



Aroclor methods

Dr. R. L. Jenkins, Anniston

June 4, 1941

REF: YOUR LETTER, MAY 30, 1941

cc: Mr. Gerber, Anniston

I am sending you, herewith, copies of our method for Moisture in Pyranols and Aroclors, and our General Method for Traces of Moisture. The first gives specific directions for the determination of moisture in the Pyranols and Aroclors, whereas the latter gives detailed instructions as to the preparation and standardization of the Karl Fischer Reagent.

We do not take any specific precautions in the sampling, nor for removing adsorbed moisture from the titration flasks. The flasks are dried in the usual type of drying oven and the solvent is titrated to the endpoint before the sample is added. This should preclude contamination of the sample with adsorbed moisture.

over

As to the cloud point method, the only information I have is that which you gave in your report of conference held at Pittsfield on August 14, 1939. In this you state that Clark used the ASTM method for the cloudpoint. You also gave a table showing the relationship between the ppm of water and the cloud point. I am showing a copy of that table below:

<u>H₂O</u>	<u>Cloud Point</u>
5 ppm.	-40°C
18 ppm.	-18°C
29 ppm.	- 7°C
50 ppm.	+ 3°C
68 ppm.	+ 8°C
100 ppm.	+20°C

In a subsequent letter to you (Oct. 10, 1939), Mr. Clark gave a correlation of the ppm. water and the dielectric strength. I am likewise showing a copy of that.

Water Content
(ppm. by weight)

Dielectric Strength (25°C)

10	42
25	34
40	26
60	15

DSW 330629

STLCOPCB4077273

The thing I particularly wanted to discuss with you while you were in St. Louis was the setting up of specifications for G. E. Pyranols, particularly No. 1476, which is now being made at Anniston. As you know, Mr. Clark was also in St. Louis about that time and I contacted him with regard to all of the Pyranols indicating to him some changes which I thought should be made. He noted these and was taking them back to Pittsfield for checking with his men up there. I am sending you, herewith, a copy of the specifications which I think Monsanto should set up for Aroclor 1254 (G.E. 1476). Should you have any comments they would be appreciated. If not, please advise me as I would like to send these along to Mr. Watt so that we can have a formal Plant B specification set up on this material along with the other Pyranols and Aroclors.

RR

M. J. Wiegand

P.S. Will you please check the figure for total chlorine content. Our old specifications indicated it to be 52.5 to 53.5, while in a memo to Dr. Kyrides, dated 5/12/38, you state that the specifications on chlorine content of G.E. 1476 is 54.5 to 55.5.

DSW 330630

STLCOPCB4077274

Materials: Aroclors and pyranols

Test: H₂O

Moisture in Aroclors and Pyranols is determined with Karl Fischer reagent (Method No. 10,100-40, (G.M. 7)). Because of the varying solubility of the products tested, different proportions of a mixed solvent are required. The analysis itself, however, is the same for all Aroclors and Pyranols.

The analysis is made as follows:

- 1) Add the solvents as listed below to a 500 ml. Erlenmeyer flask.

Material	Anhyd. Benzene	Anhyd. Methanol
Pyranol 1478 1476	0 ml. 60	50 ml. 50
Pyranol 1488	50 ml.	50 ml.
Pyranol 1495	80 ml.	50 ml.
All Aroclors	80 ml.	50 ml.

- 2) Titrate the solvent to the characteristic brown endpoint with Fischer reagent.
- 3) Refill the buret to the zero mark.
- 4) Weigh a 100.0 g. sample by difference into the solvent. Mix well.
- 5) Titrate the solution to the same endpoint as before.

Calculations: $\% \text{ H}_2\text{O} = \frac{\text{ml. Fischer reagent} \times \text{H}_2\text{O factor} \times 100}{\text{Sample Weight}}$

The method is accurate to $\pm 0.0005\%$, - 5 ppm

Report the results to the nearest 0.0005%. 5 ppm

Materials: General Method Method No. 10,100-40

Test: Water, Traces

Traces of water may be determined by means of a titration with Karl Fischer Reagent.

The reagent is prepared as follows: Place 867 ml. of dry methanol and 269 ml. of dry 2° pyridine in a dry 1000 ml. Florence flask. Add 84.7 g. of iodine and shake to dissolve. Stopper and cool to 0°-2°C in a slurry of ice and water. When

0.0033/cc 1.2220
100

10cc = 33.4 ppm
1.1cc = 3 ppm

DSW 330631

cooled to proper temperature, add 64 g. of SO_2 at such rate as to require about 90 minutes for complete addition. Keep cold during the procedure, and shake frequently. The SO_2 is best dispensed from a graduated cylinder, from which the lip has been removed, and is provided with a one hole rubber stopper through which a bent glass tube is passed. This bent tube should be connected to another tube leading into the Florence flask the outlet of which is below the surface of the liquid. Prepare the reagent in one liter quantities only. This reagent should be put into use between three and seven days after preparation. Two bottles should always be on hand. When not in use, store the reagent in a 32 ounce glass stoppered bottle.

Standardize the reagent as follows: Prepare a solution of dry methanol containing a known amount of water - about 8.0000 g. per liter. Titrate 10.0 ml. of this solution with the reagent. The endpoint is a color change from yellow to brown. Call this titration B. Also titrate 10.0 ml. of the methanol used in the preparation of the water standard. Call this titration A. Titration A will account for 10% of the water present in the methanol used for the standard. This correction is negligible. Calc'd: the water factor as follows:

$$(1) \quad \text{Ml. factor} = \frac{\text{g. of water per 10.0 ml.}}{B-A}$$

Calc'd: the amount of water originally present in the liter of methanol.

Titration A x Factor x 100

It will be found that one liter of methanol contains about 1.000 g. of water. This amount plus the amount of water added is the total amount of water in the standard. After the original calculation, the total water in the standard is known, and the following calculation for daily determinations of the factor can be employed:

$$(2) \quad \text{Ml. factor} = \frac{\text{Total g. water per 10.0 ml.}}{\text{ml. Titration}}$$

Formula (1) is used only in determining total water in the standard. Formula (2) is used for all routine standardizations. The total g. of water per 10.0 ml. of standard will be posted on the bottle containing the standard solution.

The standardization is carried out in a clean, perfectly dry 250 ml. Erlenmeyer flask. The factor will be approx. 0.00330 g. H_2O per ml. Standardize the solution every day, immediately before first use in the morning.

The reagent is dispensed from an automatic burette set-up provided with a liter reservoir and a drying bottle provided with alternate layers of glass wool and P_2O_5 . The drying agent must be renewed whenever examination shows that it is necessary. The air used in moving the reagent is drawn from the drying bottle through a pressure bulb and forced into the reservoir. All air vents are connected to the drying bottle.

SPECIFICATION FOR PRODUCT

Product No. 248 Sales Code No. 1040-280-75-03 Date 6-4-41
 Name Aroclor, Distilled - No. 1254 (G. E. Compound No. 1476)
 Chemical Formula Mixture Mol. Wt.
 Superseding Specifications 5-1-36

Property	Specification	Method No.
Color	Yellow tinted viscous liquid 150 APH Max.	10,084-40
Condition	Clear	10,084-40
Specific Gravity 65/15.5	1.495 to 1.505	10,114-40
Acid No. (Mg NaOH per g.)	0.01 Max.	10,087-40
Inorganic Chlorides	0.000015% Max.	10,118-40
Viscosity 98.9°C (210°F)	44-47 seconds, Saybolt Universal	10,086-40
Dielectric Constant 100°C-1000 cycles	4.5 - 4.5	10,119-40
Resistivity - 100°C - 500 Volts	100 x 10 ⁹ ohms/cm ³ Min.	10,120-40
Dielectric Strength	30 K.V. Min.	10,122-40
Refractive Index 25°C	1.6370 to 1.6390	10,125-40
Distillation 10%	360 - 370°C	10,117-40
50%	370 - 380°C	
90%	380 - 390°C	
Four Point (ASTM)	8 - 12°C	10,115-40
H ₂ O	0.0035% Max.	10,820-40
Loss on Heating 100°C-6 hrs.	0.4% Max.	10,116-40
Corrosion Test (Effect of 6 hrs. at 210°C in contact with aluminum)		10,126-40
1) Loss in weight of bright aluminum	None	
2) Inorganic Chlorides	0.000015% Max.	
3) Acid No.	0.01 Max.	
4) Color	400 APH	
5) Condition	Clear	

Sample for Analysis 2 x 1/2 gal. friction top tinned cans for lot samples, 4 x 1/2 gal. friction top tinned cans for advance G.E. samples.

Note: Specific Gravity changes minus 0.00095 per °C (50 to 105°C)

Special Specifications - Tested only on request.

Electrical Stability	No decrease in Resistivity	
(Aging Test)	After 96 hrs. at 100°C	10,121-40
Burn Point	Above boiling point	10,123-40
Total Chlorine	54.5 to 55.5%	10,088-40

0.00075 p.p.c