MONSANTO COMPANY
W. G. Krummrich Plant

May 18, 1967

AROCLOR CORPORATE MEETING

cc: Messrs.
R. E. Howard - G.O.
J. R. McClain
F. Macdonald - Newport
W. B. Papageorge - Anniston
A.C.W. Pemberton - Newport
W. J. Wilson

COMPANY CONFIDENTIAL

Messrs.

D. C. Armstrong
G. W. Ashworth - S. 2nd
A. Arpino - Brussels
P. G. Benignus - G.O.
G. R. Buchanan - G.O.
V. Brawley
J. G. Bryant - Anniston
H. C. Carder - G.O.
C. R. Coleman - Newport
P. D. Collins - G.O.
J. Cresce - G.O.
H. J. Day - G.O.
L. C. Fuhrmeister - Anniston
H. O. Hehner - G.O.
P. E. Heisler
H. J. Horner
H. L. Hubbard - S. 2nd
N. T. Johnson - G.O.
R. M. McCutchan
O. A. Meyer
G. W. Miller - Anniston
J. W. Molloy
R. H. Munch - S. 2nd
G.J.S. Perry - Newport
H. F. Ray
W. R. Richard - G.O.
V. W. Seager - S. 2nd
R. A. Steenrod - G.O.
R. P. Stevens - G.O.
J. D. Sullivan - G.O.
M. T. White, Jr. - G.O.
C. M. Williams - G.O.
H. L. Williams - Anniston
D. Wood - Brussels
R. S. Yates - G.O.

Attached are detailed minutes taken at the Aroclor Quality Measurement session on May 10, 1967. Minutes were recorded by Vic Seager and George Ashworth.

J W Molloy
RMM

Jack W. Molloy
Technical Services
Department

/gg

DSW 200761

AROCLOR

I. Chemical Methods of Testing Aroclors

Mr. Coleman stated that chloride titration is now being used in England in place of the Lyndal beam.

The ASTM method for acid number was designed for transformer oils and is not applicble to Aroclors. The method for acid number in use at Anniston as a control of quality has given satisfactory results in our laboratory as well as those of the customer. The same procedure gives equally good results in England. The method should be incorporated as an ASTM standard method for industry wide acceptance.

Both Krummrich and Anniston personnel stated there was a great need for a better thermal chemical chloride (TCC) test especially if meaningful results are to be obtained in determining the effect of process changes on quality.

Mr. Benignus stated that the TCC test was not designed for the low levels of chloride present in current Aroclor. The present test carried out with care is entirely satisfactory to our customers as a limit test. Values of 0.2 to 0.3 ppm are satisfactory. For a better evaluation of the effect of process variables on quality a more sensitive and reproducible test would be desirable. This would be for internal use only. Some modification of the Westinghouse test would give more reproducible results but, of course, would not include a thermal evaluation. Present indications are that such a test can be used as a measure of Aroclor quality at the 0.1 ppm level.

Mr. Ashowrth presented work he had done on the pyridine-conductivity method for the determination of hydrolyzable chlorides. This method was developed by Dr. R. Wilputte of Laborelec and was submitted to us through E. H. Reynolds in England. The method is designed to measure values from 2-100 ppm hydrolyzable chloride in Aroclor. The extension of the method down to 0.1 ppm presented serious difficulties in sensitivity and reproducibility. These are outlined as follows.

1. The conductivity of pyridine is very sensitive to moisture pickup.

2. The conductivity of pyridine increases at a decreasing rate up to two hours but a constant value is never obtained. This would represent a high blank correction.

3. At least one hour refluxing with Aroclor is necessary.

4. The sensitivity of the method is only one-third that reported by Reynolds. Additional work will be done to resolve this point.

5. The equipment necessary to measure the low conductivity with 0.1% accuracy is rather expensive.

DSW 200762

Dr. Munch stated that sufficient work had been done on the method to show thatit was not suited for use at the 0.1 ppm level.

Mr. Ashworth is to write up the work in a form suitable for presentation at a meeting of CIGRE study committee No. 1 in London on October 13. This information would show that such a method is not suitable for adoption as an ASTM method.

Dr. Wilputte of Laborelec also expressed serious objections to the Westinghouse procedure because it involves a two phase system. He proposed the use of ethyl glycol (and methanol) as a solvent to give a one phase system. (The "ethyl glycol" in the method should have read ethylene glycol monoethyl ether or cellosolve). The use of this solvent will be investigated. Dr. Munch requested Mr. Coleman to find out if Mr. Reynolds had any specific reason for using cellosolve as a solvent in his one phase system.

Mr. Ashworth reported that a small amount of acetone can be used to give a one phase system. Appreciably lower values are obtained for hydrolysis in the dark compared with illumination with fluorescent light. Different samples showed wide variation in the difference between light and dark. These differences may detect the presence of certain types of compounds present as impurities. Additional work will be done along these lines.

Mr. Armstrong suggested it would be desirable to prepare representative types of chlorinated products and test their effect on power factor and the hydrolyzable chloride methods. The compounds suggested were chlorobenzene, chlorobiphenyl, benzene hexachloride, phenylbenzyl chloride, a chlorostyrene, α, β dichloro ethyl benzene.

II. Future Quality Measurement and Testing

A. Electrical Method of Testing Aroclors

Dr. R. H. Munch stated that the Organic Research facilities have the following capacitance bridges for electrical testing on Aroclor: General Radio Type 716c bridge, a modified General Radio Type 1615A bridge, and an ESI Model 701 bridge. The modified General Radio Type 1615A bridge is on loan from General Radio for evaluation purposes.

Mr. C. R. Coleman (MCL) stated that two groups are now active in the area of international standardization of test methods. The IEC (International Electrochemical Commission) Technical Committee 10 and the CIGRE Study Committee No. 1 were set up for mineral oils, but have now extended themselves to cover synthetic liquids (e.g. askarels) and gases. The CIGRE committee discusses test methods on a somewhat less formal basis than the IEC committee.

Mr. Coleman summarized their recent activities in the area of electrical test methods for Aroclors. The MCL cell developed about 10 years ago for resistivity measurements is not entirely satisfactory for power factor measurements. Quite often readings can be reproduced within 10 per cent, but occasionally a "wild" result is obtained. On refilling the cell with fresh sample, the power factor isagain in the expected range. The variability appears to be due to random contamination in filling the cell rather than due to the cell or bridge. MCL has been testing a different type of cell for power factor measurements of askarels. This cell was developed by M. Coquillion of Prodelec. Details of his cell and method were announced at a CIGRE Study Committee No. 1 meeting on June, 1966. Mr. Coleman reported that initially they copied Coquillion's cell as closely as possible, but subsequently arranged an exchange of cells with Coquillion. While somewhat improved results can be obtained with Coquillion's cell, difficulty has still been encountered with the occasional "wild" power factor measurement. Mr. Coleman stated that he is corresponding with Coquillion on this problem. Mr. Coleman emphasized that it would be desirable to have a test method and cell that is portable, so that tests could be carried out before the customer's eyes. MCL recently acquired a new bridge, which will operate at 50 and 1000 volts.

Dr. Munch stated that he is looking into what sort of things influence these electrical measurements. A better understanding of the factors influencing the measurements is needed. As for as test voltage, Dr. Munch feels that there is less an effect of leaving the bridge voltage on, if a low voltage is used. He would prefer lower voltages (10 or 20 v.) as a standard for power factor measurements; lower voltage for resistivity measurements would be desirable also. Dr. Munch stated that use of the lower voltages involves two considerations: the technology involved and customer acceptance. Mr. P. C. Benignus stated that both high and low voltages are used in the trade--it depends on what they have on hand. Capacitors will still have to be measured at operating voltages, but Mr. Benignus thought that there would not be a great deal of prejudice against using low voltages for the liquid dielectric. Mr. Coleman stated that Coquillion uses a General Radio Type 716c bridge and favors low voltage. Dr. W. R. Richards emphasized that we will have to prove the low-voltage method is satisfactory. The design engineers will want the values at end-use voltages, as we will have to show what effect the voltage has on the measurements.

Dr. Munch outlined what he thought the recommendations of this meeting should be:

1. Get better electrical test data more easily.

2. Power factor measurements should be made with a low-voltage type transformer ratio arm bridge operating at power line frequency. Each concerned plant installation should procure a bridge of this type. These are available at moderate price.

3. Three-terminal measurements are preferred since the effects of leads and body and other stray capacitances on the bridge zero are eliminated. We are not in a position to recommend an exact type of three terminal cells at this time. The target date for a cell recommendation is October 1, 1967.

4. Measurements should be made at 100°C with enough measurements at room temperature (25°C) to correlate the 100°C and 25°C data. The 25°C data is of interest in the transofrmer industry.

5. DC resistivity measurements should probably be made with a three-terminal cell because of the resistivity range we are interested in, especially if room temperature measurements are needed.

6. The test procedure should be rapid and convenient.

Mr. Coleman emphasized the importance of ease and rapidity, especially with respect to customer tests. Dr. Munch stated that we are faced with conflicting requirements--a portable device and a precision measurement. Our efforts must first be directed toward learning how to make these measurements accurately in the laboratory.

Mr. Coleman stressed the importance of a method for sample handling and cell cleaning. The present method is inadequate for the MCL cell. The first measurement is always higher than those obtained with successive refills. Mr. Coleman illustrated this effect with the following power factor data obtained on successive refills: 0.79, 0.71, 0.65, 1.01, 0.64, 0.65, and 1.00. Two of the data points illustrate the problem of reproducibility, ie. the high results obtained occasionally.

Mr. Coleman stated that MCL used the ASTM criteria for reproducibility. The value is acceptable of two successive readings within 10 per cent of the higher reading is obtained. Dr. Munch stated that it may be necessary to modify our 10 per cent precision goal established last year.

Mr. Benignus expressed the opinion that the trade would probably be receptive to more precise and convenient test methods. The standard General Electric cell has a number of drawbacks Westinghouse objects to the glass spacer in the cell, preferring Teflon. The major stumbling block with the cell is reproducibility.

Mr. J. Miller and Mr. J. G. Bryant (Anniston) reported that they had encountered some difficulty with black specks formation on their cell electrodes. This was more pronounced in old cells. It was necessary to scrub the electrode with abrasive material to return them to their original condition. This may be more pronounced with transformer blends than with straight Aroclors. Dr. Munch states that the black specks

were probably due to pinholes in the nickel plating of their brass electrodes. He stated that we should get away from plated electrodes and that part of the cell recommendation will be the use of a single-metal electrode system, probably 316 stainless steel.

Mr. Coleman stated that they had no experience with any black specks. Their normal cleaning procedure involves a hot-water detergent wash using a test-tube type brush. With time there is some darkening of the cell above the air-Aroclor interface, but it doesn't seem to affect the results. This is removed periodically by polishing with a metal abrasive.

Mr. Benignus stated he felt the introduction of a Monsanto test cell to the trade would be of value from a prestige point-of-view.

Mr. Bryant reported on the problems of sampling at Anniston. It is difficult to get the personnel to employ a standard sampling technique. Frequently the lines are not flushed well enough or the sample bottles are contaminated. Handling degradation is a major problem. Cross-contamination occurs frequently because of plant design weaknesses. To get consistent quality, it will be necessary to isolate the Aroclor lines. Mr. Bryant stated that there is a TSD program in this regard.

Mr. Coleman reported on their drum sampling procedure. The Aroclor is withdrawn with a pipette and transferred to a 16 ounce amber glass bottle with an aluminum-lined cap. The bottle is then protected from light.

Dr. Munch stressed the importance of using clean bottles. Mr. Bryant raised the question whether flushing of the steel drum with deionized water before final assembly would be helpful.

Mr. Bryant reported on the installation of an in-line cell at Anniston. The cell uses a Viton A O-ring connector. Dr. Munch and Mr. Coleman reported that both Viton A and silicone O-rings are a possible source of contamination. Dr. Munch stated that agreement between in-line and laboratory measurements is probably up to chance. It is not possible to know what the condition of an in-line cell is.