MONSANTO CHEMICAL COMPANY
W. G. KRUMMRICH PLANT LABORATORY

STANDARD ANALYTICAL METHOD

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ATTACHMENTS:		

PRODUCT: AROCLOR 1242

TEST: Differential Infrared Scan

THIS FORM ☐ SUPERSEDES METHOD NEW ISSUED ON _____
☐ REVISES INSTRUCTIONS

SCOPE

Differential infrared (IR) spectroscopy employing a designated reference standard is used as an index of quality for electrical grade Aroclor 1242 intended for General Electric Company.

EQUIPMENT

1. Perkin-Elmer Model 21 Infrared Spectrophotometer
2. Variable Space Cell equipped with NaCl or KCl windows
3. Sealed liquid IR cell, ca 0.1 mm path length, with NaCl windows

REFERENCE STANDARD

Anniston Aroclor 1242 Sample No. AI-242 or a new one designated by customer.

INSTRUMENT CONDITIONS

Resolution	4	
Response	1	
Gain	ca 5-3/4	(or sufficient to duplicate noise level shown on attached spectra)
Suppression	1	
Chart Speed	2	

SAFETY

Aroclor 1242 is not particularly toxic, however, prolonged contact with the skin should be avoided. Wash or wipe off any that contaminates the body.

PRECAUTIONS

The salt windows of the variable space and sealed cells are highly polished salt crystals which must be handled and cleaned with extreme care. When exposed to an atmosphere of 50% humidity or higher, the windows will become foggy thus decreasing their transmissions. Actual contact with water or solutions containing over a few percent of water will ruin the windows for further use. Thus, the following precautions must be taken:

1. Do not touch windows with fingers or any object that might scratch the surface.

2. Do not handle cells nor work in the vicinity of sinks and water.

3. Clean cells with carbon tetrachloride, chloroform or other volatile but anhydrous solvents, but never with acetone. Dry with light vacuum and air of low humidity. Due to its construction and relatively large interior volume, variable space cells are best cleaned as follows:

   a. Enlarge cell depth until liquid level is visible in window when holding vertical.

   b. Invert and drain.

   c. Add carbon tetrachloride until cell is about half filled, and agitate gently.

   d. Invert cell and drain; repeat 3 times.

   e. Dry by vacuum for at least 15 minutes.

4. Remove any oil on outside of cells, or on window, with a soft tissue saturated with CCl_4.

5. Store cells in a dessicator or air tight compartment containing a solid dessicant.

PROCEDURE

1. Set wave-length to 2.000 microns as indicated on the counter.

2. Fill an approximately 0.1 mm sealed liquid cell with the designated standard Aroclor 1242 by means of a medicine dropper and plug lower opening.
   CAUTION: Inspect cell carefully for air bubbles after plugging.

3. Adjust variable space cell to a depth of about 0.15 mm and fill with the same standard in a similar manner until the visible portion of the windows is full.

4. Adjust the depth of the variable cell to the approximate depth of the 0.1 mm sealed cell.

PROCEDURE (Cont'd.)

5. Place in reference beam with shutter open.

6. Close shutter in sample beam and with the pen not engaged on the recorder paper, adjust the pen to zero transmission by means of the cable release spring.

7. Open shutter and by means of the 100% adjust knob, adjust the disengaged pen to about 0.03 absorbance.

8. Rotate recorder drum manually until pen coincides with the 2.000 micron position of the recorder paper. Engage drum to the drive mechanism by tightening the clutch knob. Disengage pen from recorder paper.

9. Start scanning at slow speed and observe movement of pen at around 3000 cm^{-1}. A positive deflection of the pen indicates the depth of the variable space cell is smaller than that of the sample cell; gradually enlarge the depth of the variable cell until the pen returns to the base-line at around 3000 cm^{-1}. A negative deflection indicates the reverse, so the cell depth would have to be made smaller.

10. Continue the scan to the region of 1420-1170 cm^{-1} and again make adjustments as in (9) above, if significant deflections occur. The purpose of these adjustments is to match the depths of both cells for differential scanning.

11. Set the Scanning Control Knob to Reverse and return to 2.000 microns without disengaging the drum.

12. Engage pen to paper at 2.000 microns and start scanning at low speed, again observing pen movement at ca 3000 cm^{-1}. If any significant deflection occurs (a visible peak), repeat steps (8) or (9) above, otherwise, allow scan to proceed to completion.

13. An acceptable scan is one that shows no sharp peaks, or, others identified with Aroclor 1242. The broad fluctuations noted in the sample spectrum are probably due to transmission differences of the windows themselves. (Chart #1, Spectrum A).

14. Disengage the pen from the recorder, and return to 2.000 microns without disconnecting drum from the drive mechanism.

15. Remove sealed cell from sample beam, clean thoroughly by drawing CCl_4 and air through the cell by vacuum. Do not change the setting of the variable space cell.

16. Re-fill sealed cell with sample to be evaluated, plug bottom drain and again make sure no air bubbles are present between the cell windows.

PROCEDURE (Cont'd.)

17. Adjust pen upward to about 0.05 absorbance by means of the 100% Adjust Knob, set scanning control to "Forward" and scan at slow speed to 15 microns.

18. If more than one sample are to be scanned at the same time, repeat from Steps (14) to (17) above with a new chart, making sure the pen coincides with the 2.000 micron mark of the chart.

19. Disengage pen from recorder and return to 2.000 microns on the wave-length counter. Leave instrument at this setting, return "Gain Knob" to original position but do not shut down.

20. Remove variable space cell and record the cell depth setting for future use. This will help the next time cells have to be matched.

21. Clean cells as instructed under "Precautions" above.

INTERPRETATION

Compare spectrum to that of acceptable W.G.K. material shown in Charts # 1B and 2. The critical regions for evaluation are at around 3000 cm^{-1} and from 1420-1100 cm^{-1}.

Peak	Evaluation
3000 cm^{-1}	A negative peak is indicative of over-chlorination and high gravity. A positive peak indicates under-chlorination and, thus, lower gravity.
1400 cm^{-1} 1335 cm^{-1}	A significant increase in absorption above that of the acceptable spectra should be considered "Suspect". Reject any sample that is similar to the "Unacceptable Spectrum" Chart #3). Spectra similar to those of the "Acceptable Samples", or if absorption is less, are "Acceptable".
Others	Hold samples showing any unusually large peaks in other regions and consult with AR&D. Strong absorption below 800 cm^{-1} may be due to CCl_4 cleanser incompletely removed.

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DISCUSSION

Since salt windows employed in IR cells are subject to gradual erosion during use, the effective path length or depth may change slowly. In addition, it is difficult to construct two sealed cells of the same depth. For these reasons it is impractical to employ so-called "matched" IR cells for differential analysis. One sealed cell should be set aside exclusively for this test and cared for. Cleaning and drying the cells is an important part of this method and must not be underestimated.

Once a variable space cell has been cleaned it is mandatory to start from Step (1) under "Procedure" for additional analyses. The accuracy and reproducibility of the settings on the variable space cell are not guaranteed.

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