

Memorandum

AIR
PRODUCTS 

To: P. J. Usinowicz Dept.: Chem. Mfg. Env./Amin. V
From: J. H. Phillips Dept./Ext: CRSD-Analytical/5673
Date: 3 September 1987
Subject: Modification of EPA Method 107 for Tergitol E-35 Column

cc: M. J. Bonin (Pensacola)
~~P. J. Clark~~
~~R. L. Duggan (Pensacola)~~
~~J. H. Frost~~
~~D. D. Helms (Calvert City)~~

Due to a methanol interference with the VCM analysis at Pensacola, APCI wishes to obtain approval to use the the Tergitol E-35 chromatographic column in place of the Porapak Q column as specified in Method 107. The Environmental Protection Agency Method 107 has a provision for modification pending Regional Administrator approval. Section 3 of the EPA Method 107 reads as follows:

" The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis requirements, subject to the approval of the Administrator. Approval is automatic provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator."

On 31 August 1987, I discussed the requirements for approval of modified EPA Methods with David McNeil of the Atlanta regional office. Dr. McNeil is responsible for reviewing and approving method modifications for Region IV. Approval is granted on a region by region basis and therefore, if the modification is to be used outside of Region IV we must reapply to the appropriate region. Dr. McNeil has clarified what is meant by "automatic approval" and I have outlined below what is required for Region IV administrator approval.

I. Supplemental Analytical Technique for Confirmation

- A. It must be confirmed that an interference (MeOH) with the analyte (VCM) exists under the conditions set forth in the method (EPA #107).

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- B. It must be demonstrated that the modified method has no interferences with the analyte (VCM) by confirmation with a second analytical technique or chromatographic column.

Action Item: A sample containing the analyte (VCM) and the interference (MeOH) should be analyzed by headspace GC/MS using both the Porapak Q and Tergitol E-35 columns. This will confirm the presence of an interference and the separation of that and other interferences from the analyte.

II. Confirmation of Adequate Resolution

- A. Not more than 10% overlap of any interference with the VCM is allowed.
- B. 40 CFR Ch. 1 (7-1-85) Edition) Part 61, Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution" should be used.

Action Item: Headspace GC/FID analysis should be performed on a sample containing the methanol interference. The Tergitol E-35 column should be used and Appendix C, Procedure 1 should be followed.

III. Precision and Accuracy of the Analysis of VCM must not be impaired.

- A. In an intralaboratory comparison between several laboratories analysis of a PVC resin yielded relative standard deviations of 2.63% for a 2.09 ppm VCM spike, 4.15% for a 1.66 ppm VCM spike and 5.29% for a 62.66 ppm spike.

Action Item: Using headspace GC/FID with the Tergitol E-35 column, analyze a PVC resin spiked with between 5-10 ppm VCM. Repeat this analysis three or four more times and calculate the percent relative standard deviation of the analysis. Quantitate the VCM in accordance with Method 107 and report the mean percent recovery.

IV. Submit All Data to the Region IV Administrator

- A. Both the Florida DER and the Kentucky DEP have been granted regulator authority by the EPA. Therefore, both states should be informed of the method modification.

Action Item: Send a copy of all data, including chromatograms, along with a request for method modification to Brian Beals, EPA region IV. Send letters to Mr. Smallwood of the Florida DER and Roger McCann of the Kentucky DEP informing them that we have made a request to EPA Region IV for a modification of Method 107.

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Since some of the above action items may have been completed, it is not necessary to repeat them as long as the work is well documented. IF analytical assistance is needed in completing any of the action items we do have headspace GC/FID/MS capabilities within the CRSD-A Separation Group. Complete addresses for the Federal and State officials are listed below:

Mr. Brian Beals
APTMD Region IV
345 Courtland Street, NE
Atlanta, GA 30365

Mr. Smallwood
Florida DER
Bureau of Air Quality Management
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, FL 32301

Roger McCann
Division of Air Quality
Department for Environmental Protection
Fort Boone Plaza
18 Weilly Road
Frankfort, KY 40601


J. H. Phillips

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