ORGANIC CHEMICALS DIVISION RESEARCH

St. Louis Research Report No. 2188

FINAL REPORT
ON
INVESTIGATION OF ANALYTICAL METHODS FOR CHLORINATED BIPHENYLS

Job No. 2-02-750.01-3427

June 24, 1958

Written By: G. W. Ashworth

Work Done By: R. E. Keller

Monsanto

ST. LOUIS, MO.

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MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
ST. LOUIS RESEARCH DEPARTMENT

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INTRODUCTION

General Electric Company capacitor failures attributed to Aroclor 1242 with high chloride content have made it necessary to critically evaluate existing analytical methods and to develop more accurate methods to be used in process evaluation, as well as for specification purposes. It was proposed that these methods would replace or supplement inaccurate Tyndall beam procedures used by industry. Work on Aroclor 1242 was concentrated along three lines as follows:

1. To further refine the Monsanto thermal stability method for chlorides developed by Dr. Munch in 1947.

2. To evaluate the aqueous alkali digestion method developed by General Electric for chlorides.

3. To evaluate the alcholic alkali digestion method developed by Westinghouse for chlorides.

Limited work was done on the use of these methods for Aroclor 1254 and 1260.

SUMMARY AND CONCLUSIONS

In the Monsanto thermal stability method volatile hydrogen chloride from the decomposition is swept out of Aroclor 1242 at 210°C. with air, absorbed in water and titrated with silver nitrate solution. Certain impurities if present in chlorinated biphenyls break down at elevated temperatures with the liberation of hydrogen chloride. The results are expressed as parts per million chloride obtained during a 16 hour thermal stability test period. The accuracy and reproducibility of this method have been improved over that obtainable when used at an earlier date for trichlorobenzene. This was accomplished by careful manipulation and close attention to small details.

In the General Electric method titratable chlorides are produced by a 6 hour aqueous alkali digestion of Aroclor 1242 at reflux temperature. The results are dependent upon the rate of stirring and the material of construction of the stirrer. High blanks for high quality Aroclor 1242 and poor reproducibility make the method unsuited for the detection or determination of small amounts of impurities.

In the Westinghouse method titratable chlorides are produced by a 1 hour alcoholic alkali digestion of Aroclor 1242 at reflux temperature. It is rapid, reproducible, requires no special apparatus as does the Monsanto method, gives low decomposition chloride blanks and it has good sensitivity. The ratio of total chlorides to decomposition blank chlorides is greater than that obtained with either the Monsanto or General Electric methods.

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In general, analyses data indicate that the Westinghouse method adequately differentiates between good and bad Aroclor 1242. It does not, however, measure some of those impurities found by the Monsanto thermal stability method. Correlation between analytical data is often poor. Since the methods are testing for different properties, lack of correlation may not be significant. The relationship between these properties and the performance of a finished capacitor is not known. For this reason it is recommended that both the Monsanto and Westinghouse stability methods should be used until sufficient data have been accumulated for correlation with capacitor performance and for the setting of specification figures.

High chloride figures obtained from decomposition studies with typical plant Aroclor 1254 and 1260 show that high blanks are obtained with the Westinghouse method. For this reason, it is possible that the Westinghouse method cannot be used with the higher chlorinated biphenyls.

Data for typical plant material must be accumulated and correlated with performance tests as a basis for specification limits.

ANALYTICAL METHODS

A. Monsanto

The following is a detailed write-up of the Monsanto method for determining the thermal stability of Aroclor 1242.

1. Thermal Stability Method for Aroclors

(See following five pages)

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THERMAL STABILITY METHOD FOR AROCLORS

Scope

This method measures the thermal stability (chloride content) of chlorinated biphenyls used primarily as dielectrics. It is used for determining the quality of finished Aroclors.

Principle

Certain impurities if present in chlorinated biphenyls will break down at elevated temperatures with the liberation of HCl. The volatile HCl is swept out of the sample with air, absorbed in water and titrated with silver nitrate solution. The results are expressed as parts per million chloride obtained during a 16 hour thermal stability test period.

Reagents

1. Acetone. No special grade is required. It must contain no titratable chlorides.

2. 1% HNO_3. Dilute 1 ml. concentrated HNO_3 to 100 ml.

3. 0.005 N$AgNO_3$. 0.8495g to 1000 ml. 5 ml. 0.1 N$AgNO_3$ (if available) to 100 ml.

Apparatus

1. Pressure Regulator. Moore - Model 40 - 2 - 0-50" Water. Moore Products Co. H & Lycoming St., Philadelphia 24, Pennsylvania.

2. Thermoregulator. Cenco - 99015 - (Central Scientific Co.)

3. Relay. Ebert Micrelay SPST Std. Type. Ebert Electronics Corp., Queens Village, N.Y. (Any sensitive, reliable relay can be used).

4. Stirring Motor. Bodine NSI-13 B-2224 1/40 HP. 1725 R.P.M.

5. Bath Fluid. Dow Corning 550. 5 gallons

6. Leeds & Northrup ac. operated pH Meter - Cat. No. 7664

7. Silver wire electrode

8. Mercurous Sulfate Reference Electrode. Modified L & N calomel reference electrode prepared as follows. Dismantle the internal element from the salt bridge tube of a standard L & N calomel reference electrode. Discard the saturated KCl solution from the tube and clean out the mercurous chloride and mercury from the internal element. Clean parts thoroughly. Add sufficient new clean mercury to the internal element to make contact

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with electrode wire and repack chamber of internal element with mercurous sulfate moistened with 0.5M potassium sulfate. Seal the chamber with non-absorbant cotton. Fill the salt bridge tube with 0.5M potassium sulfate and reassemble units.

9. Burette. 1.0 ml. microburette - Koch - Fisher Scientific Co. - 20-110.

10. Magnetic Stirrer and glass covered stirring bar

11. Glass Apparatus for Samples in Bath. See attached diagram.

12. Capillaries. Glass capillaries approximately 0.2 mm in diameter and cut to a length that permits a flow of 45 ml. per min. of air

13. Variac. 2 KVA.

14. Air Supply. Air under 40 lb. pressure is available in our laboratories. This air is purified by passing through a scrubber bottle containing 40% NaOH, an empty bottle which serves as a safety, a second bottle containing conc. H_2SO_4 and a trap immersed in dry ice and acetone. The purified air is connected to a glass manifold having one connection for each sample. Capillaries of the appropriate length are connected between the manifold and the outlet for each sample. In this manner a constant flow of air can be obtained on all samples by applying a constant pressure to the manifold.

15. Heating Bath. A stainless steel bath constructed according to the specifications given in the attached diagram is used. The bath is heated by applying 85 Volts to 3-500 Watt G.E. strip heaters bolted to the bottom of the bath. One 500 Watt Immersion heater is connected to the thermostat. Dow Corning 550 silicone is used as the bath liquid. The temperature is maintained at $210^{\circ} \pm 0.2^{\circ}C$. The bath should be placed in hood and the tests carried out in total darkness. Two 3 X 5" stainless steel plates not shown in the sketch are placed on top of the straightening vanes in the bottom of the bath. This provides better stirring to the ends of the bath. Twelve samples can be run in the bath at one time.

Procedure

Weigh a 290 ± 1 g. sample of Aroclor into the 300 ml. Erlenmeyer flask. Insert the gas inlet tube and position the flask in the $210^{\circ} \pm 0.5^{\circ}C$. bath. The bath fluid level should be approximately one inch below the bottom of the ground glass joint on the flask. Place 10 ml. distilled water in the Volhard flask absorber and attach to the receiver tube from the Erlenmeyer flask. Connect the purified air supply from the capillary to the inlet down tube in the flask. Bubble air through the sample for 16 hours at the rate of 35 to 45 ml. per minute. (The apparatus and/or sample must be kept in the dark during the 16 hour period). Transfer the water from the absorber to a 100 ml. beaker using approximately 50 ml. acetone, and 2 drops 1% HNO_3 solution and titrate with 0.005N $AgNO_3$

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solution using a magnetic stirrer. The titration is stopped at 75 mv. which represents the point of maximum potential change and the titration endpoint. Silver wire and mercurous sulfate electrodes (Ag-Hg, Hg_2SO_4, 0.5M K_2SO_4 system) are used for the titration.

Calculations

(Total ml. 0.005 $NAgNO_3$ used)(0.61) = ppm chloride

1 ml. 0.005 $NAgNO_3$ is equivalent to 0.0001773g. chloride or 0.61 ppm.

Precision and Reliability

The precision of the test (standard deviation) is 0.02 ppm at the 0.5 ppm chloride level and 0.06 ppm at the 2.8 ppm level. The method as written does not necessarily quantitatively measure the total unstable chlorides present. For screening purposes a total chloride figure is not necessary. Experience has shown that there is good correlation between the chloride figures obtained by this 16 hour thermal stability test and the quality of chlorinated biphenyls.

Discussion

The air supply can be checked for chloride contamination by passing the air through an empty sample flask immersed in the bath. Not more than 0.03 ml. 0.005 $NAgNO_3$ should be required to give the endpoint. The air supply can be checked for ammonia by measuring the pH of the absorber solution or titrating with 0.01N HCl. The pH should be between 6 and 7. Compressed cylinder air available for breathing purposes can perhaps be used without any purification. Experience has shown that nitrogen gives low chloride figures. This indicates that air is a necessary part of this test and that nitrogen cannot be used as a substitute. Gum rubber tubing is used in making all connections. The apparatus is cleaned with acetone.

2. Method Development Work

A large sample of Aroclor 1242-Lot T406 was fortified with bad Aroclor (prepared in the laboratory by R. Simone NBP A-99370 containing 38 ppm chloride by the Monsanto test) to give approximately 0.5 ppm and 3 ppm.

a. Reproducibility of Method

6 samples in bath at 210°-16 hrs. in the presence of ordinary fluorescent light.

1 - 0.48 ppm
2 - 0.53
3 - 0.51 Average 0.50 ppm
4 - 0.50 Std. Deviation 0.02 ppm
5 - 0.53
6 - 0.48

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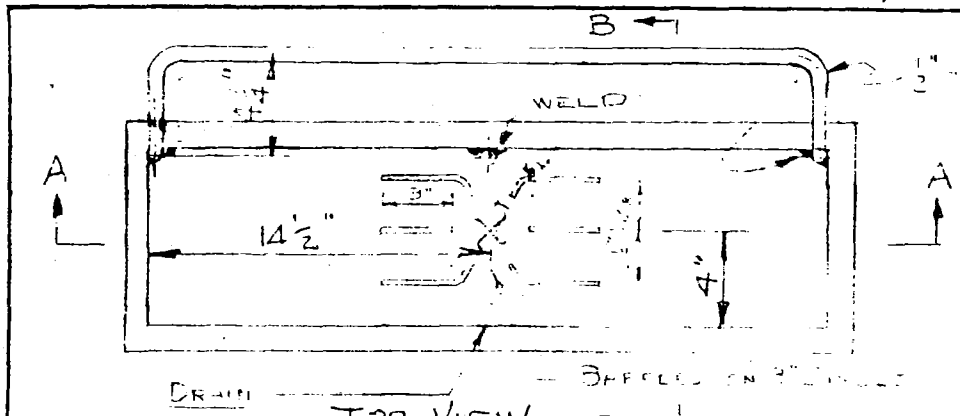
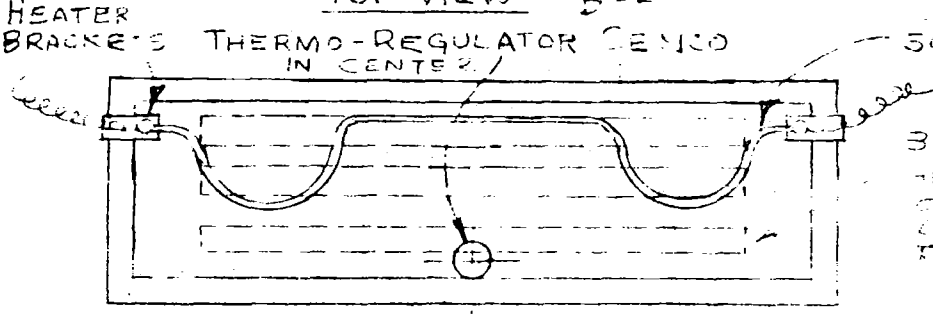
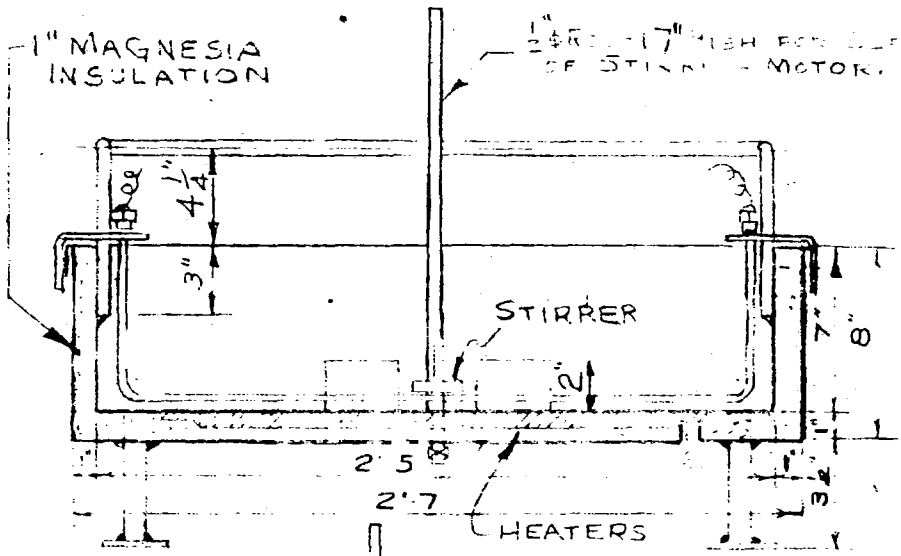


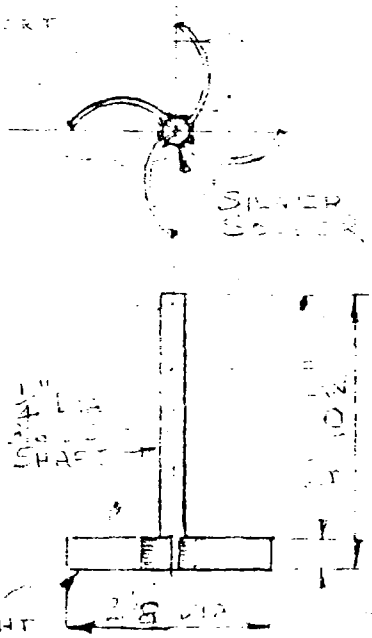
TOP VIEW B-B'



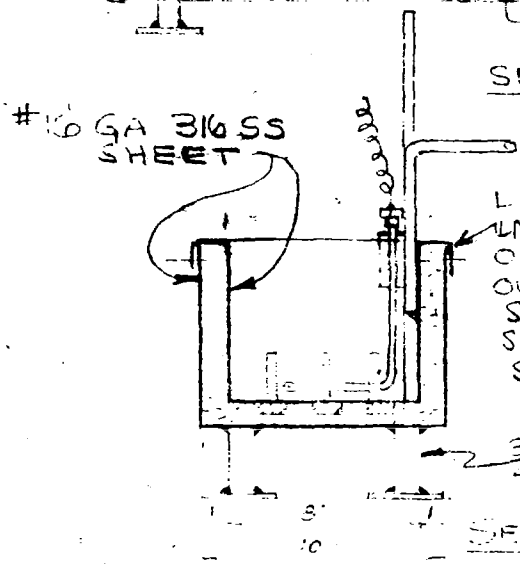
TOP VIEW SHOWING HEATERS



SECTION A-A



STIRRER DETAIL



SECTION B-B

NO.	DATE	REVISION	C'K'D
1	3-28-57	GEN'L REDESIGN	FALEK

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PROJECT

AROCLOR TEST APPARATUS

		APP. BY	
DRAWN BY FALEK	DATE	1	4
CHECKED BY	DATE	2	5
SCALE		3	6

DWG. NO. RA 882

3127

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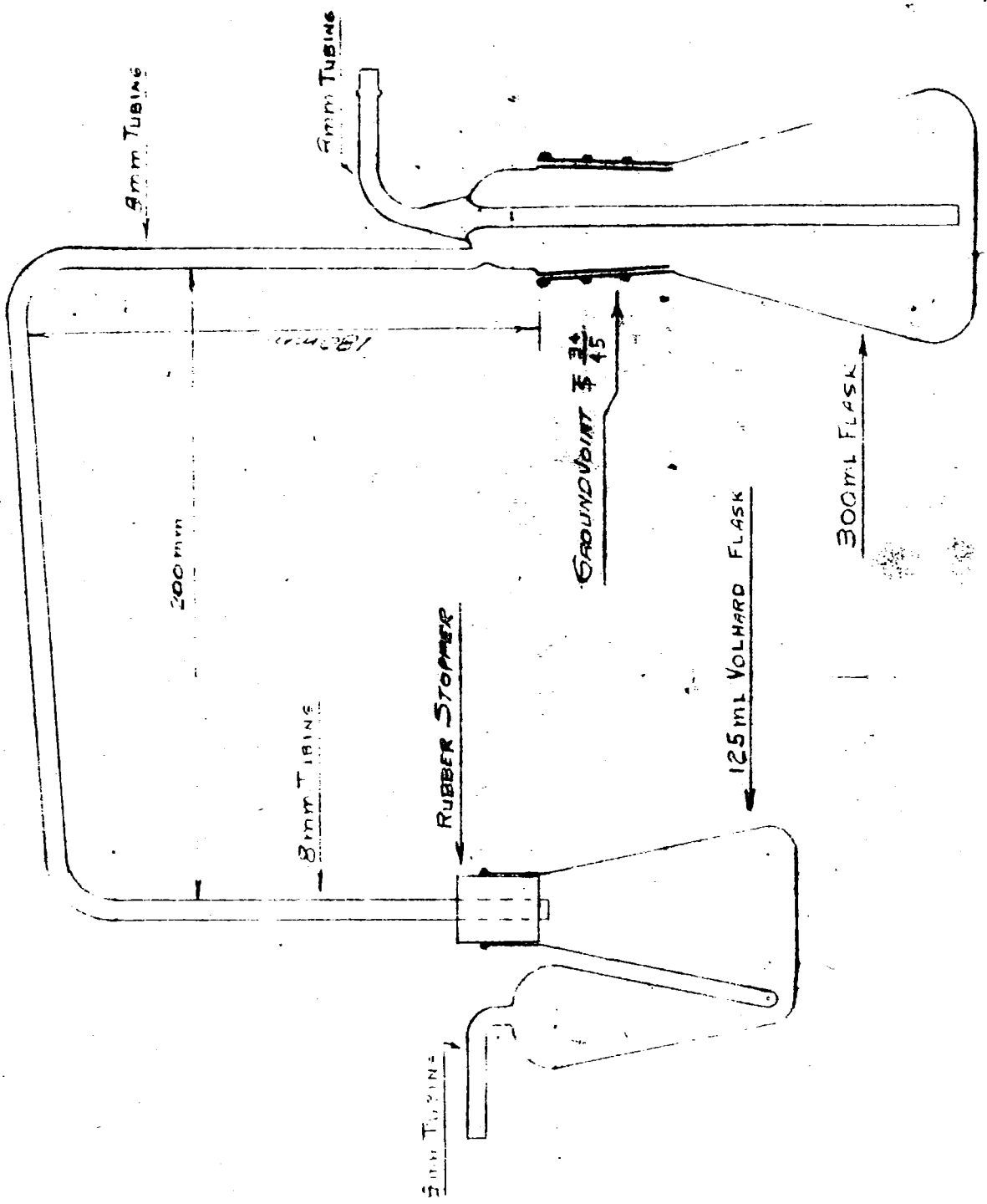

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PROJECT

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NO.	DATE	REVISION	C'K'D

DRAWN BY [illegible] DATE [illegible]

CHECKED BY _____ DATE _____

SCALE _____

APP. BY

1	4
2	5
3	6

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b. Reproducibility of same sample run at different times. These samples were run in complete darkness. (See effect of light)

5-7-57	0.43	
5-14-57	0.40	
5-15-57	0.37	Average 0.41
5-20-57	0.38	Std. Deviation 0.03
5-23-57	0.47	
6-10-57	0.43	

c. Effect of fluorescent light at two levels of impurities. 2-15 Watt white G.E. tubes approximately 12" above bath.

1 - 3.30	
2 - 3.20	
3 - 3.99	Average 3.50
4 - 1.28	Std. Deviation 0.35
5 - 1.14	
6 - 0.86	

Same samples run in total darkness.

1 - 2.60	
2 - 2.82	Average 2.77
3 - 2.89	Std. Deviation 0.06
4 - 0.43	
5 - 0.42	Av. 0.43
6 - 0.43	Std. Dev. 0.02

The results above indicate that light has a pronounced effect upon the absolute values obtained. This can be important in meeting specifications. The average value obtained with general laboratory light was 0.50 while the value obtained in the absence of light was 0.43. Six individual determinations run over a month's period gave 0.41 which is in excellent agreement.

The values from 4-5-6 in the presence of light were not averaged because of the very poor reproducibility. This may be due to the lack of uniform exposure to light on the three samples. It does appear that the impurities are not affected as much by the light as the Aroclor itself.

Since light causes excessive decomposition, all stability tests reported were run in complete darkness.

d. Efficiency of Absorbers

The air is bubbled through the Aroclor at 210° and the HCl swept out with the air and absorbed in 10 ml. of water in a Volhard absorption flask. Many times there is insufficient

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water to form a seal so the air simply passes over the surface of the water. To determine whether this might affect the quantitative recovery of HCl in the air stream water seals were made with three flasks and four were tilted so they were not sealed. The results below show that quantitative recovery is obtained in both cases.

Control - Sealed		Not sealed	
2.60		2.74	
2.82	Av. 2.77	2.81	Av. 2.82
2.89	Std. Dev. 0.06	2.55	Std. Dev. 0.23
		3.18	

e. Effect of Air Flow

The effect of air flow was determined by using approximately one half and twice the standard flow of 45 ml./minute. The results given below indicate the air flow should not be greater than the standard value but can be considerably less.

25 ml./minute		45 ml./minute		90 ml./minute	
2.57		2.60		1.30	
2.67	Av. 2.68	2.82	Av. 2.77	1.32	Av. 1.46
2.79	Std. Dev. 0.09	2.89	Std.Dev.0.06	1.76	Std.Dev. 0.21

3. Analytical Work

a. Aroclor 1254 and 1260

The values obtained by this method are about 0.1 ppm for the best plant Aroclor 1242. Still lower values can be obtained by careful manipulation but the reproducibility is not good at very low concentration levels.

Higher values are obtained with the higher chlorinated Aroclors. The reason has not been definitely established but the chlorides evolved are believed to represent the inherent instability of the higher chlorinated Aroclors and not the same impurities found in Aroclor 1242.

Ten plant samples of Aroclor 1254 were analyzed with an average of 0.41 ppm with a standard deviation of 0.08. Nine samples of Aroclor 1260 had an average value of 0.40 ppm with a standard deviation of 0.10.

b. Rate of Chloride Evolution in Monsanto Test

It is known that all the unstable chloride material is not decomposed in the 16 hour heating period. The rate at which this decomposition occurs was determined on three samples

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changing absorbers every 16 hours for a total of 12 periods or 192 hours. The samples chosen were 1) Lot 512 which was the worst lot made, 2) a synthetic sample of about the same quality by the Monsanto test, and 3) a very good sample of Aroclor 1242 prepared in the laboratory. The results are as follows:

16 Hour Test Period	Anniston Lot 512	Synthetic Sample	High Quality Aroclor
1	2.73 ppm	2.48 ppm	0.23 ppm**
2	1.07	1.01	0.092
3	1.01	0.89	0.085
4	0.99	0.81	0.092
5	0.97	0.76	0.092
6	0.92	0.68	0.085
7	0.86	0.65	0.085
8	0.83	0.59	0.085
9	0.83	0.59	0.085
10	0.83	0.59	0.085
11	0.84	0.56	0.092
12	0.78	0.51	0.085

* Prepared by H. L. Merten NBP 93028

** A previous determination gave 0.10 for the 1st period. The high value here is believed due to contamination.

The above data show that the decomposition is not complete even after 12-16 hour periods. On very poor samples the amount of hydrogen chloride evolved is not directly proportional to the time of the test so that a definite interval must be designated to obtain reproducible and comparable data.

B. General Electric Company Test

The following is the method submitted by General Electric on March 7, 1957. This test is based on the amount of chlorides hydrolyzable by refluxing the sample with 20% sodium hydroxide. The chloride addition compounds are more readily hydrolyzed than the Aroclor itself.

1. Original Method - "Dehydrochlorination Test for Pyranol"

a. Discussion of Method

This method is based on the ability of concentrated sodium hydroxide to remove the elements of hydrogen chloride from an organic compound having hydrogen and chlorine atoms on adjacent carbon atoms, provided that the dehydrochlorination leaves a stable double bond. The ionic chloride thus formed is then titrated potentiometrically with silver nitrate solution.

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b. Apparatus

500 ml. 3-neck, round-bottom flask. Center neck standard taper 34/45 and sides 24/40.

500 ml. heating mantle and 5 amp. variac.

Precision-bore stirrer, 34/45, with teflon paddle, reflux condenser.

Stirrer motor and stand.

500 ml. separatory funnel.

Glass and rubber stoppers.

c. Reagents

20% sodium hydroxide solution (from reagent grade sodium hydroxide).

d. Method

125 ml. of Pyranol are placed in the flask and weighed to the nearest 0.5 g. 125 ml. of 20% sodium hydroxide are then added and the apparatus assembled. The contents of the flask are then heated at a gentle reflux under vigorous stirring for 6 hours. The flask is immediately detached from the rest of the apparatus and clean rubber stoppers placed in the three openings. (Glass joints tend to "freeze" in the presence of sodium hydroxide.)

When cool enough to handle (about 50°C.), the contents of the flask are poured into a 500 ml. separatory funnel and the Pyranol (lower layer) is separated from the sodium hydroxide solution. The Pyranol is then washed twice, each time with 100 ml. of hot distilled water. (The viscosity of warm Pyranol is lower, permitting more efficient washing.) The washings are added to the original sodium hydroxide layer and the total carefully made up to 500 ml. with distilled water.

e. Titration Procedure

1. Apparatus

1 - Beckman Model GS pH Meter
1 - Beckman #1261 Silver Electrode
1 - Beckman #1190-60 Glass Electrode
1 - Micro Automatic Burette 2.00 ml. capacity, 0.01 ml. Divisions. (Scientific Glass Apparatus Co., Bloomfield, N.J. - Cat. No. JM-570)
1 - Magnetic Stirrer
1 - Teflon covered stirring bar 5/8" long by 5/16" dia.
1 - 150 ml. beaker
1 - 25 ml. pipette
1 - 10 ml. pipette
1 - 100 ml. Graduate

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2. Reagents

0.010 N Silver Nitrate
Methanol (chloride free)
50% Nitric Acid (chloride free)

3. Procedure

A 25 ml. aliquot of the final sodium hydroxide solution is pipetted into a 150 ml. beaker and acidified with 10 ml. of 50% nitric acid. 75 ml. of methanol are added from a 100 ml. graduate and the solution is cooled to room temperature before titration. The solution is stirred with a magnetic stirring bar while being titrated potentiometrically with 0.010 N silver nitrate using a silver-glass electrode system and the "B" (expanded range) scale of the Beckman Model GS pH Meter.

4. Calculations

The change in millivolt readings per increment of silver nitrate added (Δ MV/ΔML) is plotted against total ml. of silver nitrate added. In such a plot, ΔMV/ΔML rises rapidly and linearly as the end-point is approached, and falls rapidly and linearly after the end-point has been passed. The end-point is the intersection of these two straight lines and represents the volume of 0.010 N silver nitrate solution required to titrate 25 ml. of the final sodium hydroxide solution.

Results are expressed in mg. of chloride per ml. of final sodium hydroxide solution, using the relationship:

1 ml. 0.010 N silver nitrate = 0.3546 mg. chloride ion. For the 25 ml. samples used in this analysis, this relationship becomes:

1 ml. 0.010 N silver nitrate = 0.0142 mg. chloride ion per ml. of final sodium hydroxide solution.

$$\text{ppm chlorides} = \frac{\text{Total Chlorides - Blank}}{\text{Grams Pyranol}} \times 1000$$

Where: Blank = mg. of chloride per ml. of the original 20% sodium hydroxide solution X 125

Total Chloride = mg. of chloride per ml. of final sodium hydroxide solution X 500

2. Monsanto Modification of the Method

The General Electric procedure as evaluated in our laboratory did not give as reproducible results as must be obtained if small amounts of contaminants are to be determined. The following method developed in our laboratory represents a modified version of this procedure.

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Weigh 170 g. + 0.5 g. Aroclor into a 500 ml. 3 neck round bottom flask. Add 125 ml. 20% (Wt. 1 Vol) sodium hydroxide and weigh the flask and contents with the tantalum wire stirrer and bearing to the nearest 0.1 g. Place in heating mantle, connect reflux condenser and place glass stopper in extra opening. Dow Corning high vacuum grease is used as a lubricant for the ground joints and the bearing. Reflux gently for six hours with vigorous agitation. The stirring should provide a uniform mixture of the two phases. After the 6 hours of refluxing, shut off the stirrer, allow the mixture to cool somewhat and reweigh the flask and contents with stirrer. There should be no loss in weight on refluxing. Adjust to the original weight if necessary by the addition of distilled water and mix thoroughly. At this point there are two alternatives. If only one 6 hour test is desired, the entire mixture is transferred to a 500 ml. bottle and cooled to room temperature. If a second 6 hour test is desired the entire mixture is transferred to a 500 ml. separatory funnel. Rinse the flask with distilled water and allow the flask to drain. The rinses are discarded. When separation of the phases is complete, draw off the Aroclor into the original 3 neck flask. Do not allow any of the alkali solution to go forward with the Aroclor. Transfer the clear alkali solution to a bottle and cool to room temperature. Add 125 ml. 20% sodium hydroxide solution to the Aroclor in the flask, weigh with stirrer in the same manner as was done originally.

The chloride titration is run on an aliquot of the aqueous layer. Pipette 6.25 ml. into a clean dry 150 ml. beaker, add 1 drop thymol blue indicator and concentrated nitric acid drop by drop until fully acid (red) to the indicator. One or two drops excess does no harm. Cool to room temperature, add 100 to 110 ml. acetone and titrate potentiometrically with 0.005 N silver nitrate to approximately 275 mv. using silver wire indicator electrode and mercurous sulfate electrode ($Ag-Hg-Hg_2SO_4-0.5\ M\ K_2SO_4$). Continue the titration in 0.02 ml. increments allowing sufficient time for equilibrium to be established between additions. The end-point occurs between 225 and 175 mv. (The use of the special expanded scale of the Leeds and Northrup pH meter is helpful. 350 mv. true value is expanded to full scale or 700 on the meter.) Calculate the end-point to the nearest 0.001 ml. from the differentials between the increments.

A reagent blank is determined by titrating a 6.25 ml. sample of the 20% sodium hydroxide solution in the same manner as above. This includes any chlorides present in the nitric acid and the acetone used. The ml. 0.005 N silver nitrate obtained in the blank determination is subtracted from the value obtained in the experiment. This net titration volume is used in the calculations.

ppm chloride = 20.86 X net ml. 0.005 N silver nitrate

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3. Method Evaluation Work

An attempt was made to determine the reproducibility of the General Electric Method using the equipment we had available. The high salt concentration interferes seriously with the magnitude of the break at the end-point. The L & N pH meter which we used does not have the sensitivity of the Beckman Model GS specified in the method. For this reason poor reproducibility was obtained. It is believed that the alternate modified procedure gives better reproducibility in a shorter time and it does not require a sensitive meter.

The data obtained using both procedures and Aroclor 1242 are given below.

a. Reproducibility of Titrations

Blank for Sodium Hydroxide Used

General Electric Procedure	Monsanto Modification
	0.060 ml. 0.005 N silver
	0.054 nitrate
6 values < 0.05 ml. 0.005 N	0.060 Av. 0.057 ml.
Silver Nitrate. Poor end-	0.055 Std. Dev. 0.003
point makes exact values	0.060
impossible.	0.055

Sample of Aroclor - 0.43 by Monsanto Test

(Krummrich Plant Lot T 406 + laboratory prepared bad Aroclor 1242, R. Simone NBP A-99370 to give 0.43 by Monsanto Test)

Replicate titrations from one experiment

10.64 ppm		7.13	
7.51	Av. 9.84 ppm	7.13	Av. 7.20 ppm
10.22		7.19	
8.34	Std. Dev. 1.41	7.17	Std. Dev. 0.12
11.26		7.09	
11.05		7.47	

b. Separate Refluxing Experiments

9.84		7.20	
8.65	Av. 10.35 ppm	6.03	Av. 7.08 ppm
12.05		5.60	
11.96	Std. Dev. 1.24	7.45	Std. Dev. 0.98
9.91		7.78	
9.70		8.42	

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Values obtained at three different levels of impurities by original General Electric Procedure and by our modification.

Monsanto Thermal Stability Values	General Electric Test	
	Unmodified	Modified
0.43	10.35	7.08
0.78	11.16	8.97
2.77	21.48	14.68

The data for the blank and the solutions show that quite reproducible results can be obtained. The results from 6 separate experiments have 8 times the standard deviation which shows that the reproducibility of the original method is poor. The fact that considerably higher absolute values are obtained using the General Electric Method suggests that some unknown variable is not being considered.

c. Stirring

General Electric stated that 6 hours refluxing was sufficient to completely hydrolyze the unstable impurities. They did not furnish any information as to how much chloride would be obtained on repeated treatment or from the best quality Aroclor available.

Experiments were run on the sample of Aroclor 1242 (RS NBP A99370) which gave 0.43 ppm by the Monsanto Thermal Stability Method. Three stirrers were used. Glass impeller type , teflon paddle and a tantalum wire stirrer. The Aroclor was separated and rerun the specified number of times.

6 hour periods	Glass Stirrer	Teflon Stirrer	Wire Stirrer	Aroclor Lot 512 Anniston Wire Stirrer
1	15.56 ppm	27.82 ppm	23.84 ppm	335
2	15.52	18.23	18.27	11.0
3	10.88	9.13	9.63	6.1
4	8.05	8.01	7.42	7.1
5	5.84	6.72		6.3
6	4.78	6.92		
7	5.42	4.36		
8	5.40			
9	5.34			

A sample of the best Aroclor 1242 available (prepared in the laboratory by Dr. Merten) was run for three periods with the glass stirrer followed by two periods with the wire stirrer. The results given below show that the stirring is extremely important and must be brought under much better control before reproducible results can be obtained.

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6 hour periods	Aroclor 1242 (HLM, NBP 93028)	
1	5.75 ppm	Glass Stirrer
2	4.84	" "
3	4.88	" "
4	7.38	Tantalum Wire Stirrer
5	6.97	" " "

Teflon stirrer with 20% sodium hydroxide only

6 hour periods	
1	4.46 ppm
2	3.86
3	3.94

The flasks where the teflon stirrer was used showed very bad corrosion on the bottom. In view of the above evidence as to the importance of the stirrer and the difficulty of getting a reproducible blank value for pure Aroclor, further work on the method was not deemed warranted.

C. Westinghouse

1. Westinghouse Method for Aliphatic Chlorine in Aroclor

The following procedure represents the method as used by Westinghouse in October 1957.

a. Reagents

0.1 N sodium hydroxide in methanol (methanol must be free of chloride. Some is good enough as received. Many lots must be distilled from silver nitrate) 0.01 N silver nitrate for chloride titration. (Method did not state if this is in water or methanol)

b. Apparatus

1. 300 ml. round bottom ground joint boiling flask and reflux condenser to fit.

2. Heating mantle or hot plate.

3. Potentiometric titration equipment using silver electrode of about six square centimeters area as indicator electrode and glass electrode as reference electrode. A Precision-Dow automatic titration was used.

c. Procedure

Place 100 ml. of 0.1 N methanol-sodium hydroxide solution in boiling flask, add 10-100 g. sample depending on aliphatic chlorine content, attach reflux condenser and reflux for 1 hr. Cool, neutralize with 3 ml. 1:1 sulfuric acid-water. Titrate

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entire sample potentiometrically with 0.01 N silver nitrate. Calculate amount of chlorine in ppm and report as aliphatic chlorine.

Notes: With the electrode system used, the emf was -0.2V at the start and + 0.05 at the end-point and was carried to + 0.3V. The slope was 0.02 V/0.1 ml. 0.01 N silver nitrate at the end-point.

2. Westinghouse Method As Used by Monsanto

The following procedure describes the Westinghouse method given in Analytical Methods, C, 1, as used by Monsanto.

a. Scope

The method is designed as a rapid, reproducible method for determining the quality of Aroclor 1242. It is well suited to process control as well as analysis of the finished Aroclor.

b. Principle

The chlorine addition impurities in Aroclor 1242 are hydrolyzed by one hour refluxing with tenth normal methanolic sodium hydroxide. The Aroclor 1242 is not hydrolyzed. The chloride formed is titrated with 0.005 N silver nitrate.

c. Reagents

Methanol - chloride free
Sodium hydroxide - A.R. Grade
Sulfuric Acid - A.R. Grade
Silver nitrate
Acetone
Benzene

d. Apparatus

300 ml. round bottom flask with ground joint and reflux condenser. The flask is scratched inside in a few places (with coarse emery paper) to prevent bumping.

300 ml. Glas-col heating mantle
Variac
L & N A.C. Operated pH Meter
Silver indicator and mercurous sulfate reference electrode.
1.0 ml. microburette - Koch - Fisher Scientific Company 20-110.
Magnetic Stirrer and molded teflon covered stirring bar. Standard laboratory glassware.

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e. Reagents and Standard Solutions

1. Approximately 0.1 N Sodium Hydroxide. Weigh out sodium hydroxide pellets in a dry beaker to the nearest tenth of a gram and transfer to the methanol as indicated below and shake occasionally until dissolved.

15.8 g. sodium hydroxide in 1 gallon methanol
11.6 g. " " " 5 lb. bottle of "

2. 0.005 N Silver Nitrate. 0.8495 g. silver nitrate to 1 liter. 5 ml. 0.1 N silver nitrate solution to 100 ml.

3. 1-1 Sulfuric Acid. Add 100 ml. conc. sulfuric acid slowly to 100 ml. water, cool to room temperature and store in a glass stoppered bottle.

f. Procedure

Weigh the 300 ml. round bottom flask to the nearest tenth of a gram. Weigh into the flask the 100.0 $\pm$ 0.5 grams of Aroclor sample.

Add 100 ml. 0.1 N sodium hydroxide measured in a graduated cylinder to the flask and connect the reflux condenser.

Mix the contents by swirling the flask and place in the heating mantle. Apply full line voltage to the mantle until the sample shows signs of boiling or it starts to bubble from the bottom. Turn down the voltage to about 85 volts where the sample should reflux vigorously as indicated by a uniform white mixture.

Reflux for 1 hour $\pm$ 5 minutes.

Transfer the contents of the flask to a 400 ml. beaker containing 3 ml. 1-1 sulfuric acid. Rinse the flask with acetone to effect a quantitative transfer using not less than 25 ml. More may be used if desired.

Cool in ice to room temperature or below.

Titrate with 0.005 N silver nitrate to a potential of approximately 220 mv. At this potential add 0.05 ml. increments of standard silver nitrate solution allowing time for equilibrium to be established after each addition. The end-point is calculated from the data obtained.

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A reagent blank is refluxed and titrated in exactly the same manner as the sample. This blank is run on each bottle of 0.1 N sodium hydroxide prepared.

In analyzing Aroclor 1254 or 1260, 20 ml. of benzene is also added to the sample before refluxing. No other changes in the procedure are necessary.

g. Calculations

The titration data for a blank determination are given below:

Burette Reading	mv.	Mo. Per 0.05 ml.	Incremental Change
0.00	270	5	
0.05	265	7	
0.10	258	13	
0.15	245	20	7
0.20	225	50	30
0.25	175	30	20
0.30	145	10	
0.35	135	10	
0.40	125		

$$\text{End-Point} = 0.20 + \frac{0.05 \times 30}{30 + 20} = 0.20 + 0.030 = 0.230$$

Similar titration values for a high grade sample - 1st run of #3 Still - Lot 175 - 9-25-57.

Burette Reading	mv.	Mo. Per 0.05 ml.	Instrumental Change
0.00	260		
0.10	251		
0.20	240	10	
0.25	230	15	5
0.30	215	42	27
0.35	173	35	7
0.40	138	15	
0.45	123	10	
0.50	113	10	
0.60	103		

$$\text{End-Point} = 0.30 + \frac{0.05 \times 27}{27 + 7} = 0.30 + 0.040 = 0.340$$

Net ml. 0.005 N silver nitrate = 0.340 - 0.230 = 0.110
ppm chloride = 0.110 X 1.773 = 0.19 ppm

1.0 ml. 0.005 N silver nitrate = 0.0001773 g. chloride
or = 1.773 ppm chloride
(100 g. Sample)

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h. Precision and Accuracy

No series of experiments have been run to specifically establish the precision of the method but examination of the limited data obtained indicates a reproducibility of ± 1% of the amount present for the samples with high values (10 ppm or greater) and ± 0.1 ppm absolute for the low values (1.0 and less). This applies only to the analysis of Aroclor 1242.

In the analysis of the higher Aroclors, the method will have to be modified to get acceptable results because of very high chloride figures.

The accuracy of the method for chlorine containing impurities cannot be determined since these impurities have not been identified.

i. Discussion

1. Aroclor 1242

The method as described gives excellent results with Aroclor 1242. The decomposition of the Aroclor itself is 0.10 ppm or less so that no correction is necessary for hydrolysis, the reproducibility of the method is excellent and the time of refluxing is not critical. The values are independent of sample size where a smaller sample than 100 g. is taken because of the large amount of chloride expected.

2. Aroclor 1254

Since the solubility of Aroclor 1254 in methanol is considerably less than 1242 and the density greater, smooth refluxing and mixing of the sample does not take place. The addition of 20 ml. benzene provides smooth refluxing. A reproducible blank of about 0.98 ppm was obtained on one sample of Aroclor 1254. If this represents some hydrolysis of the higher chlorinated biphenyls then another Aroclor prepared in a slightly different manner might not give the same value. The solvent used is also important. Because of the difficulty of stirring in a reproducible manner, the General Electric method was not evaluated for Aroclor 1254.

3. Aroclor 1260

Aroclor 1260 gives much more erratic results than 1254. Blank values are quite non-reproducible at the level of 8 to 11 ppm so that it cannot be used on 1260 with any degree of confidence. Further work may evolve a suitable procedure.

3. Final Westinghouse Method

At the conclusion of this work a final Westinghouse method (Scientific paper 8-0423-P 14) was received but not evaluated. It is included at this point for reference purposes.

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Analytical Test for Hydrolyzed Chlorine in Askarel liquids.

a. Reagents

1. Methanol, C.P.

Remove any chloride impurities by refluxing with silver nitrate for one hour and distilling into a chloride free glass stoppered bottle.

2. Sodium Hydroxide

0.1 N Methanolic. Dissolve 4 grams of reagent grade sodium hydroxide pellets in one liter of the purified methanol.

3. Silver Nitrate 0.01 N

Weigh 1.698 grams of reagent grade silver nitrate into a one liter volumetric flask. Dissolve in 500-700 ml. of purified methanol and dilute to mark with methanol.

4. Boiling Stones

Silicon carbide (carborundum) pellets Grit No. 6.

5. Sulfuric Acid (1:1)

Dilute one volume of C.P. Reagent grade sulfuric acid with one volume of distilled water.

b. Apparatus

1. Glassware

Pyrex flasks, 300 to 500 ml. with 24/40 standard taper joints, reflux condensers with mated 24/40 standard taper joints.

2. Measuring Electrode Silver

One end of a silver bar 1.0 X 3.0 X 0.3 cm is sealed into a suitably sized and shaped glass tube with DeKhotinsky cement. Connection is made to the potentiometer through a copper lead previously soldered to the sealed in end of the bar.

3. Reference Electrode

Either calomel or glass. Calomel electrode. Any commercial electrode with a lead of suitable length. Remove the saturated potassium chloride. Wash thoroughly with distilled water. Refill with saturated potassium nitrate or potassium sulfate solution. Glass Electrode. Any commercial electrode with a lead of suitable length.

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4. Potentiometer

Any potentiometer capable of measuring 0.1 millivolt will suffice with the silver electrode-calomel electrode system. For the silver electrode-glass electrode system a vacuum tube potentiometer will be needed. Hot plate or heating mantle may be used as a source of heat for refluxing.

c. Procedure

Weigh into a 300 to 500 ml. flask to the nearest 0.1 g. 150.0 g. of sample.

Add with a graduated cylinder 100 ml. of 0.1 N methanolic sodium hydroxide.

Add 5 or 6 boiling stones.

Attach the reflux condenser and reflux vigorously for one hour.

Measure the reflux time from the start of boiling.

In a well-ventilated hood transfer the contents to a 400 ml. beaker.

Wash the reflux flask 3 or 4 times with 5 ml. portions of the purified methanol. Add washings to 400 ml. beaker.

Cool to room temperature.

Add 3 ml. of 1:1 sulfuric acid.

Titrate potentiometrically past the break in the curve. Take the mid-point of the inflection as the end-point.

Note: The silver electrode should be cleaned before each use. This is best done by buffing with No. 4/0 emery cloth followed by rinsing with distilled water.

Repeat the determination without the sample to obtain a reagent blank.

d. Calculation

ppm Cl = (ml. silver nitrate for sample - ml. silver nitrate for blank) X 2.36.

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This analytical procedure has been used on some model compounds. With aromatic chlorine compounds: Chlorobenzene, 1,2,4 trichlorobenzene, orthochlorotoluene and parachlorotoluene only 1 to 5 parts per million of chlorine were detected. But benzene hexachloride, (hexachlorocyclohexane), and hexachlorpentadiene showed 28.4 and 33.5 percent chloride, respectively. Chlorocylohexane, another non-aromatic compound, seems to be more resistant to removal of hydrogen chloride, and gave only 500 parts per million of chlorine in the one hour hydrolysis test. This is still several hundred times higher than that of normal aromatic compounds, however. With this test, eight determinations on a deliberately contaminated askarel yielded an average value of 36.5 ppm of hydrolyzable chlorine with a reproducibility (estimated standard deviation) of 1.6 ppm.

4. Method Evaluation Work

The following work was done with the Westinghouse method as used by Monsanto as described in Analytical Methods, C, 1.

a. Reproducibility

Duplicate samples were run on a variety of Aroclor 1242. The results are as follows:

Sample	ppm Chloride Run 1	Run 2	Average
Good lab. sample (HLM NBP 93028)	0.40	0.54	0.47
" " " (check)	0.40	0.39	0.41
G. E. Refined Storage	0.93	0.99	0.96
" " Run 114	174.0	176.0	175.0
" " Run 118	62.0	62.0	62.0
Synthetic Sample	9.40	9.40	9.40 (0.43 by Monsanto method)
" "	14.0	13.8	14.0 (0.78 by Monsanto method)
" "	36.1	35.8	36.0 (2.77 by Monsanto method) (36.3 obtained using 20 g. sample in place of 100 g.)
Anniston Lot 512	327.0	330.0	328.0

The above data show reproducibility within 1% at levels of 10 or above and within 0.1 ppm absolute for the lower levels.

b. Determination of Blank for Aroclor Itself

If one assumes that all the unstable impurities in Aroclor are hydrolyzed in one hour, a second experiment can be run for a much longer period and the increase in the value obtained

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is due to the hydrolysis of the Aroclor. The validity of the assumption can be checked by running several samples for different times. The decomposition per hour will be independent of the experimental time.

Aroclor 1242 - Good Quality Material

Time of refluxing	ppm Chloride Total
1 hr.	0.41
21 hrs.	2.36

This leaves a difference of 1.95 ppm chloride for 20 hrs. refluxing or essentially 0.10 ppm per hour. This would represent the limiting value one could obtain for Aroclor 1242. Since the values obtained with the regular samples are much larger than this no correction is made for the Aroclor itself.

Aroclor 1254 - Lot 130

Time of refluxing	ppm Chloride		Remarks
	Total	per hour	
1 hr.	2.01	-	20 ml. benzene used
3 hrs.	3.94	0.97	" " "
21 hrs.	21.70	0.98	" " "
1 hr.	6.75	-	20 ml. acetone used
16 hrs. 10 min.	47.1	2.5	" " "
1 hr.	2.26	-	stirring - no solvents
18 hrs.	14.45	0.72	" " "

From the above data, the empirical nature of the test is evident. The use of acetone in alkaline solution is open to considerable question because of condensation reactions. The critical nature of stirring in connection with the General Electric method indicates this approach should be avoided. Dilution with benzene seems to produce a very reproducible blank which is about ten times that for Aroclor 1242. Before this blank value is used as a correction, the blank obtained with a series of Aroclor 1254 should be run to determine the true decomposition blank.

Aroclor 1260 - Lot 1598

Time of refluxing	ppm Chloride		Remarks
	Total	per hour	
1 hr.	18.8	-	20 ml. benzene added
2 hrs.	27.3	8.5	
5 hrs. 20 min.	69.0	10.6	

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The extremely high values obtained and the lack of reproducibility suggest that high decomposition blanks are obtained and the method should not be used. Additional work on the method is necessary to give reliable results.

c. Effect of Solvents

In the analysis of Aroclor 1242, reproducible results are obtained without the addition of any solvent. However, in extending the method to 1254 or 1260, the use of mechanical stirring or some means of increasing the solubility of the Aroclor in the alcohol or of lowering the density of the Aroclor so that smooth boiling takes place without overheating.

All data where solvents were used are included for reference purposes. Further work is necessary to draw definite conclusions.

Aroclor 1242
20 ml.

Sample	ppm chloride	Solvent	Remarks
Synthetic Std.	9.40	none - control	0.43 by Monsanto Test
" "	10.80	carbitol	" " "
" "	10.25	acetone	" " "
Best Aroclor	0.41	control	A-93028
" "	4.81	acetone - 21 hr.	

This last result represents 0.22 ppm chloride per hour compared to 0.10 obtained without acetone. Due to the possibility of reaction in the alkaline solution acetone should be avoided.

Aroclor 1254
20 ml.

Sample	ppm chloride	Solvent	Remarks
Anniston Lot 245	5.74	none - control	overheating may cause high results
"	3.52	carbitol	
"	0.95	benzene	
Anniston Lot 130	2.26	none-stirring	1 hr.
"	14.45	" "	18 hrs.-0.72 ppm per hr.
"	6.75	acetone	1 hr.
"	47.1	"	17 hrs.10 min. - 2.5 ppm per hr.
"	2.01	benzene	1 hr.
"	3.94	"	3 hrs. - 0.97 ppm per hr.
"	21.70	"	21 hrs. - 0.98 "

The use of benzene apparently gives very reproducible results although the blank is rather high. Acetone gives a much higher value under the same conditions. The effect

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of the amount of benzene has not been determined. If the increase in chloride obtained is due to the higher chlorinated products being more easily hydrolyzed then another lot of 1254 may give a reproducible blank but not the same absolute amount. If this method is to be used on Aroclor 1254 further developmental work will be necessary.

Aroclor 1260

Sample	ppm chloride	20 ml. Solvent	Remarks
Lot 1598	18.8	benzene	1 hr.
"	27.3	"	2 hrs. - 8.5 ppm per hr
"	69.0	"	5 1/3 hrs. - 10.6 "

The much higher values obtained with 1260 and the lack of reproducibility make this test meaningless at its present state of development.

COMPARISON OF METHODS

A. Types of Impurities Measured

The Monsanto Method measures the thermal stability of Aroclor. The chlorine addition compounds are more unstable than the Aroclor itself but the decomposition is not quantitative for these impurities in the 16 hours of the test. The differences observed between the samples are fairly reproducible but good results are obtained only with extreme care in carrying out the test. The equipment necessary is rather expensive and the test itself requires considerable time.

The General Electric test measures the chemical stability of the Aroclor to boiling 20% aqueous sodium hydroxide. Chlorine addition compounds are more readily hydrolyzed under the conditions but excessive decomposition of the Aroclor itself gives rise to very high and non-reproducible blanks. This makes the method unsuited for the determination of impurities at low levels.

The Westinghouse Method measures the chemical resistance of the Aroclor to boiling 0.1N alcoholic sodium hydroxide. The method is quite specific for chlorine addition compounds. It appears to hydrolyze these quantitatively with no appreciable chloride from the Aroclor itself. This method is the one best suited for small amounts of impurities, is the most rapid and requires less equipment. At the present time much higher blanks are obtained with Aroclor 1254 and very much higher blanks with Aroclor 1260. Further work is needed before applying the method to the higher Aroclors.

There is no known information as to what impurities cause the electrical failure of Aroclor in a capacitor or poor performance in a transformer. It is recommended that data should be accumulated for plant material and correlated with performance. This can be used as a basis for setting specification figures for each method.

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B. Accuracy

Nothing is known as to the accuracy of the various methods since the unstable compounds are not available for method evaluation work.

C. Reproducibility and Sensitivity

The reproducibility and sensitivity are best for the Westinghouse Method on Aroclor 1242. This procedure gives complete hydrolysis of the labile chlorine together with extremely low blanks. The reproducibility of the Monsanto Method is fair but there is much less difference between the bad sample and the blank which makes much greater care necessary to obtain reliable results. The high blanks obtained with the General Electric method and the lack of reproducibility makes this method almost useless for evaluating Aroclors of normally good quality.

D. Simplicity

The Westinghouse method is by far the simplest, most rapid and most reproducible of the three methods. For the analysis of Aroclor 1254 and especially 1260 the Monsanto Method appears the most attractive because of low chloride data compared to very high non-reproducible data obtained with the Westinghouse procedure.

IDENTITY OF SAMPLES

The following is a list of the samples used in the work. A more detailed identification of the samples is given here but not included in the various tables where the analyses of these samples are reported.

A. Aroclor 1242

Good Laboratory Sample - Prepared by Dr. H. L. Merten - NBP A93028. Chlorinated at 135-140°C. with 0.015% catalyst. Best quality of Aroclor 1242 available at the time.

Synthetic Samples - Synthetic samples of poor quality Aroclor 1242 at 3 different quality levels were prepared from Krummrich plant Lot T-406 Aroclor 1242 fortified with some bad material prepared in the laboratory by Mr. R. Simone (A-99370). This poor quality product was prepared by using a deficiency of catalyst in the chlorination and by allowing the temperature to fall too low at one stage. This bad material contained approximately 35 ppm by the Monsanto test. Three different levels of poor quality material were prepared. These gave 0.43, 0.78 and 2.77 ppm by the Monsanto test.

General Electric Refined Storage - This represents Aroclor 1242 from their refined storage tank at Hudson Falls, June 11, 1957. This is current Monsanto production.

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General Electric Run 114)
General Electric Run 118) - This is Aroclor 1242 drained from capacitors made during the time (August, 1956) that poor quality capacitors were being made. The samples represent 2 different batches or run numbers of General Electric. Each run number represents a uniform sample of Aroclor 1242 but this does not correspond to a specific lot of Monsanto Aroclor 1242.

Lot 512 - Anniston - GATX 2988, received at Fort Edward, 8-17-56. This is the poorest lot of Aroclor 1242 produced during this time at Anniston.

A-93048 - Chlor. at 100°C.)
A-93048 - Chlor. at 110°C.) - These are two samples prepared by Dr. H. L. Merten in the laboratory. These were chlorinated at the temperature indicated. 0.03% catalyst was used in both cases.

B. Aroclor 1254

Lot 130 - A 57458 (Ellenburg) This was a regular lot of 1254. A large sample was available for laboratory studies. This lot was later found to contain about 3% biphenyl. This sample was also sent to General Electric for their infrared studies.

Lot 245 - Regular Anniston Production
Lot 247 - " " "
Lot 248 - " " "
Lot 256 - " " "
Lot 257 - " " "
Lot 258 - " " "

C. Aroclor 1260

Lot 1598 - Normal Anniston production
Lot 1589 - " " "
Lot 1600 - " " "
Lot 1602 - " " "
Lot T1016- " Krummrich plant production

COMPARISON OF ANALYTICAL DATA

A limited number of samples have been run by all three methods. These results are given in the table below. The lack of complete correlation suggests that the methods are measuring different impurities. Correlation between these results and the electrical properties of the capacitors is needed for proper evaluation. High Quality Aroclor 1242 will of course give very low values by all methods.

	ppm chloride			
			Monsanto modification	
Sample - Aroclor 1242	Westinghouse	Monsanto 16 hrs.	General Electric wire stirrer 1st 6 hrs.	2nd 6 hrs.
Synthetic Sample	9.4	0.43	7.08	-
" "	13.8	0.78	8.97	-
" "	36.0	2.77	23.8	18.3
Gen. Elec. Ref. Storage	0.96	0.16	7.2	4.7
" " Run 114	175	1.27	176	16.7
" " Run 118	62	1.22	64.1	7.9

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HLM NBP A-93048 chlor. at 100°C.	1.94	0.20	9.8	5.7
HLM NBP A-93048 chlor. at 110°C.	1.60	0.12	8.0	5.9
Anniston Lot 512	328	2.73	335	11.0

Aroclor 1254 and 1260

The General Electric method was not run on these samples. The 1254 samples were corrected for 0.97 ppm (Westinghouse method) which was the reproducible value obtained on one sample of 1254. The values for the 1260 are not corrected.

Sample	Monsanto	Westinghouse
Aroclor 1254 - Anniston Lot 245	-	0.38
" " - " " 130	0.31	1.44
" " - " " 247	0.57	1.10
" " - " " 248	0.49	0.63
" " - " " 256	0.43	0.77
" " - " " 257	0.43	0.63
" " - " " 258	0.31	1.02
" 1260 - " " 1589	0.40	22.7
" " - " " 1598	0.40	19.2
" " - " " 1600	0.46	17.0
" " - " " 1602	0.33	17.7
" " - Krummrich " T1016	0.64	19.2

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NOTEBOOK PAGE NUMBERS

Monsanto Method

A-81876 - 78 Incl.
A-95056, 95059, 95062, 95065, 95075, 95082, 95085-to 1 test period, 95087, 95086, 95089, 95092, 95091, 95093, 95094, 101406, 101408, 101413, 101434, 101435, 101446, 4308, 4334

General Electric Method

A-95079, 95080, 95083, 95084, 95088, 95096, 95097, 95098, 95099, 95100, 101401-101405 Incl., 101408, 101413

Westinghouse Method

101413, 101432, 101433, 101434, 101435, 101436, 101437, 101438, 101444, 101445, 101446, 4308, 4334

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sk/ 12/24/58 R. E. Keller G. W. Ashworth

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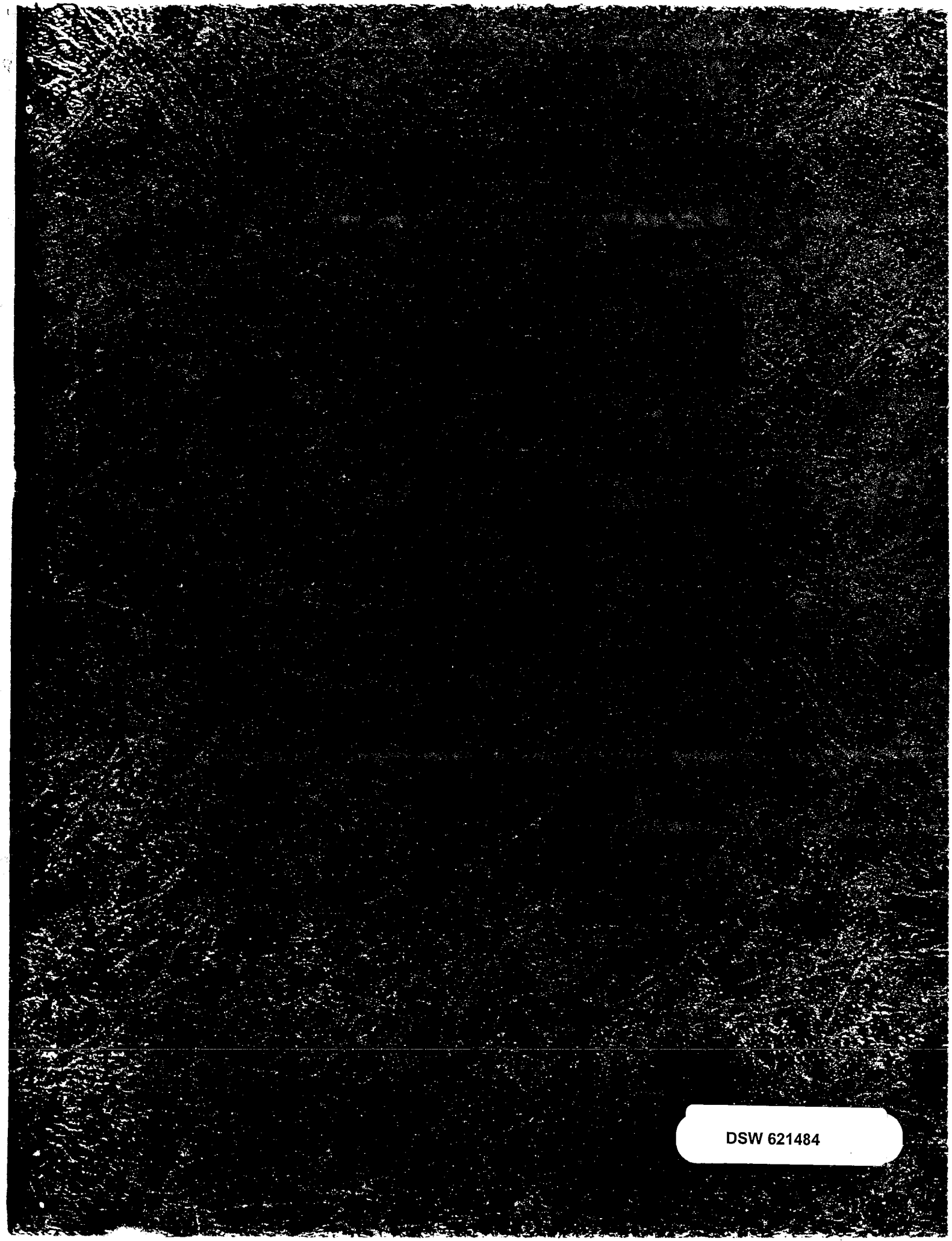
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