility of VCM in PVC accurately follows Henry's Law behavior up to a VCM content of at least 4000 ppm. Writing Henry's Law as

$$S = KP_v \quad (1)$$

where S is the concentration of VCM dissolved in the PVC in ppm by weight VCM/PVC, and P_v is the VCM partial pressure in mm. Hg, we find the Henry's Law constant, K , is 6.52 ppm/mm at 90°C. The analytical procedure simply involves sealing a weighed PVC sample containing VCM in a

suitable container at room temperature, heating the container to 90°C to establish VCM equilibrium between PVC and vapor phases, then analyzing a sample of the "head-space" vapor to determine its VCM content. Henry's Law is then applied to calculate the original VCM content of the PVC as follows:

Let us say that the PVC sample of weight $\underline{m}$ grams originally contains $\underline{W_t}$ grams VCM. When equilibrated at 90°C in a closed container, the same amount of VCM is now distributed between PVC and vapor phases, or

$$W_t = W_v + W_p \quad (2)$$

where W_v and W_p are the weights of VCM in the vapor and polymer phases respectively. Henry's Law gives

$$\frac{W_p}{m} = K P_v \quad (3)$$

and, assuming ideal gas behavior,

$$W_v = \frac{M_v P_v V_g}{R T_2} \quad (4)$$

where M_v is the molecular weight of VCM, V_g is the volume of the vapor phase, (container volume less volume of PVC sample), R is the gas constant, and $T_2 = 363^\circ\text{K}$. Combining (2), (3) and (4) gives

$$\frac{W_t}{m} = P_v \left(K + \frac{M_v V_g}{m R T_2} \right) \quad (5)$$

While the PVC and vapor are at 90°C, a sample of vapor is taken by syringe and brought to room temperature, T_1 , and pressure, P_1 where its volume is $\underline{Z}$ ml., then injected into the gas chromatograph. From the VCM peak area, (or height), the weight, $\underline{Y}$, of VCM in the syringe sample is obtained by

applying the appropriate response factor, peak area (or height) per g VCM. The response factor is determined through calibration of the GC response on known gas mixtures. The syringe sample thus contains Y/M_v mols VCM and $P_1 Z/RT_1$ total mols gas. The mol fraction VCM, ϕ_v , in the vapor, both in the syringe at room temperature and in the vial at 90° is thus

$$\phi_v = \frac{Y/M_v}{P_1 Z/RT_1} = \frac{YRT_1}{M_v P_1 Z} \quad (6)$$

Since the gas sample was simply cooled in going from vial to syringe,

$P_1/T_1 = P_2/T_2$, so

$$\phi_v = \frac{YRT_2}{M_v P_2 Z} \quad (7)$$

The partial pressure of VCM at 90° is thus

$$P_v = \phi_v P_2 = \frac{YRT_2}{M_v Z} \quad (8)$$

Introducing (8) into (5) we have

$$\frac{W_t}{m} = \frac{YRT_2}{M_v Z} \left(K + \frac{M_v V_g}{mRT_2} \right) \quad (9)$$

This expression gives the VCM content of the PVC sample, W_t/m , directly from the GC analysis of the vapor phase.

The time required for equilibration of VCM between vapor and PVC phases can be estimated from recently reported diffusion data (3). From the solution of Fick's diffusion equation for spherical geometry (4), the time required for 99% of the original VCM dissolved in a PVC sample to diffuse out into a VCM-free environment is given by

$$t_{.99} = \frac{0.45 d^2}{D} \quad (10)$$

where d is the particle diameter in cm and D the diffusion coefficient in cm^2/sec . For VCM in PVC at 90°C , D is approximately $2 \times 10^{-10} \text{ cm}^2/\text{sec}$ (3), hence t_{99} will be less than 1 hour for PVC particles of less than 25 microns effective diameter. In our experience, a one-hour heating time at 90°C has proven satisfactory for the analysis of most commercial PVC resins in the as-polymerized powder form. Our analytical method is less satisfactory for molded, extruded, or otherwise fused PVC samples because of the very long equilibration times required.

In developing the head space method of analysis, we have used a Hewlett Packard 5710 gas chromatograph equipped with a flame ionization detector. The column used for the analysis is a 6 ft. x 1/8 inch stainless steel column, packed with 80/100 mesh Porapak QS. The column oven is controlled at 140°C , the detector at 250°C and the injection port at 150°C . Carrier gas (helium) flow is 30 cc per minute. Under these conditions, vinyl chloride elutes from the chromatograph in approximately 280 seconds.

A response factor for the chromatograph is determined by injecting known, standard mixtures of vinyl chloride and air.

Samples for analysis are prepared by weighing approximately two grams of PVC powder into a 12 cc vial and sealing the vial with a fold-over septum. The sample vials are then placed in an air oven or water bath controlled at 90°C and allowed to equilibrate.

After equilibrium is established an aliquot of the vapor space in the vial is removed with a gas-tight syringe. The volume of this vapor sample at room temperature and pressure is noted. The sample is injected into

the chromatograph for analysis. Determination of the residual vinyl chloride in the sample is made using equation (9) above.

To verify the accuracy of the head space analysis, a PVC resin was stirred of all residual vinyl chloride via vacuum-thermal techniques. This "clean" resin was then weighed into sample vials which were then septum-sealed. Known amounts of vinyl chloride gas were injected into the vials and allowed to equilibrate for one hour at 90°C. The vial head space was then analyzed as described. Table 1 compares the head-space analytical results with the calculated ppm vinyl chloride.

TABLE 1

Resin Weight	VCl Charged	Calculated	Head-Space Analysis
<u>(gms)</u>	<u>(cc)</u>	<u>ppm VCl</u>	<u>ppm VCl</u>
3.2	2.	1600	1586
3.2	1.	793	759
3.2	.5	397	379
3.2	.025	20	23.3

The head-space method has also been compared to a direct method of analysis in which residual vinyl chloride is determined without relying on established method of polymer/vapor equilibrium. In this method, 20-30 mg of PVC resin is weighed into a glass tube which fits into the injection port of the H-P 5710 chromatograph. The PVC powder is held in the heated section of the tube with a glass-wool plug. The injection-port septum is removed, the sample tube dropped into the injection port, and the septum replaced. The heat of the injection port (150°C) drives the volatiles out of

the polymer into the carrier gas stream. To produce plug flow through the column, its first 2 or 3 inches are chilled with dry ice before the sample is introduced. After sample insertion, the column oven is programmed at 16°C per minute to 200°C. Vinyl chloride elutes in approximately six minutes. The vinyl chloride peak area is a direct measure of the total VCl content of the PVC sample. While this method of analysis appears reliable, the small PVC sample size introduces a possible weighing error and the need to chill and program the column temperature makes the method somewhat less convenient than the head-space method. Table 2 compares data from the two methods.

TABLE 2

<u>Sample</u>	Head-Space Analysis	Direct Method of Analysis
	<u>ppm Residual VCl</u>	<u>ppm Residual VCl</u>
PVC Homopolymer "A"	30	30
	31	29
	31	20
	29	24
PVC Homopolymer "B"	46	47
	45	45
	43	48

To demonstrate the reproducibility of the head-space analysis, a PVC homopolymer resin was divided into 20 samples each sealed in a glass jar. Two of these samples were analyzed by the head-space method each day for ten days. Table 3 shows the results of this experiment.

TABLE 3

Head-Space Analysis	
<u>Sample Identification</u>	<u>ppm Residual VCl</u>
First Day	6.1
	5.9
Second Day	6.4
	7.2
Third Day	5.2
	5.4
Fourth Day	7.4
	7.5
Fifth Day	7.5
	7.4
Sixth Day	7.6
	7.8
Seventh Day	5.8
	6.6
Eighth Day	7.5
	8.4
Ninth Day	7.9
	8.4
Tenth Day	8.4
	7.5

Average: 71. ppm

Std. deviation: 1.0 ppm

In closing, we suggest that a similar analytical method might be generally applicable for the determination of volatile components in polymers. To establish the method for a new polymer/penetrant system would require, first, the determination of conditions where Henry's Law applies and of the Henry's Law constant, and second, an estimate of the time required to establish vapor-polymer equilibrium.

The authors gratefully acknowledge M. Mele's contribution of data in Table I and The B. F. Goodrich Company's permission to publish this note.

-7-

References

- 1) Chem. Engr. News, Jan. 28, 1974, p.6.
- 2) A. R. Berens, Polymer Preprints 15 (2) 197 (1974).
- 3) A. R. Berens, Polymer Preprints 15 (2) 203 (1974).
- 4) J. Crank, The Mathematics of Diffusion, Oxford Univ. Press, London, 1956.

→ G. V. SCHMIDT

B. F. GOODRICH CHEMICAL COMPANY

Standard Test Procedure No. 999-T

TITLE: Residual Vinyl Chloride Monomer Content of Polyvinyl Chloride Resins

TYPE OF ANALYSIS: Vapor Space Equilibrium, Gas Chromatographic

PRODUCT: Polyvinyl Chloride Resin, Finished Product

I. SCOPE

This procedure is suitable for determining the residual vinyl chloride monomer content of polyvinyl chloride (PVC) resins. The method cannot be used for polymer in other forms such as compounds or final fused products.

II. PRINCIPLE

The basis for this method relates to the vapor equilibrium which is established between residual vinyl chloride monomer (RVCM), PVC resin and air in a closed system. It has been demonstrated that the RVCM in a PVC resin will equilibrate in a closed vessel quite rapidly, provided the temperature of the PVC resin is maintained above the glass transition temperature of that specific resin.

III. INTERFERENCES

- 1) Normally this vapor will contain only air, vinyl chloride monomer and traces of water. Any other volatile material present in the closed vessel could change this equilibrium relationship. Impurities in the low ppm range will generally have only a very small influence on this equilibrium relationship.
- 2) Any material which elutes from the chromatographic column at approximately the same time as vinyl chloride will cause high RVCM results.

IV. PRECISION AND REPRODUCIBILITY

Initial data obtained with this method, covering a range of 0.0025 to 0.2000 wt. percent, indicates that one standard deviation (determined from duplicate results) is approximately 2.1%. Absolute accuracy has been checked by comparing results to another, totally independent method. In this case agreement between the two methods is $\pm 5\%$ in the range 0.0025 to 0.2000 wt. %. These results were obtained by two operators in one laboratory.

V. SAFETY

- 1) Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting and purging with vinyl chloride - air mixtures must be held to an absolute minimum. When venting and/or purging is required the vapor must be routed to outside air. Vinyl chloride, even at low ppm levels, must never be vented inside the laboratory. It can be vented into a properly functioning fume hood.
- 2) Be careful not to come into contact with heated parts of the chromatograph, such as the injection port, detector, heated column, etc. Handle all electrical connections with care.

VI. APPARATUS

- 1) Gas Chromatograph, Hewlett Packard Model 5711A, dual flame ionization detector, equipped with following options:
 - (a) 005 Dual flow controller
 - (b) 006 Substitute linear oven programmer (5702A) for isothermal oven controller
 - (c) 011 Substitute auxiliary heated zone controller (5708A)
 - (d) 017 Substitute universal heated injection port with disposable glass liners (18747)
 - (e) 024 Rotometer kit for carrier gas
- 2) Integrator -- Hewlett Packard Model 3380A with built-in recorder
Alternate Integrator -- AutoLab System 4
- 3) Chromatographic Column -- An 8 ft. x 1/8 in. stainless steel column packed with 80/100 Poropak QS and conditioned at 200° C.
- 4) Oven, forced draft capable of maintaining a constant temperature of 90° ± 1.0° C. (Fisher Cat. No. 13-244-176 Isotemp Oven)
- 5) Gas Sampling Bottles, 250 ml capacity equipped with glass stopcocks on both ends plus a septum fitting (Fisher Cat. No. 11-134-190)
- 6) Serum Bottles, 10 cc volume with rubber septums (Fisher Cat. No. 3-210B serum bottles and Cat. No. 3-215 rubber stoppers)
- 7) Analytical Balance, capable of weighing to ± .0001 gram
- 8) Hydrogen Cylinder with regulator
- 9) Flowmeter, 0 to 2 liters per minute

VI. APPARATUS (continued)

- 10) Helium Cylinder with regulator
- 11) Clean air supply at 25 psig
- 12) Bubble Flow Indicator

VII. REAGENTS

Calibration gases, vinyl chloride in air mixtures

- 1) Low level between 15 and 25 parts per million
- 2) A high level between 800 and 1200 parts per million

These can be obtained from:

Precision Gas Products, Inc.
P. O. Box 538
Linden, New Jersey 07036

VIII. PREPARATION OF GAS CHROMATOGRAPH

- 1) Install the 8 ft. x 1/8 in. Poropak QS column and condition at 200° C.
- 2) Adjust flow rates for the H. P. 5711A according to the operating manual (30 cc's per minute helium carrier gas, 30 cc's per minute hydrogen and 130 cc's per minute air.)
- 3) Set conditions for analysis as follows:

Injection port - 150° C.
Detector - 250° C.
Oven - 160° C.

- 4) After these conditions have been reached and the chromatograph has stabilized it is ready for calibration.

IX. CALIBRATION

- 1) Fill the clean glass 250 ml gas sample bottle with the low level (15 to 25 ppm) vinyl chloride air standard. The procedure for doing this is detailed in Attachment 1. Always use the same bottle for this low level standard.
- 2) With the 1 cc gas syringe draw out of the calibration bottle, through the septum, in excess of 1 cc of the gas sample. After the plunger on the syringe is pulled all the way, wait five seconds before removing the syringe needle from the bottle septum.
- 3) After the five second delay remove the needle from the septum and move plunger to read exactly 1 cc on the syringe.

IX. CALIBRATION (continued)

- 4) Immediately inject the 1 cc sample into the chromatograph injection port (as rapidly as possible).
- 5) Remove the needle from the injection port and start the chromatograph recorder and integrator.
- 6) In approximately three minutes the vinyl chloride will elute.
- 7) Wait one minute after the VCl elutes and starting at Step 2 above repeat Steps 2 through 6. Do this four times.
- 8) Fill in the data obtained in the four runs above on the calibration sheet (Attachment 2).
- 9) If the peak areas from these four runs do not deviate more than 5% from the average of the four runs, this step is complete. If there is a deviation greater than 5% from the average, repeat Steps 1 through 8. See Note 1.
- 10) After the above calibration is completed, recharge the second glass calibration cylinder with the high level (800 to 1200 ppm) vinyl chloride air standard. Starting at Step 2 above repeat this procedure through Step 9 using the high level standard.
- 11) This calibration is complete if the requirements of Step 9 are met. If Step 9 is not met, refer to Note 1.
- 12) For the third calibration point use the high level standard, but in this case use only a 0.5 cc sample.
- 13) Draw out an excess of 0.5 cc with the gas syringe.
- 14) Wait five seconds after the plunger is pulled back before removing the syringe needle.
- 15) After the five second delay remove the syringe and adjust the plunger to read exactly 0.5 cc.
- 16) Inject this sample into the chromatograph and record the peak area on the calibration sheet.
- 17) Repeat Steps 13 through 16 four times.
- 18) If the peak areas from these four runs do not deviate more than 5% from the average of these four runs, this calibration is complete. If there is a deviation greater than 5% from the average, repeat Steps 13 through 16. See Note 1.

IX. CALIBRATION (continued)

- 19) The data generated in the above calibration is used to calculate the response factor for the calculation of residual vinyl chloride in the samples analyzed. The response factor calculations are detailed on the calibration sheet.

Note 1: If the deviation is still greater than 5%, shut down and locate the source of the problem.

X. SAMPLE ANALYSIS (Samples must be run at same range and attenuation used for calibration standards. If range or attenuation is changed for the sample, then new standards must be run at this range and attenuation.)

- 1) All samples should be kept in tightly sealed glass jars. Samples must be run promptly (within 30 minutes) from the time they are received in the laboratory. Within 30 minutes the sample should be weighed into the serum bottle and placed in the oven. The exact total volume of the serum bottles must be measured. See Attachment 5.
- 2) Prepare duplicates for each sample. Take one serum bottle plus rubber septum, mark with identification and obtain a tare weight to four decimal places. Record tare weight on analysis work sheet. See Attachment 3.
- 3) Remove serum bottle from balance, remove septum and pour in the proper amount of resin to be analyzed. Replace rubber septum. Be certain that rubber septum is fully seated and sealed.

<u>Approximate Bottle Volume</u>	<u>Add</u>
12 cc	2 grams $\pm$.5

A simple technique can be devised based on a volumetric resin addition. Small cups, having the proper volume, can be used to rapidly obtain the resin sample. This can then be added to the serum bottle with a tiny funnel.

- 4) Replace serum bottle on balance and record the weight to four decimal places.
- 5) Remove bottle from balance.
- 6) Place bottle in oven (at $90^{\circ} \pm 1.0^{\circ}$ C.) and allow it to remain 15 -0 +1 minutes.
- 7) At the end of the heating cycle remove the bottle from the oven and immediately insert the needle from the 1 cc gas syringe into the bottle through the septum. (Do not allow the needle to contact the resin sample.)

X. SAMPLE ANALYSIS (continued)

- 8) Sample Size - Use a 1 cc sample volume when the RVCM content is expected to be less than 100 ppm. Use a 0.5 or 0.3 cc sample for expected higher concentrations.
- 9) Draw out the proper volume sample (plus slight excess) from the bottle. Wait 5 seconds after pulling the plunger back before removing the needle from the sample bottle.
- 10) After the 5 second delay remove the syringe needle from the serum bottle and adjust plunger to read the proper volume.
- 11) Inject the sample into the chromatograph, start integrator and recorder.
- 12) The vinyl chloride peak will elute in approximately 3 minutes.
- 13) Wait one minute after the vinyl chloride peak elutes and repeat Steps 7 through 11 using a duplicate sample.
- 14) Record sample peak area, gas sample size, attenuation, sample identification, etc. on work sheet (Attachment 3).
- 15) One minute after the vinyl chloride elutes you are ready to run another sample.

XI. CALCULATION OF RVCM IN SAMPLE

The following information is needed to calculate the residual vinyl chloride content of the resin.

- 1) Response factor for vinyl chloride = R (counts/nanogram VCl)
(as determined in the calibration step)
- 2) Weight of polyvinyl chloride resin in vial = W (grams)
- 3) Volume of sample injected into chromatograph = V (cc)
- 4) Area of the chromatograph peak for the vinyl chloride = A (integrator counts)
- 5) Volume of sample bottle (serum vial) = B (cc)
- 6) Atmospheric Pressure = P (mm Hg)

$$\text{ppm RVCM} = \frac{\left[\frac{A \times 10^{-1}}{A \times 10^{-9} + (1.16 \text{ RV} \times 10^{-3})} \right]}{100 - \left[\frac{A \times 10^{-7}}{A \times 10^{-9} + (1.16 \text{ RV} \times 10^{-3})} \right]} \times \left[\frac{(29)(363)P}{(29.5)(62.5)} \right] \times \left[(6.5 \times 10^{-6}) + \frac{62.5 \times (B - 1.4)}{(62400)(363)(W)} \right]$$

This calculation can very easily be set up on a Monroe 1860 Programmable Calculator. It should not be routinely performed on a hand calculator because of a high probability for errors. The Monroe 1860 program for this method is attached. See Attachment 4.

ATTACHMENT 1

Method for Preparation of Standard VCM/Air Mixtures

- 1) Clean the 250 ml gas sampling bottles with acetone and blow thoroughly dry with clean air or nitrogen.
- 2) Apply low vapor pressure stopcock grease to both stopcocks after washing and drying.
- 3) Attach a septum to the septum stem.
- 4) Using the proper standard VCM/air cylinder equipped with regulator attach the sample bottle using a short piece of tubing. Attach the other end of the bottle to the rotometer.
- 5) Open both stopcocks on the gas sampling bottle. Open valve on gas cylinder. Very slowly and carefully open the regulator on the cylinder to provide a flow rate of approximately 1.5 liter per minute.
- 6) Purge for two minutes. Be certain that the VCM/air mixture is properly vented as stated in Section V.
- 7) At the end of the purge time turn off the valve at the cylinder and then both stopcocks to off, closing the stopcock closest to the cylinder first.
- 8) Gas sampling bottle is now ready to use.

ATTACHMENT 2Calibration Data Sheet and Calculations

Run No.	Sample Identification	Injected Sample Size, cc	Peak Area	Avg. Peak Area	Deviation from Avg.	Counts per Nanogram VCl	Avg. Counts per Nanogram VCl
1	19 ppm Std	1.0	4600		.2		
2	"		4565		1.0		
3	"		4690		.4		
4	"		4585	4610	.5	95.7	
5	805 ppm Std	0.5	95370		.8		
6	"		96485		.4		
7	"		95732		.4		
8	"		96901	96122	.8	94.2	
9	805 ppm Std	1.0	193946		1.4		
10	"		190241		.5		
11	"		189789		1.4		
12	"		190916	191,223	.2	93.7	94.53

Using a 1.0 cc sample, each ppm VCl in the standard contains 2.5352 nanograms VCM.

Runs 1 through 4 each contain $(19)(2.5352) = 48.17$ nanograms VCM

Runs 5 " .8 " " $(805)(2.5352)(.5) = 1020.4$ nanograms VCM

Runs 9 " 12 " " $(805)(2.5352) = 2040.8$ nanograms VCM

Then Average Peak Area $\div$ Nanograms VCM = Counts per Nanogram VCM

The Response Factor = Avg. Counts per Nanogram VCM = 94.53

COLORITE 013289

BFG Standard Test Procedure No. 999-T

ATTACHMENT 3

Analysis Work Sheet and Results

Date _____

GC Run No.	Sample Identification	Sample Weight (grams)	Vapor Sample Volume (cc)	GC Peak Area	Atm. Pressure mm	Response Factor	Volume of Serum Bottle	PPM RVCM in Resin
		W	V	A	P	R	B	
1	"A"	2.0012	1	43,210	753	94.53	12.0	6.4
2	"B"	1.9873	.3	769,343	↓	↓	↓	213
3	"C"	2.1249	.5	143,356				22.6
4	"D"	2.0141	.5	19,423				3.2
5	"E"	1.9981	.5	117,324				19.4
6	"F"	2.0476	.5	4,422				0.7

COLORITE 013290

ATTACHMENT 4

Program for Calculating % RVCM in Resin - Monroe 1860

Operating Procedure:

1) Br () (); 0; 0

Program No. 133 prints

2) Enter

A) Area Counts

B) Response Factor, counts/nanogram VCM

C) Syringe Volume, cc

D) Atm. Pressure, mm

E) Sample Weight, grams

F) Volume of Serum Bottle, septum in place, cc

3) Prints answer in ppm RVCM in resin

```
133.0000 _____ PROGRAM NO.
.....
144,735.0000 _____ AREA COUNTS
  94.5300 _____ RESPONSE FACTOR
   0.3000 _____ SYRINGE VOLUME
 753.0000 _____ ATM. PRESSURE
   2.0012 _____ SAMPLE WEIGHT
  12.0000 _____ SERUM BOTTLE VOLUME

  39.8641      A
.....
.....
```

133.0000
.....

0000	116	$\bar{0}$
1	000	0
2	001	1
3	003	3
4	003	3
5	060	
6	176	
7	056	
8	110	↓
9	001	1
0010	060	
1	056	
2	110	↓
3	002	2
4	060	
5	056	
6	110	↓
7	003	3
8	060	
9	056	
0020	110	↓
1	004	4
2	060	
3	056	
4	110	↓
5	005	5
6	060	
7	056	
8	110	↓
9	010	0
0030	060	
1	065	
2	111	↑
3	001	1
4	023	X
5	026	(
6	001	1
7	000	0
8	025	a ^x
9	001	1

0040	013	-
1	027	)
2	020	=
3	110	↓
4	006	6
5	111	↑
6	001	1
7	023	X
8	026	(
9	001	1
0050	000	0
1	025	a ^x
2	011	9
3	013	-
4	027	)
5	020	=
6	021	+
7	026	(
8	111	↑
9	002	2
0060	023	X
1	111	↑
2	003	3
3	023	X
4	012	
5	000	0
6	000	0
7	001	1
8	001	1
9	006	6
0070	027	)
1	020	=
2	110	↓
3	007	7
4	111	↑
5	006	6
6	024	÷
7	111	↑
8	007	7
9	020	=
0080	110	↓
1	006	6
2	111	↑
3	001	1
4	023	X
5	026	(
6	001	1
7	000	0
8	025	a ^x
9	007	7

0090	013	-	0140	005	5
1	027	)	1	023	X
2	020	=	2	026	(
3	024	÷	3	111	†
4	111	†	4	010	8
5	007	7	5	022	-
6	020	=	6	026	(
7	013	-	7	111	†
8	021	+	8	005	5
9	001	/	9	024	÷
0100	000	0	0150	001	/
1	000	0	1	012	
2	020	=	2	004	4
3	110	↓	3	027	)
4	007	7	4	027	)
5	111	†	5	020	=
6	006	6	6	024	÷
7	024	÷	7	026	(
8	111	†	8	111	†
9	007	7	9	005	5
0110	020	=	0160	023	X
1	110	↓	1	006	6
2	001	/	2	002	2
3	003	3	3	004	4
4	006	6	4	000	0
5	003	3	5	000	0
6	023	X	6	023	X
7	111	†	7	003	3
8	004	4	8	006	6
9	023	X	9	003	3
0120	002	2	0170	027	)
1	011	9	1	020	=
2	020	=	2	021	+
3	024	÷	3	026	(
4	026	(	4	006	6
5	002	2	5	012	
6	011	9	6	005	5
7	005	5	7	023	X
8	023	X	8	012	
9	006	6	9	000	0
0130	002	2	0180	000	0
1	012		1	000	0
2	005	5	2	000	0
3	027	)	3	000	0
4	020	=	4	001	/
5	110	↓	5	027	)
6	002	2	6	020	=
7	006	6	7	110	†
8	002	2	8	003	3
9	012		9	111	†

0190	001	/
1	023	X
2	111	↑
3	002	2
4	023	X
5	111	↑
6	003	J
7	020	=
8	061	A
9	176	
0200	176	
1	065	
2	065	
3	065	
4	127	Br
5	000	o
6	000	o
7	000	o
8	000	o
9	377	

ATTACHMENT 5

Establishing True Volume of Serum Bottle

- 1) Clean serum bottle (water and detergent) and rinse with acetone. Air dry thoroughly.
- 2) Allow to condition for at least 15 minutes at room temperature.
- 3) Weigh serum bottle plus septum to 0.0001 gram on an analytical balance.
- 4) Using distilled water which has been conditioned to within $\pm 2^{\circ}$ F. of room temperature fill the bottle to the very top.
- 5) Carefully insert and seal the septum. This will tend to force a small amount of water out of the bottle.
- 6) Dry the outside of the bottle and weigh again on the analytical balance.
- 7) Knowing the temperature of the water, determine, using a table of density of water at various temperatures, the volume of the bottle.
- 8) Check three bottles and average the results. Individual results should not differ more than ± 0.3 cc from the average. (Bottles normally have a volume of approximately 12.0 cc.)