INDUSTRIAL CHEMICALS CO.

RESEARCH & DEVELOPMENT

Report

REPORT NO.- P-1733

JOB NO.- 43-000-760.22-1648005
43-000-760.22-1348305

PROCESS FOR MANUFACTURE OF
INTERIM QUANTITIES OF MCS 1043

DATE - June 18, 1973

WRITTEN BY - D. R. Cova

Monsanto

ST. LOUIS, MISSOURI

COMPANY CONFIDENTIAL

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MONSANTO INDUSTRIAL CHEMICALS COMPANY

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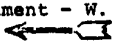
15. " "  "
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INTRODUCTION

MCS 1043 is a chlorinated biphenyl containing 28-29% chlorine as compared to Aroclor 1016 which contains 41.3% chlorine. MCS 1043 contains low concentrations of tetrachlorobiphenyls and essentially non-detectable levels of pentachlorobiphenyls. Its use is in dielectric fluids.

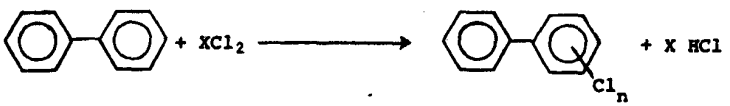
It was made in drum quantities in the Monsanto Research Corporation pilot plant in Dayton, Ohio. The purpose of this report is to describe the procedure for manufacture of interim quantities in the Aroclor department at the W. G. Krummrich Plant.

This process was written to cover specifically the case of a short plant run (probably only one fractionation batch) and was tailored to fit into the Aroclor 1016 equipment at the W. G. Krummrich Plant. It is for this reason that product yields are low (60% of theoretical). With an extended run in proper facilities, product yields would be higher (about 88% of theoretical). The reason for this is that with an extended run it would be possible to recycle some of the non-product streams from the process.

SYNOPSIS OF THE PROCESS

Aroclor 1129 is manufacturered by chlorinating biphenyl. This material is then fractionated under vacuum through a 30 plate sieve tray column. This is a batch fractionation in which two cuts are taken. The first cut is a biphenyl-rich forecut. The second cut is the main fraction. This fraction after Porocel treatment is MCS 1043. The stillpot residue can be distilled in a straight takeover still and then converted to Aroclor 1254. The forecut can be recycled to chlorination to make Aroclor 1016 or 1254.

CHEMISTRY OF PROCESS



Where X = 1.76 for Aroclor 1129. Theoretically, n can range from 0 to 8. However, for Aroclor 1129 homologs with n greater than 6 have not been detected. The distribution of homologs resembles a normal statistical distribution. For Aroclor 1129 the dichlorobiphenyl isomers (n = 2) comprise the major homolog in the mixture.

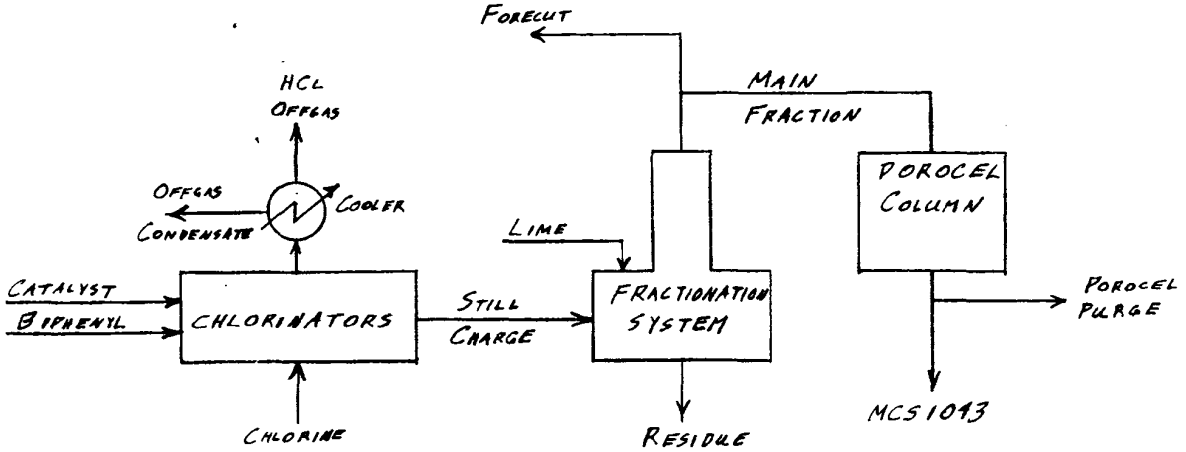


FIG. 1

FLOW DIAGRAM FOR MCS 1093

TABLE I

MATERIAL BALANCE FOR MCS 1043

Component \ Stream	Biphenyl Feed	Catalyst Slurry	Chlorine Gas	Offgas Condensate	HCl Offgas	Aroclor 1129 Still Charge	Lime	Forecut	Main Fraction to Porocel	Stillpot Residue	Porocel Purge	MCS 1043
Biphenyl	113.9	6.1				4.2		4.1	0.1			0.1
Aroclor				1.6	0.03	161.3		29.3	103.9	28.0	4.0	99.9
Lime							0.6			0.6		
$FeCl_3$		0.06				0.06				0.06		
Chlorine			97.0									
HCl					49.9							
TOTAL	113.9	6.2	97.0	1.6	49.9	165.6	0.6	33.4	104.0	28.6	4.0	100.0
Temperature, °C	100	100	25	40	40	190	25	65	65	275	65	65
Volume, Gal.	14.2	0.8	535	0.1	562	17.5	-	3.8	10.1	2.8	0.4	9.7

BILL OF MATERIALS

The following quantities of raw materials are required to produce 100 lbs. of MCS 1043:

Biphenyl	120.0 lbs.
Chlorine	97.0 lbs.
$FeCl_3$ Catalyst	0.06 lbs.
Lime	0.6 lbs.

Product yield on both biphenyl and chlorine is 60% of the theoretical. Most of the remaining 40% will be converted to Aroclor 1254.

PROCESS IN DETAIL

A. Chlorination

The chlorination is carried out in the 4-stage continuous chlorination system presently used at the W. G. Krummrich Plant for making Aroclor 1142. Except for a lower usage of chlorine the manufacture of Aroclor 1129 is very similar to that for Aroclor 1142.

The chlorination is best started up by charging appropriate amounts of biphenyl and $FeCl_3$ catalyst to each stage and then chlorinating each stage up to its normal operating level. The following table gives the chlorine concentration and specific gravity for the product overflowing each stage:

Table II

Chlorination Levels in Each Reactor Stage

Stage No.	Wt. % Chlorine in Product	Specific Gravity of Product at 65°C
1	8.8	1.041-1.061
2	16.9	1.100-1.120
3	23.4	1.152-1.172
4	29.0	1.210-1.220

After each stage has been chlorinated to its proper level, the system can be placed into the continuous mode. 113.9 lbs. of biphenyl and 6.2 lbs. of catalyst slurry (0.06 lbs. of $FeCl_3$ in biphenyl) are fed to the first chlorination stage. A total of 97.0 lbs. of chlorine gas is fed to the reactors. Each stage is fed an equal portion of chlorine (or 24.2 lbs.). The chlorine gas feed rate to each stage is further adjusted so that the product overflowing each stage falls within the specific gravity range given in Table II.

Reaction temperatures for each stage will be the same as for Aroclor 1142, 150°C in the first stage, 160°C in the second, 180°C in the third, and 190°C in the last stage.

HCl gas is given off as a product of chlorination. This gas carries with it some biphenyl and Aroclor. The gas from all the stages is collected in a common header and is then cooled to 40°C. This causes condensation of 1.6 lbs. of organic material from the gas stream. This offgas condensate is collected as a separate stream. The cooled HCl offgas (49.9 lbs.) is piped to the chlorosulfonic acid department. At this point it contains 0.03 lbs. of Aroclors.

165.6 lbs. of Aroclor 1129 overflows the last chlorination stage and is collected for batch fractionation in the 30-tray column in the department.

B. Fractionation and Porocel Treatment

The fractionation is carried out in the 30-tray column presently used in the manufacture of Aroclor 1016. The system is operated under vacuum with a pressure of 40 torr at the head of the column. This should give a pressure in the stillpot of about 145 torr. Under these conditions stillpot temperatures will range from 230°C at the start of distillation to 280-290°C at the end of the cycle. Column head temperatures will rise from 165°C at the start to 215°C at the end of the distillation. The distillation is carried out batchwise.

Aroclor 1129 is charged to the stillpot in the amount of 165.6 lbs. 0.6 lbs. of lime is added to the charge to react with dissolved HCl in the charge and with any traces of HCl formed during distillation. The system is placed under vacuum and heat is applied to the reboiler. The system is brought to total reflux and is allowed to line out. After lining out, distillate flow to the forecut receiver is started at a reflux ratio of 15/1. Flow to the forecut receiver is maintained until biphenyl concentration has fallen to 0.2%. At this point the forecut will be finished.

Biphenyl concentration can only be obtained by gas-liquid chromatography (GLC). It will be necessary then to sample the distillate frequently as the end of the forecut approaches. Since some time will be required to generate analytical results it will be necessary to place the column on total reflux during this time. When the result has been obtained, it can be decided whether or not to continue with the forecut. In this way, 33.4 lbs. of forecut will be obtained.

When the forecut is finished, the distillate flow is diverted to the Porocel column. Distillate temperature should be adjusted to 65°C before passing through the Porocel unit. The first 4.0 lbs. of distillate passing through the Porocel column will be the Porocel purge. It should be collected separately from the main fraction. When the purging has been completed, the flow of Aroclor from the Porocel unit is diverted to the MCS 1043 storage tank. The reflux ratio is reduced to 3/1. This reflux ratio is maintained until a total of 112 lbs. of distillate has been collected (this includes the forecut and Porocel purge). At this point, the reflux ratio is increased to 5/1 and is maintained there through the remainder of the distillation.

The distillation is finished when the overall concentration of 4,4'-dichlorobiphenyl (4,4') in the MCS 1043 in the storage tank approaches 2.0%. It is desirable to approach close to the 2.0% level (say 1.8%) in order to keep up the yield and to keep at a high level the concentration of 2,4'-dichlorobiphenyl in the MCS 1043. This latter component has valuable dielectric properties. It will distill over before the 4,4', but there will be some mingling of the two components. The endpoint of the distillation will be best found by chromatographic analysis of storage tank samples. This means that it will be necessary to place the column on total reflux while awaiting the results of analyses. Fig. 3 in the appendix gives a plot of 4,4' concentration in distillate versus crystallizing point. We estimate that at the point at which the 4,4' concentration in the storage tank approaches 2.0%, the instantaneous concentration of 4,4' in the distillate coming off the column will be about 20%. This however is only an estimate. We recommend that when the instantaneous concentration of 4,4' rises over 7%, the column be placed on total reflux and the storage tank contents be analyzed.

In this way, 100.0 lbs. of MCS 1043 is collected in the storage tank. The fractionation is now shut down and the stillpot residue is pumped to residue storage to await straight takeover distillation. Residue distillation should be the same procedure as the distillation of the stillpot residue from the Aroclor 1016 process.

DISCUSSION OF VARIABLES

A. Chlorination

1. Reaction Temperatures

The reaction temperatures chosen were the same as those for Aroclor 1142. It is recommended that higher temperatures not be used, because otherwise losses to the offgas

condensate stream will be too large. Lower temperatures may give a product that is less stable.

2. Continuous versus Batch Chlorination

Laboratory process work used batch chlornated Aroclor. Comparisons of batch and continuously chlorinated Aroclors have led us to the conclusion that continuous chlorination will be adequate for this case. Nonetheless, the Aroclor 1129 produced should be checked against the tentative specifications given in the appendix. It is likely that continuous chlorination will give a product with higher biphenyl concentration than would batch chlorination. However, we believe that it will fall well within the specifications.

3. Percent Chlorine in Product

The crude Aroclor should contain 29 weight % chlorine. A lesser degree of chlorination will leave too much unreacted biphenyl in the crude product. This could cause a large increase in forecut size at the expense of the main fraction. A higher degree of chlorination would cause a larger stillpot residue and a smaller forecut. Up to 31% chlorine, the size of the main fraction may not be affected very much. Above that point we will lose yield.

4. Chlorine Distribution

The chlorine gas should be approximately equally distributed to all four reaction stages. Any gross variations from an equal distribution could lead to changes in Aroclor homolog distribution such as increased biphenyl and residue component concentrations.

B. Fractionation and Porocel Treatment

1. Column Pressure and Temperatures

A head pressure of 40 torr was chosen. With this pressure and an estimated column pressure drop of 105 torr, the stillpot pressure will be 145 torr. With this pressure in the stillpot, temperatures will not exceed 300°C. Temperatures below 300°C are desirable to prevent possible thermal decomposition. Therefore, a head pressure of above 40 torr should not be used. Lower pressures will only reduce column capacity and increase column vent losses.

2. Distillate Freezing Points

There should be no difficulty here. The highest distillate freezing point should not exceed 65°C except possibly for the

first small portion of forecut. If this is relatively pure biphenyl the crystallizing point could be 70°C but no higher.

3. Reflux Ratios

The recommended reflux ratio program was demonstrated in the laboratory. There should be no need to deviate from this program, unless the biphenyl content of the crude is too high. In this case it would be necessary to run through the forecut at a higher reflux ratio to reduce the forecut size.

4. Column Control

There are four points in this fractionation where action has to be taken. These are: (1) the end of the forecut, (2) the end of the Porocel purge simultaneous with a lowering of the reflux ratio and the beginning of the product fraction, (3) an increase in reflux ration, and (4) end of the distillation.

The end of the forecut is best found by chromatographic analysis. This is the only accurate way in which low levels of biphenyl can be detected.

The second point, the end of the Porocel purge, is best determined by the volume fed through the Porocel bed. In other words, when the distillate integrating flow meter has indicated that a volume equivalent to 4.0 lbs. has passed through the Porocel column, the flow from the Porocel column is then diverted to product storage and the reflux ratio is lowered.

The third point, at which the reflux ratio is increased, is again best detected by the integrating flow meter. When the meter indicates that a total volume of distillate equivalent to 112.0 lbs. has been taken off, the reflux ratio is increased.

The last point, the end of the fractionation, is determined by the concentration of 4,4' in the product in the storage tank. When this level approaches 2% the fractionation is finished and the distillate flow must be cut off. The level of 4,4' in the product can only be accurately determined by gas-liquid chromatography. As an estimate only, the crystallizing point of the distillate coming off the column can provide a guide to the approach of the end of the distillation. When the crystallizing point of the distillate rises to +10°C, the end of the distillation is near. This

crystallizing point test should only be applied after point 3 in the cycle has been passed, because there will be distillate with higher crystallizing points in the early part of the forecut and in the first portion of the main fraction.

5. Product Changeover

In going from Aroclor 1016 to MCS 1043 the greatest possibility of contamination is after fractionation, specifically in the Porocel bed and in piping and the storage tank. The storage tank and piping should be carefully cleaned. The last material through the Porocel bed will have been Aroclor 1016 from the end of the fractionation. This material from the end of the fractionation will contain high levels of highly chlorinated biphenyls (the High Boiling Homologs or HBH). This material should be displaced from the Porocel bed. This can best be done by circulating Aroclor 1016 from the storage tanks through the Porocel bed until the high HBH material has been displaced. The Porocel bed will now contain Aroclor 1016 which has a relatively low HBH content (0.4% is the specification, but product analyses generally show in the range of 0.1%). At this point, the Porocel bed should be drained of Aroclor 1016 as well as possible. However, Aroclor 1016 absorbed in the packing cannot be removed. The Porocel purge with 4.0 lbs. of MCS 1043 distillate will displace some of this Aroclor 1016, but there is no doubt that some will remain to leach out into the MCS 1043 main fraction. Contamination (within reasonable limits) with Aroclor 1016 or Aroclor 1142 before fractionation should give no problem, because the fractionation procedure will keep this material in the reboiler.

Going from MCS 1043 to Aroclor 1016 will also require precaution and cleanup. Any equipment (as, for example, intermediate Aroclor 1129 storage) which has contained Aroclor 1129 should be carefully cleaned before any Aroclor 1142 is placed in it. The contents of the chlorinators at the end of the MCS 1043 run can simply be chlorinated up to their proper chlorine levels for Aroclor 1142 production. The first Aroclor 1016 distillate off the Porocel units should be examined for biphenyl content. It may be necessary to discard a first small portion of the first Aroclor 1016 run. The Standard Manufacturing Process for Aroclor 1016 describes the changeover from MCS 1109 (monochlorobiphenyl) to Aroclor 1016 and this may provide some guidance for this changeover.

MATERIAL SPECIFICATIONS AND ANALYTICAL PROCEDURES

Material specifications are given in Appendix A. Included are tentative specifications for the crude, Aroclor 1129. These specifications on the crude should be met if the refining system is to produce satisfactory MCS 1043.

Analytical procedures are given in Appendix B. Also, method numbers for standard methods are given in the specifications in Appendix A.

CONTROL ANALYSES

Control analyses are required in both process steps, chlorination and fractionation.

In chlorination, specific gravities must be run on effluent from each chlorination stage and especially from the last stage. The sampling procedures and frequency used now for making Aroclor 1142 should be adequate for this process. Samples of Aroclor 1129 should be analyzed by GLC to ensure that the material meets the tentative specifications. A 16 oz. sample taken once a day should be sufficient for this purpose.

In fractionation, control samples will be needed to determine the end of the biphenyl forecut and to determine the end of the fractionation. As the end of the forecut nears, samples of the instantaneous distillate should be taken for every 1 or 2% of the charge distilled (approximately 200 to 400 gallon increments). A 16 oz. sample would be sufficient. A GLC analysis to determine biphenyl content will be required.

In determining end of the fractionation, it will be necessary to analyze both the material accumulated in the distilate receiver and the material accumulated in the product storage tank. GLC analysis will be required, and a 16 oz. sample should be sufficient. As the end of the fractionation nears, it is recommended that samples be taken for every 1 or 2% of the charge distilled (an increment again of 200 to 400 gallons of distillate). From analysis of these two samples it can be decided whether to take off more distillate or to shut down.

TOXICITY AND HAZARDS

The following section has been lifted bodily from the report "Suggested Procedure for MCS 1043" by J. E. Silver (Report No. P-1584).

SAFETY & TOXICITY DATA

Safety Equipment

The following is a list of safety equipment (personal and departmental) required in department 246.

1. Fire extinguishers:
   - A. Dry Chemical (3) - Southwest corner Bldg. CR - Main Floor.
     - West wall at top of stairs of Second Floor.
   - B. Carbon Dioxide Snow - Southwest corner of Bldg. CR, ground level.
2. Chlorine Gas Masks (4) - Southwest corner, second floor
   - Control room, second floor
   - East side, outside control room
   - Wall, Southwest corner of sprinkler house, main floor.
3. Scott Air Masks - West of Lean-To
4. Safety Showers (2) - Southeast corner of Bldg. CR, ground level.
   - Southeast corner of Bldg. CR, 2nd Floor.
5. Davis Vapotester
6. Eye Baths

Safety Precautions

The following are safety precautions followed in the operation of department 246.

1. Safety Equipment of Employee
   Each employee has the following safety equipment: (1) Hard hat (2) Safety glasses (3) Goggles (4) Rubber and canvas gloves Special equipment may be issued as necessary for performing a particular job in a safe manner.

SAFETY & TOXICITY DATA (Cont'd.)

Safety Precautions (Cont'd.)

2. Sampling of Crude Aroclor
The samples of Aroclor taken during the chlorination reaction are hot (170°C.). Precaution must be taken to avoid skin contact of samples and inhalation of Hydrogen Chloride, Chlorine and Aroclor vapors.

3. Fires
Fires may result from the ignition of leaking gas, Xylene, Diphenyl, rags, paper, wood, etc. Automatic fire extinguishers and sprinklers have been installed to combat fires.

4. Explosions
Explosions may be caused from accumulated pockets of natural gas or a rapid burning of Diphenyl in a closed container such as a Chlorinator. Chlorinators are equipped with a 3" diameter pressure relief line with a rupture disc made to burst at 47 psi in case Diphenyl within the Chlorinator ignites.

5. Toxicity
Toxicity does not present a serious problem. However, a complete daily change of clean clothes is supplied to each person and time is allotted at the end of each shift to take a bath. Instructions are also issued to avoid actual skin contact with Biphenyl and Aroclor as much as possible and to wash hands and face before eating.

6. Miscellaneous
There are many accidents that can occur that may be classified as health and safety hazards. These accidents may result from such things as broken process lines, acid leaks, steam leaks, etc.

The worst inherent hazards are as follows:

1. Free Chlorine from ruptured line or major leak.
2. HCl Gas from ruptured line or major leak.
3. Dropping bottoms from stills.
4. Hot Aroclor & Diphenyl.

AROCLOR

Toxicity: This material is a liquid under normal conditions and has a medium toxicity range for liquid ingestion and high toxicity for vapor inhalation. The maximum allowable concentration (M.A.C.) is 1 mg/cubic meter.

Fire Hazard: There is no fire hazard under normal conditions. Aroclor mixes (Xylene, Toluene, Butyl Acetate, etc.) may burn and explode at high temperatures when exposed to flames.

SAFETY & TOXICITY DATA (Cont'd.)

Other Hazards: This material can cause dermatitis, systemic poisoning from the fumes, and yellow atrophy of the liver. There are skin, mucous membranes and eye hazards encountered in handling this material.

Treatment and Antidotes: Call a physician and remove patient from fumes.

Storage and Handling: Protect the skin, lungs, and eyes by wearing approved protective clothing, respirator, and goggles. People who handle this material at elevated temperatures should have medical supervision, and the air should be sampled to insure compliance with M.A.C. There are no special storage restrictions under normal conditions except store in a ventilated area.

Shipping: There are no special shipping restrictions.

FERRIC CHLORIDE ($FeCl_3$)

Toxicity: This material is a solid under normal conditions and is non-toxic or has a rating as low.

Fire Hazard: There is no fire hazard under normal conditions, but it will emit toxic fumes at high temperatures.

Other Hazards: Some people are allergic to this material and may develop skin rash, and there is an eye hazard.

Treatment and Antidotes: Call a physician.

Storage and Handling: Protect the skin with approved protective clothing, avoid the breathing of $FeCl_3$ dust, wear approved respirator with adequate cartridge, and protect the eyes with approved goggles. There are no storage regulations.

Shipping: There are no shipping restrictions.

DIPHENYL ($C_6H_5C_6H_5$)

Toxicity: This material is a solid at normal conditions and is medium toxic. The M.A.C. of vapor is 2 mg/cubic meter of air. If ingested, the lethal dose is 2.4 g/Kg of body weight. The fumes are not dangerously deadly, but they are poisonous over a prolonged time.

Fire Hazard: Diphenyl is very flammable and will burn when exposed to heat or flame or oxidizing materials.

Other Hazards: Diphenyl is not too corrosive to skin, but it will affect the eyes.

SAFETY & TOXICITY DATA (Cont'd.)

DIPHENYL ($C_6H_5C_6H_5$) (Cont'd.)

Treatment and Antidotes: Wash eyes with water and get the patient to fresh air. Try to get patient to vomit, and call a physician.

Storage and Handling: This material can be stored in steel containers away from fire hazards and oxidizing agents. Wear suitable clothes and approved goggles when handling diphenyl.

Shipping: Ship in cars or other suitable containers with a red warning label of flammability.

LIME $Ca(OH)_2$

Toxicity: Lime is not dangerously toxic, and it is regarded as more of a nuisance. This material is a solid or slurry.

Fire Hazard: This material is not flammable.

Other Hazards: Lime is very hazardous to the eyes. It will burn the skin also, and the dust will affect the mucous membranes.

Treatment and Antidotes: Flood eyes and skin with large quantities of water. If lime is ingested, get patient to vomit and call physician.

Storage and Handling: Lime can be stored in steel containers, and people should wear protective clothing, respirators, and rubber or chemical safe goggles when handling lime.

Shipping: There are no special restrictions for shipping, but the containers should have a white label, a caustic warning.

SAFETY & TOXICITY DATA (Cont'd.)

ATTAPULGUS EARTH:

Toxicity: This earth is not toxic in general.

Fire Hazard; There is no fire hazard.

Other Hazards: Attapulgus earth can create a dust hazard similar to other dusts that contain silica.

Treatment and Antidotes: Consult a physician.

Storage and Handling: Attapulgus earth presents no special storage problem and should be handled by persons wearing an approved respirator.

Shipping: There are no special restrictions on shipping.

HYDROGEN CHLORIDE (HCl)

Toxicity: This material is a gas under normal conditions and is very toxic. The M.A.C. is 5-10 ppm and a concentration of 1500-2000 ppm would be fatal to humans in a few minutes.

Fire Hazard: There is no fire hazard under normal conditions, but wet gas reacts with some metals to form hazardous hydrogen.

Other Hazards: This material is hazardous to teeth, skin, eyes, and mucous membranes.

Treatment and Antidotes: Wash profusely with water, if injested, eat chalk, magnesium hydroxide, egg whites, and milk, and also administer oxygen. Call a physician.

Storage and Handling: This material should be stored in a cool, well ventilated area away from oxidizing agents. This material can be stored in rubber lined, glass, or tantalum vessels. People handling this material should wear suitable clothing like rubber aprons, shoes, and gloves. Also rubber goggles or chemical safe grades and approved pack or canister gas masks.

Shipping: Shipping containers must have a white warning label and a non-flammable label. Ship in appropriate containers such as rail car or carboy.

CHLORINE (Cl_2)

Toxicity: This material is a gas under normal conditions and is very toxic. The M.A.C. is .35 to 2 ppm. A concentration of 1000 ppm would be rapidly fatal. Least concentration with detectable odor is 3.5 ppm

SAFETY & TOXICITY DATA (Cont'd.)

CHLORINE (Cl_2) (Cont'd.)

Fire Hazard: There is no fire hazard under normal conditions, but there is an explosion hazard if chlorine is mixed with a strong reducing agent such as hydrogen or hydrocarbons at high temperature.

Other Hazards: This material causes eye, skin, and mucous membrane irritation

Treatment and Antidotes: The patient should be given oxygen immediately and treated for shock. Call a physician.

Storage and Handling: This material should be stored in a ventilated and cool place away from fire hazards. When handling, wear a gas mask or approved respirator, clothing and rubber or chemical safe goggles. In strong chlorine concentrations, wear canister gas masks, Scott air pack, or oxygen breathing apparatus.

Shipping: The shipping containers must have a green gas label and a non-flammable label on. Ship in steel cars or cylinders.

ENVIRONMENTAL PROTECTION

There are six non-product streams leaving this process. These are: offgas HCl which contains some Aroclor; the forecut fraction; the Porocel purge; the stillpot residue; the column vent loss; and the offgas condensate. These may be handled as follows:

The offgas HCl, 49.9 lbs. of gas containing an estimated 0.03 lbs. of Aroclor, can be handled in the same fashion as the offgas from the present Aroclor 1016 process. The gas is piped to the chlorosulfonic acid department where it is passed through a chlorosulfonic acid scrubber. The Aroclor content of the gas leaving the scrubber is down to the non-detectible level.

The forecut fraction, 33.4 lbs. of biphenyl and monochlorobiphenyl, and the Porocel purge, 4.0 lbs. of essentially monochlorobiphenyl, can be handled in two alternate fashions. They can either be piped back to biphenyl storage for subsequent chlorination to Aroclor 1142 or else they can be fed to the batch chlorinators for chlorination to produce Aroclor 1254.

The stillpot residue, 28.6 lbs. of the higher Aroclors, can be handled in the same way as the present stillpot residue from the Aroclor 1016 process. This material should be straight-takeover distilled to remove lime and $FeCl_3$ catalyst. It can then be further chlorinated to produce Aroclor 1254.

The Standard Manufacturing Process for Aroclor 1016 shows a vent loss from the column of 0.09 lbs./day when city water is flowing to the vent condenser. Based on this and vapor pressure data, we estimate a column vent loss when running on MCS 1043 of 0.4 lbs./day with the vent condenser on. This will contain some biphenyl but will consist mostly of mono- and dichlorobiphenyls.

The offgas condensate (1.6 lbs.) is composed of unreacted biphenyl and Aroclors. It can be recycled to chlorination to make Aroclor 1254.

D. R. Cova

Approved by:

J. F. Quinn
Manager
Engineering Specialties

Appendix A

Material Specifications

Monsanto
ORGANIC CHEMICALS DIVISION

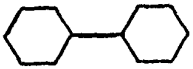
RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
82601	DIPHENYL (BIPHENYL)
PLANT WGK	**USING DEPTS.** 246
DATE EFFECTIVE 10/23/70	**SUPPLIER** Anniston Plant

APPROVALS

PRODUCTION A. E. Leisy	**RESEARCH** W. R. Richard
PROCUREMENT J. Leyerle	**QUALITY CONTROL (Chief Chemist)** R. Blowers

GENERAL DESCRIPTION



CHEMICAL FORMULA

mol wt. 154.20

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
2/6/69	2 x 16 oz. W.M. Bottles	S. Shaulter/G. Stites

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
(R) Appearance and Color	Light Yellow Cryst. Solid	10,252
(R) Crystallizing Point	68.7°C., min.	10,298
(R) H_2O	250 ppm max.	13,155
GENERAL INFORMATION		
Distillation Range:		
First Drop to	2.5°C., max.	10,299
Dry Point	Must include 255°C. within range	

MGNS 057743

(R) Routine Analysis. All others Analyzed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

FINISHED PRODUCT SPECIFICATION

PRODUCT			232
Chlorine (Snift Gas)			
PLANT	PRODUCT CODE	SITE	
WGK	81064	Internal	
DATE EFFECTIVE	SALES CODE		
11/69			

APPROVALS

PRODUCTION	RESEARCH
T. W. Dalton A. E. Leisy	W. R. Richard
SALES	QUALITY CONTROL (Chief Chemist)
	R. M. Blowers

GENERAL DESCRIPTION: Mixture

CHEMICAL FORMULA:

SPECIFICATION NO.	SUPERSEDES SPECS. OF	SAMPLES FOR ANALYSIS	ISSUED BY
	10/66	100 c.c. Gas Burette	Higgerson/Stites

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
	Typical Analyses	
Chlorine (Cl_2)	86%, By Volume	
Oxygen (O_2)	3% " "	
Carbon Dioxide (CO_2)	1.5% " "	
Carbon Monoxide (CO)	0.1% " "	
Hydrogen (H_2)	1.0% " "	
Inert Gasses (By difference)	8.4% " "	
Moisture (H_2O) @ Std. Conditions	2.4×10^{-5} lbs./ft.^{3} (Average)	

Direct pipeline transfers to using Depts.

233, 236, 237, 246

This specification is for internal use at WGK Plant only.

MONS 057744

(R) Routine Analysis. All others Analysed by request Only

Monsanto
ORGANIC CHEMICALS DIVISION
J. G. Krummrich Plant
RAW MATERIAL
SPECIFICATION

MATERIAL CODE: 33500	MATERIAL: Ferric Chloride Anhydride
PLANT: WGK	USING DEPTS.: 233
DATE EFFECTIVE: Jan. 31, 1969	SUPPLIER: McKesson & Robbins

APPROVALS

PRODUCTION: E. Leisy	RESEARCH: W. R. Richard
PURCHASING AGENT: J. Leyerle	QUALITY CONTROL (Chief Chemist): R. Blowers T. Bell

GENERAL DESCRIPTION

CHEMICAL FORMULA: $FeCl_3$

Mol wt. 162.21

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
1/1/66	1 x 8 oz. W.M. Bottle	Higgerson/Stites

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
		J.F.Q.
Appearance	Black powder or Granules	16-A
Assay	97.0%, min.	22-570
Sulfates	0.05%, max.	120-570
Ferrous Salts	Trace, max.	S-570-1
Solubility:		112-B
10:10 of Water	Complete to slightly turbid, max.	
10:10 of Formula 30 Alcohol	Complete to slightly turbid, max.	

MONS 057745

Routine Analysis. All others Analyzed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERIAL
38800	Lime, Hydrated
PLANT: W.G.K.	USING DEPTS.: 287, 750, 246
DATE EFFECTIVE: 4/7/70	SUPPLIER: Mississippi Lime Co.

APPROVALS

PRODUCTION	RESEARCH
A. E. Leisy T. W. Dalton	W. R. Richard
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
J. Leyerle	R. Blowers

GENERAL DESCRIPTION — CHEMICAL FORMULA

$Ca(OH)_2$

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
8/13/69	1 x 16 oz. W.M. Bottle	

CHARACTERISTIC	LIMITS	METHOD
Appearance	Fine white or slightly gray powder, free from lumps or gritty material.	10,188
Assay ($Ca(OH)_2$)	92.00%, min.	10,191
Carbonates as ($CaCO_3$)	3.00%, max.	10,193
Magnesium	0.50%, max.	10,192
Sulfates (as SO_3)	0.30%, max.	10,189

Supplier's certificate of analysis to be sent on day of shipment to:

Chief Chemist, Monsanto, Sauget, Illinois

MONS 057746

(R) Routine Analysis. All others Analyzed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

MATERIAL CODE	MATERIAL
48289	REGULAR POROCEL
PLANT: WGK	USING DEPTS.: 246
DATE EFFECTIVE: 8/3/70	SUPPLIER: Porocel Corp. (A Subsid. of Mineral & Chemicals Phillip Corp.)

APPROVALS

PRODUCTION	RESEARCH
J. E. Leisy	W. R. Richard
PROCUREMENT: J. Leverle	QUALITY CONTROL (Chief Chemist): R. M. Blowers

GENERAL DESCRIPTION: ACTIVATED BAUXITE

CHEMICAL FORMULA:

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
9/69	2 x 16 oz. W. M. Bottles	Higgerson/Stites

RESTRICTED INFORMATION

CHARACTERISTIC	LIMITS	METHOD
Appearance	Free from lumps and foreign material	
Mesh Test:		10,692
Less than 60 mesh:	3%, max.	
20 to 60 mesh inclusive:	67%, min.	
Greater than 20 mesh:	30%, max.	
Water	1%, max.	10,100
Volatile Matter (at 1000°C. to constant weight)	2%, max.	

MONS 057747

(1) Routine Analysis. All others Analyzed by request only.

TENTATIVE SPECIFICATIONS FOR AROCLOR 1129

Biphenyl Content	< 5.0%
2,4'-Dichlorobiphenyl (includes 2,3-Dichlorobiphenyl) Content	>17.0%
4,4'-Dichlorobiphenyl Content	< 6.3%
Specific Gravity at 65°C =	1.215

TENTATIVE SPECIFICATIONS FOR MCS 1043

		Method No.
Biphenyl Content	< 0.1%	See GLC Method
2,4'-Dichlorobiphenyl (includes 2,3-Dichlorobiphenyl) Content	>20.0%	" " "
4,4'-Dichlorobiphenyl Content	< 2.0%	" " "
Higher Boiling Homologs	< 0.02%	T02302
Resistivity	>5000 x 10^9 ohm-cm	See procedure in appendix
Power Factor	< 0.1%	See procedure in appendix

APPENDIX B

Analytical Procedures

Physical Chemistry
Method No. 69-13
Job No. 1630022

GAS CHROMATOGRAPHIC DETERMINATION OF THE 4,4'-DICHLOROBIPHENYL CONTENT OF ISOMERIZED AROCLOR 1232

PURPOSE OF ANALYSIS

To measure the 4,4'-isomer content of isomerized Aroclor 1232.

CHROMATOGRAPHIC CONDITIONS

Instrument: Perkin-Elmer Model 800

Type Detector: Flame Ionization

Column: 0.02" X 50' Carbowax 20M S.C.O.T. (support coated open tubular) capillary

		Flow Rates	
Column Temperature:	215°C		
Detector Block Temperature:	250°C	Helium:	15 ml/min.
Injection Port Temperature:	300°C	Auxiliary Gas:	15 ml/min.
Sample Volume Injected:	0.001 ml	Hydrogen:	30 ml/min.
Split 30-to-1		Air:	400 ml/min.

RETENTION TIMES (UNCORRECTED) OF COMPONENTS SEPARATED (CHROMATOGRAM ATTACHED)

Biphenyl (3.4'); Orthochlorobiphenyl (4.9'); Metachlorobiphenyl (6.6'); Parachlorobiphenyl (7.0'); 2,6'-Dichlorobiphenyl (7.5'); 2,2'-Dichlorobiphenyl (8.0'); 2,4'- and 2,5'-Dichlorobiphenyl (8.2'); 2,3'-Dichlorobiphenyl (9.6'); 2,4'- and 2,3'-Dichlorobiphenyl (10.3'); Unknown A (12.3'); 2,5,2'-Trichlorobiphenyl (13.0'); 3,3'- and 3,4'-Dichlorobiphenyl (13.6'); 3,4'-Dichlorobiphenyl (14.4'); 4,4'-Dichlorobiphenyl (15.6'); 2,3,2'-Trichlorobiphenyl (16.4'); 2,5,4'-Trichlorobiphenyl (17.7'); 3,4,2'-Trichlorobiphenyl (19.6'); Unknown B (20.5'); Unknown C (22.1'); 3,4,4'-Trichlorobiphenyl (31.6').

CALCULATION TECHNIQUE

Normalization.

PRECISION

No determination of precision was made. The variability of a single GC analysis at the 95% confidence limits is usually ±1 to 2% relative.

db

Monsanto Company
Organic Chemicals Division
Applied Sciences Section
St. Louis, Missouri

2/20/69 - G. M. Gasser, E.M. Emery

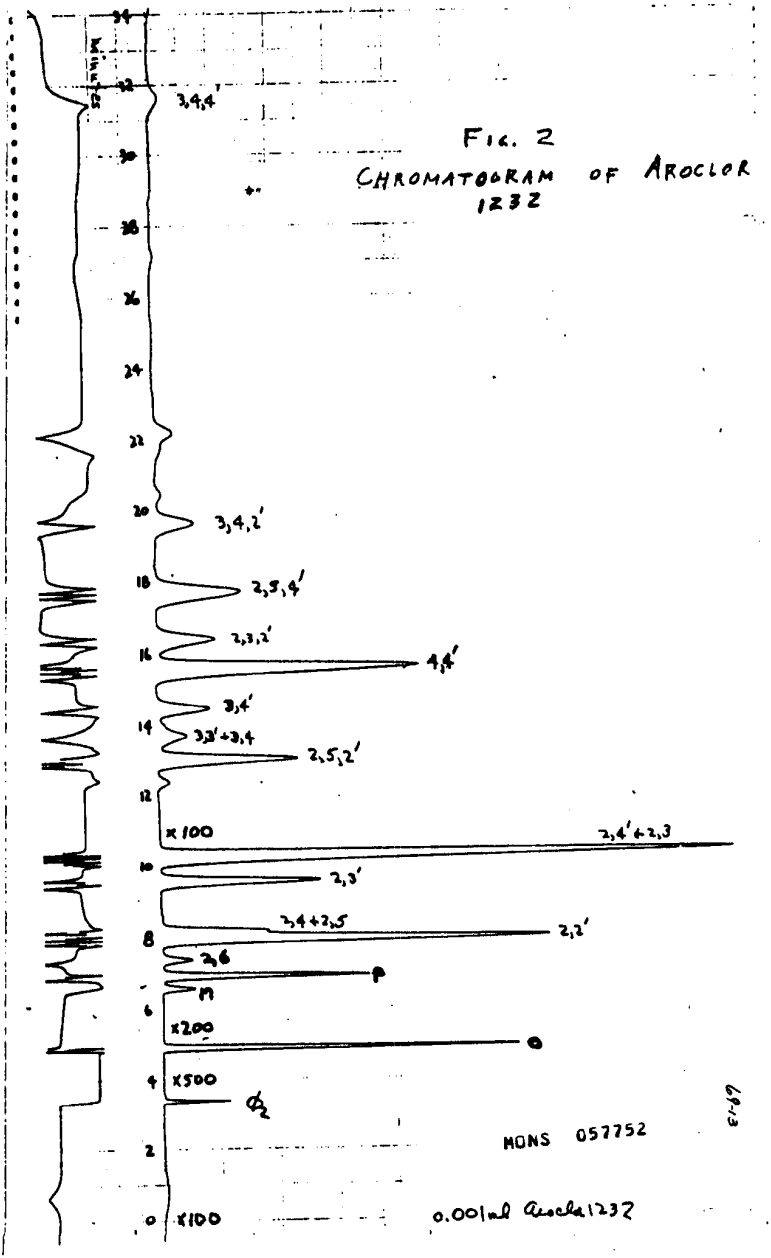


FIG. 2
CHROMATOGRAM OF AROCLOR 1232

MONS 057752

6-13

PROCEDURE FOR MEASURING DISSIPATION FACTOR OR POWER FACTOR, DIELECTRIC CONSTANT AND RESISTIVITY OF ASKARELS

INTRODUCTION

This procedure covers the measurement of dissipation factor, dielectric constant or permittivity and resistivity of electrical insulating fluids. The dissipation factor and the power factor of a fluid differ by less than one percent of the numerical value when the dissipation factor is less than 0.14. Since good insulating fluids have dissipation factors much below this, their dissipation factors and power factors can be considered equal for ordinary purposes. In some cases, power factor is given in percent. Care must be used in comparing values to be sure that all are given as fractions or in percent. Calculations given in this procedure give the fractional value of the dissipation factor. To convert these values to percent, multiply by 100.

Precautions: In the early development of this procedure Aroclor 1242 was found to be light sensitive. Light from sources such as fluorescent lamps, mercury vapor lamps, and incandescent light bulbs caused a marked increase in dissipation factor and a decrease in resistivity. For this reason all dissipation factor and resistivity measurements should be made in subdued light.

When measuring fluids having low dissipation factor values such as those found in Askarel fluids, it is extremely important that the utmost care be taken to prevent contamination of the sample. Minute quantities of contamination introduced into the sample either from the test cell, glassware, or in other ways will cause extreme differences in dissipation factor values. As little as 0.1 part per billion of some impurities can be detected. Therefore, the test

cells must be handled only with the cell holders or tongs. No part of the test cell or glassware which might contact the sample should be touched with hands or any support. Strict adherence to the cleaning and sample preparation sections of the procedure is a must. Dissipation factor measurements have been made using test cells that were cleaned and stored for 16 hours in the drying oven with satisfactory results; however, longer storage periods give increased dissipation factor values.

The cells are ordinarily cleaned without disassembling. However, if coatings or impurities remain after the standard cleaning procedure the cells can be taken apart and cleaned with soap and water on a soft cloth or bristle brush. This should not be done unless several passes through the standard cleaning procedure fail to get the cell clean enough.

To take the cell apart for cleaning, first unscrew the 874 connector shell and pin from the top. Then unscrew the outer electrode from the body of the cell. Finally, unscrew the knurled nut from the center electrode and push it gently out of the body. No attempt to further disassemble the cell should be made. To reassemble, reverse the above procedure.

After cleaning the cell parts with soap and water, it should be reassembled, rinsed with distilled water and reagent grade acetone, then put through the standard cleaning procedure until it gives the correct value when used to measure a known good sample.

Caution should be exercised when making resistance measurements on the Type 1644A Megohm Bridge. Never touch the cell or leads from the test cell to the bridge when in the charge zero or measure position. 500 volts

is applied to the cell in either position. Always return the switch to the discharge position after completing the resistance measurement before attempting to remove the leads from the bridge to the test cell. The red light on the upper right corner of the panel is on when the switch is in either position. However, it is important to look at the switch itself; the pilot light could be burned out.

APPARATUS

1. Electro Scientific Industries Capacitance Measuring System Model 701.
2. General Radio Type 1644A Megohm Bridge.
3. 110-24 Volt Step Down Transformer, Selenuim Rectifier type RT201, Chicago Standard Transformer Corp., Chicago, Ill., with plug in adaptor unit for 60 cycle measurements.
4. Special Plug in Connector for measurements above 60 cycles
5. Two General Radio Type 874-Q9 or 777-Q3 adaptors.
6. General Radio Decade Resistor Type 1433-L or box made using one General Radio Type 510D and one 510E decade resistor in series in a suitable metal box.
7. General Radio Type 274 NQS Patch Cord.
8. Coaxial Cable lead (General Radio Type 874 Patch Cord Cat. No. R22A).
9. Patch cord with banana plug terminals such as H. H. Smith Type 1863-36-102, Allied Radio Stock No. 47D1904.
10. Balsbaugh Test Cells (Stainless Steel) Type 350G modified according to Monsanto specifications.

11. Constant Temperature Bath, a 12" x 12" Pyrex jar, insulated.

    a. Constant Temperature of 100°C. is maintained with a Fisher Proportional Controller using a thermister probe as a temperature sensing device.

    b. A 125 watt Cenco knife blade heater is used for intermittent heating controlled by the Fisher Proportional Controller.

    c. A 450 watt Cenco knife blade heater is used as a continuous heater controlled by a variac to supply enough heat so that the intermittent heater is on about half the time.

    d. A Bodino stirring motor with attached stainless steel propeller is used to maintain good agitation of bath liquid. The propeller is surrounded by a draft tube. It should draw the bath fluid up through the draft tube.

    e. Glycerine is used for the bath medium with the liquid level maintained to within 3/4 inch from the top edge of the bath cover.

    f. Standardized Centigrade thermometer for temperature read out (Princo Q-7, range 75°-110°C.)

    g. Bath cover constructed from Extren 500 manufactured by J. T. Ryerson & Son, Inc., 2558 West 16th St., Chicago, Ill. 60608.

12. Drying Oven (Fisher Isotemp Junior Model 202)

13. Xylene Vapor Bath containing calcium oxide lumps (Test Cell Cleaning).

14. Boiling 29% NH_4OH Bath (Test Cell Cleaning). A 1.5 liter beaker.

15. Distilled H_2O steam bath (Test Cell Cleaning).

16. Acetone Vapor Bath (Test Cell Cleaning).

17. Xylene Vapor Bath (Glassware Cleaning).

18. Distilled H_2O Steamer (Glassware Cleaning).

19. Sample Vessel (Glass).

20. Teflon Lid for Sample Vessel.

21. Supporting Clamp (Sample Vessel).

22. Special Wrench for removing center pin connection from test cell.

23. 50 ml. Pyrex Pipette (Taper Tip cut off to allow faster sample delivery).

24. Fisher Thermix Hot Plate Cat. No. 11-493 (Acetone Vapor Bath).

25. Teflon Covered Stirring Bar (For Acetone Vapor Bath).

26. Laboratory Tongs (Fisher Cat. No. 15-202).

Drawings of the items of apparatus which cannot be obtained from laboratory supply companies are attached. Numbers on drawings correspond to numbers on apparatus list.

ELECTRICAL CONNECTIONS

1. Electro Scientific Industries Capacitance Measuring System, Model 701.

   a. With the power switch off, plug the power cord of the Model 701 into a standard 110 volt outlet. This will supply power for the bridge null detector and internal power supply.

b. Remove the plug-in shield covering the generator output and bridge in put terminals and the links which connect them. To make measurements at 60 Hz, connect the 12 volt terminals of the 110-24 volt transformer to the terminals marked GEN 1 and 2 on the bridge through a switch. A GE Type 47 pilot lamp connected in series with a 100 ohm resistance across the 24 volt leads serves to indicate when the bridge power is on. The switch and pilot lamp with its resistor are mounted in a small metal shield box with banana plug connectors in its back so that the 60 Hz 24 volt source may be plugged into or disconnected from the generator terminals of the bridge quickly and easily. The 110 volt terminals of the transformer are connected to a 110 volt outlet with a suitable cord and plug.

c. To make measurements at other frequencies than 60 Hz, connect the generator output terminals marked GEN OUT to the bridge GEN 1 and 2 terminals with the links provided or more conveniently with a connector made of a 1/4" x 1-1/4" x 1-3/4" Lucite block with 4 properly spaced banana plugs mounted in it and wired to connect the proper terminals.

d. To make dissipation factor measurements at higher values than provided by the Dissipation Factor Dial of the bridge, make the following connections: Remove the link connecting the EXT D ADJ terminals. Plug one end of a General Radio

Type 274 NQS double plug patch cord into these terminals and the other into the terminals of the decade resistor.

e. The Balsbaugh cells are connected to the unknown terminals of the bridge as follows: Plug the center connector of a General Radio 874-Q9 or 777-Q3 Adaptor into the Unknown No. 1 terminal so that its shield terminal connects to the GRD terminal. Use the General Radio Type 874 Patch Cord to connect between the adaptor and the Type 874 connector on top of the Balsbaugh cell. Plug one end of the banana plug patch cord into the Unknown No. 2 terminal of the bridge and the other end into the banana plug jack at the top of the cell.

2. General Radio Type 1644A Megohm Bridge

a. With the bridge power switch off, plug the power cord into a 110 volt outlet.

b. Connect the Balsbaugh cell to the bridge as follows. Plug the center connector of a General Radio 874-Q9 or 777-Q3 Adaptor into the bridge terminal marked + Unknown. Connect the shield terminal of the adaptor to the bridge terminal marked Guard. Connect the Guard and Gnd terminals of the bridge with one of the links supplied with the bridge. Then use the Type 874 patch cord to connect the 874 adaptor on the bridge and the Type 874 connector at the top of the cell. Use the banana plug patch cord to connect the Unknown - terminal of the bridge and the banana plug jack at the top of the cell.

MATERIALS

1. Glycerin for constant temperature bath.
2. Xylene, Reagent Grade.
3. 29% Ammonium Hydroxide, Reagent Grade.
4. Distilled H_2O.
5. Acetone, Reagent Grade.
6. Benzene (Non-Reagent Grade).
7. Acetone (Non-Reagent Grade).
8. Calcium Oxide Lump Form (Fisher Cat. No. C-116).
9. Boiling Chips (Used in cleaning baths).

PROCEDURE

A. Clean Test Cell

1. Using tongs place test cell into cell holder.
2. Clean in xylene vapor bath for 15 mins.
3. Clean in boiling 29% NH_4OH for 10 mins.
4. Clean in distilled H_2O steam bath for 30 mins.
5. Clean in acetone vapor bath for 15 mins.
6. Dry in $100^{\circ}C$-$110^{\circ}C$ oven for 30 mins.

Note: Cells must be handled only with cell holders or tongs. The cleaning baths require cleaning once per week.

B. Cleaning Glassware

1. Clean Pipette.
   a. Use tongs to hold pipette.
   b. Benzene wash.
   c. Acetone wash.

d. Distilled H_2O wash.

e. Reagent grade acetone wash.

f. Draw clean air through pipette.

g. Dry by flaming with bunsen burner.

h. Draw clean air through pipette.

Note: No part of the pipette which might contact sample should be touched with hands or any support.

2. Clean Sample Vessel

a. Pour sample into solvent can.

b. Benzene wash.

c. Acetone wash.

d. Rinse off glycerin with water.

e. Acetone wash.

f. Clean in xylene vapor bath for 15 mins.

g. Fill 3/4 full with 29% NH_4OH boil for 10 mins.

h. Steam for 30 mins.

i. Dry at 100°-110°C for 30 mins.

j. Draw clean air through vessel before using.

C. Sample Preparation

1. Remove sample vessel from drying oven. Use tongs.

2. Draw clean air through sample vessel.

3. Pipette sample into sample vessel by means of a suction bulb.

4. Remove test cell from drying oven and insert coaxial cable, connector shell and pin.

5. Place test cell in sample (check to make sure that the sample level is just above the bottom of the holes on the unguarded electrode).

6. Fasten sample vessel supporting clamp in such a position to allow 1/8 inch of the vessel to extend above the top edge of the clamp. This insures proper immersion depth in the bath.

7. Connect leads to the cell and put on Teflon lid.

8. Place sample vessel in the 100°C constant temperature bath and record entrance time.

Note: The required amount of sample is just enough to insure sample flowing through the holes on the unguarded electrode (outside). This amount is approx. 50 ml.

D. General Operating Procedure for Electro Scientific Industries Model 861A AC Generator Detector

1. 60 cycle Measurements

   a. Plug in the step-down transformer adaptor into Gen. position 1 & 2 on capcitance bridge.

   b. Turn the AC line switch on, allow 15 min. warm up.

   c. Rotate the output Power control fully counterclockwise.

   d. Set Frequency Control for 60 cycles.

   e. Set Range Control at the 1X position.

   f. Set the Selectivity control to the Sharp position.

   g. Turn the Log-Linear Switch to the Log position.

h. Turn outer Sensitivity control know to such a position that a deflection on the meter can be seen. (Not more than 50% of scale)

i. Rotate the Fine Tuning control to the position giving the maximum meter deflection. Detector is now properly tuned for 60 cycle measurements.

2. For Frequencies Above 60 Cycles

a. Plug in special adaptor in generator out terminals 1 & 2.

b. Turn the AC line switch on.

c. Set the output Impedence switch to 100-ohm position.

d. Rotate the output Power control full clockwise.

e. Set Frequency Control for desired frequency.

f. Set Range Switch to match frequency.

g. Set Selectivity control to Sharp position.

h. Turn the Log-Linear Switch to the Log position.

i. Turn outer Sensitivity Control knob to such a position that a deflection on the meter can be seen. (Not more than 50% of scale)

j. Rotate Fine Tuning control to the position giving the maximum meter deflection. Detector is now properly tuned.

E. Make Dissipation Factor Measurements Using ESI Type 701 Capacitance Measuring System

1. Turn on bridge null detector (allow 15 mins. warm up).

2. Set Frequency control to desired frequency using proper plug in unit, tune detector for maximum sensitivity (See General Operating Procedure for Model 861A AC Generator Detector, Section E).

3. Set Capacitance range switch in the Picofarads C range.
4. Set Dissipation factor range switch to the 10^{-3} position.
5. Connect leads from the test cell to the bridge.
6. Apply bridge power.
7. Adjust bridge sensitivity (inner knob) until needle reads 40 on the meter scale.
8. Balance the bridge by adjusting the capacitance and then the dissipation factor knobs alternately until the best null is achieved at 10^{2} sensitivity range.
9. A 1K and 10K step external decade resistance box is used to extend the bridge dissipation factor scale readings. Each 1000 ohm step on the decade box is equal to 100 divisions on the disspiation factor scale. It then becomes necessary to divide the total reading of the decade box by 10. This value is added to the Dissipation Factor scale reading.
10. Record the capacitance and dissipation factor readings 30 mins. after placing sample in the constant temperature bath.

Formula for calculating dissipation factor:

$(S + R) \times A \times F$ = Dissipation Factor

Where S = Dissipation factor scale reading

R = External Decade box reading in ohms divided by 10

A = Dissipation factor range

F = Frequency in KHz (kilocycles)

Example for 60 cycle measurements:

$S = 41$, $A = 10^{-3}$, $F = 6 \times 10^{-2}$, R = not needed to balance bridge

From the formula $S \times A \times F$ = Dissipation Factor

$$(41 \times 10^{-3})\ 6 \times 10^{-2} =$$

$$41 \times 6 \times 10^{-5} = .00246$$

Example when the External Decade Box is needed to extend the Dissipation Factor Scale readings

$S = 41$, $A = 10^{-3}$, $F = 6 \times 10^{-2}$, $R = 250$

From the formula $(S + R) \times A \times F$ = Dissipation Factor

$$(41 + 250) \times 10^{-3} \times 6 \times 10^{-2} =$$

$$291 \times 6 \times 10^{-5} = .01746$$

Example For 100 cycle measurements

$S = 41$, $A = 10^{-3}$, $F = 1 \times 10^{-1}$, R = not needed

From the formula $S \times A \times F$ = Dissipation Factor

$$41 \times 10^{-3} \times 1 \times 10^{-1} =$$

$$41 \times 10^{-4} = 0.0041$$

Example for 1000 cycle measurements

$S = 41$, $A = 10^{-3}$, $F = 1$, R = not needed to balance bridge

From the formula $S \times A \times F$ = Dissipation Factor

$$41 \times 10^{-3} \times 1 = .041$$

Dielectric constant calculations can be made by using the following formula.

Formula for calculating Dielectric Constants.

$\frac{C_s}{C_a}$ = Dielectric Constant

C_s = Measured capcitance of sample

C_a = Air Capacitance of the test cell in air at room temperature

F. Make Resistivity Measurements Using Type 1644A Megohm Bridge

1. Turn on bridge to discharge position, allow 10. min. warm up.
2. Connect leads from test cell to Megohm Bridge.
3. Zero resistance bridge while charging zero at 500 volts for 30 seconds.
4. Measure for 30 seconds.
5. Read 1 minute after applying bridge voltage.
6. Return switch to discharge position.
7. Record resistance reading.

8. Resistivity to be measured immediately after 30 minute dissipation factor measurement.

Note: Never touch cell or leads when bridge is in charge or measure position. After completing resistance measurement return switch to discharge position before removing leads.

Formula for Calculating Resistivity:

$R \times M \times k$ = Resistivity

Where R = Resistance reading at 500 volts

k = test cell constant (The cell constant is obtained by measuring the air capacitance of the cell at room temperature and multiplying this value by 11.3)

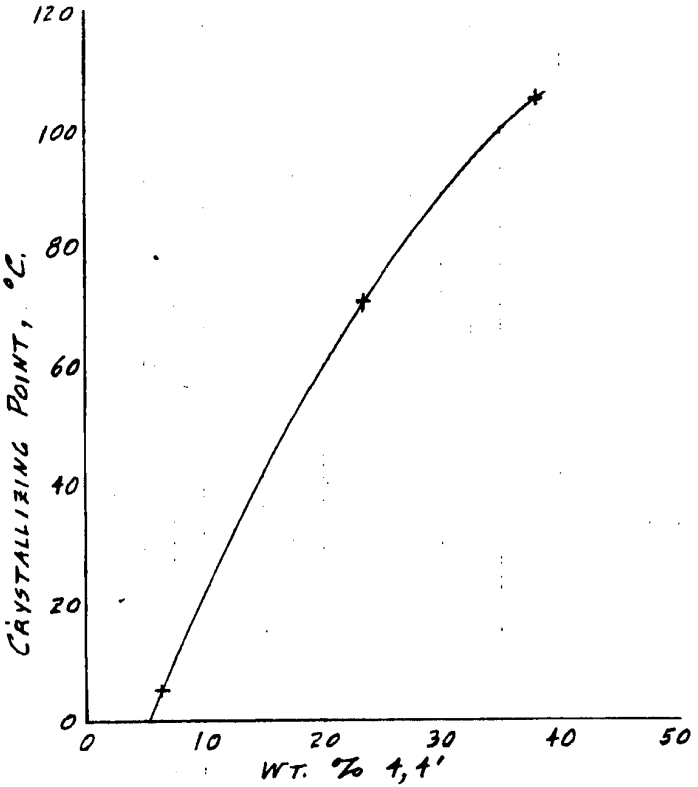
M = Resistance multiplier

Appendix C

Stream Data

FIG. 3

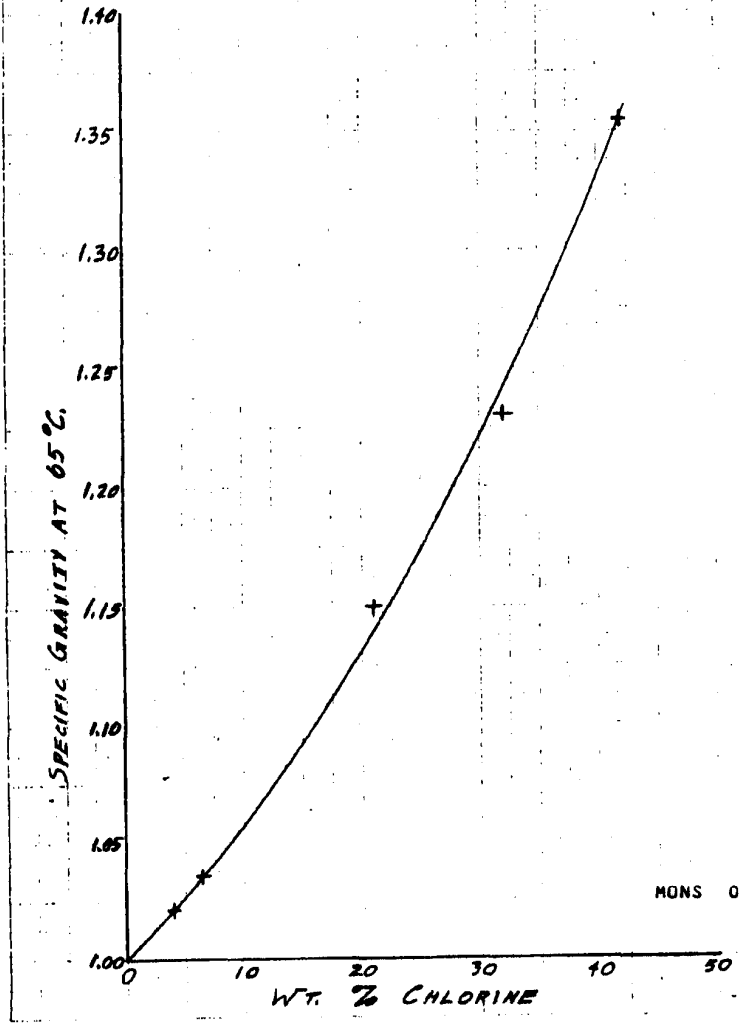
CRYSTALLIZING POINT OF MCS1043 DISTILLATE VERSUS 4,4' CONTENT



MONS 057769

FIG. 4

SPECIFIC GRAVITY VERSUS CHLORINE CONTENT OF AROCLOR



MONS 057770