CC: Messrs.
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J.F. Reeves (2)
A.M. Ellenburg
Jack Clegorn
R.P. Loewen
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PHOSPHATE RESEARCH DEPARTMENT

MINUTES OF THE MEETING ON LIQUID DIELECTRICS

Anniston, Alabama
January 24, 1947

Present: Mr. F. M. Clark of General Electric Company
Mr. J. F. Reeves
Mr. A. M. Ellenburg
Mr. Jack Clegorn
Mr. R. P. Loewen
Dr. R. L. Jenkins
Dr. E. E. Hardy
Mr. F. A. Barker

Purpose of the Meeting: To discuss Aroclors and other matters of mutual interest to General Electric and the Phosphate Division of Monsanto Chemical Company.

Recently General Electric Company had made a complaint to our Sales Department about the disagreement of analyses on Aroclor in our plant and their laboratory. These differences, though not serious enough to throw the Aroclor out of specifications, tend to disturb the analysts. Explanations for some of these disagreements and other fine points of the analyses of Aroclors were discussed in some detail.

The viscosity determinations were first examined. General Electric obtains about 1-1/2 Saybolt seconds more on viscosity than the Anniston plant. This difference has been discussed before and is in the specifications. Mr. Clark said this was alright if the results were consistently running similar to each other.

Concerning pour point, there had been some criticism. In particular, Mr. Raab, the man in charge of the analysis at General Electric, had felt that pour points were running over specification. On checking the pour points by five different people at Anniston, 10°C. was obtained. General Electric got 13°C. there. The test allows a 5°C. variation in the same laboratory and 10°F. deviation in different laboratories, according to the ASTM method. This easily explains the difference of 3°C. obtained by Monsanto and General Electric.

The pour point could be checked by running the dielectric constant at the low temperatures in the vicinity of the pour point. The maximum di-

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electric constant of the Aroclors occur at the pour point. Mr. Clark stated that curves showing the dielectric constant at varying temperatures down to about -5°C., determined by the Bell Laboratories, could be found in the literature.

Mr. Clark inquired whether the variations in gravity corresponded to the variations in pour points. In particularly, he was interested to know whether a high gravity meant also a high pour point. Mr. Loewen pointed out that on rechecking the gravity on the samples in question they were found to be O. K.

One possibility where differences might arise is the question whether ice or liquid air is used in the pour point equipment. General Electric normally uses liquid air while ice is normally used at Anniston. This may give rise to a different cooling lag in the equipment which may be responsible for the discrepency in the results between Monsanto and General Electric laboratories. Mr. Clark promised that he would check into this possibly satisfactory explanation of differences.

In general, Mr. Clark said that he did not think that any of these discrepencies in the analyses between the Monsanto and General Electric testing laboratories were of any serious nature.

He considered two analyses particularly critical. These were the corrosion test and the power factor stability. It was pointed out that there is a difference in specification between Monsanto and General Electric as far as the corrosion test is concerned. The General Electric specification reads .1 ppm. free chloride and the Monsanto specification reads .15. It is believed that this is due to a change in methods which gives a maximum sensitivity in the test of .1 while we report at Monsanto a higher sensitivity.

A significant difference is obtained in the determination of resistivity between the General Electric and the Anniston laboratories. General Electric uses 500 volts in their testing laboratory and 2,000 volts in the factory, while a 90 volt potential is used at Anniston. This is the possible explanation of the discrepancy in specification which for General Electric reads 500 at 500 volts, while Monsanto specification reads 100 at 90 volts.

Mr. Clark emphasized that this was of importance since if the resistivity was high and consistant, they would be able to cut out their Fuller's treatment. Most of the material received from Monsanto has a resistivity of over 1500 ohm-cms., which is the specification on material which General Electric uses in their plant.

Apparently, our limit of 100 at 90 volts actually corresponds to General Electric's 500 at 500 volts, since their results on identical samples run consistently higher than our's by a factor of at least five.

There is some question as to the effect of aging on resistivity, as in sealed glass tube, it has been observed to go up or down on aging.

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Mr. J. Fred Reeves stated that he felt that the Anniston laboratory should seriously look into going to 500 volt resistivity testing and convert their equipment to this voltage if at all possible in order to have the same test as General Electric uses.

A question was raised concerning the impregnation test. Mr. Clark stated that one car had been returned several years ago, but he said that if all other tests are met except, for this one usually there will not be any trouble. The trouble that was encountered in this case was corrected by Fuller's earth treatment.

General Electric treats every car received from Monsanto with Fuller's earth. They would be interested in avoiding this and might be able to avoid it by setting these specifications higher. The additional cost to General Electric encountered by Fuller's earth treatment is about 10%. Mr. Clark feels that the tank cars are sufficiently well sealed so that no appreciable change in resistivity, due to moisture entering the tank car, would result from shipment.

Mr. Clark stated that at Pittsfield they have storage capacity equivalent to about four weeks production. At Fort Edwards they have storage capacity equivalent to about six to eight weeks production. They have some new storage for crude at Pittsfield but this was not too good since no heating coils were provided in the storage tanks. They did have breathers on the storage tanks.

Their normal inside storage tanks have coils and excellent breathers of a better construction than the breathers on the crude storage. Their crude storage holds between three and four tank cars. Their refined storage is connected to a vacuum source.

Mr. Clark pointed out that with the Pyranols there was very little trouble experienced compared with trouble encountered with the formerly used oils. He stated that Westinghouse does not treat with Fuller's earth and neither does Allis Chalmers.

The impregnation test in the commercial unit is, at present, too expensive to be installed either at St. Louis or Monsanto Anniston testing laboratories. It may be necessary to do so, according to Mr. Clark, when the patents run out, since this may be needed for protection.

Mr. Clark stated that they did not have any water content in the specification for Aroclor or Pyranol, since they treated with Fuller's earth anyhow. There had been some difficulty experienced last summer to get the water content below 30 parts per million. The question was raised as to the drying

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of the Fuller's earth. At Anniston, drying at 150°C. for twenty-four hours is used. General Electric uses 400°C. for one-half hour. Mr. Clark stated that he did not think that this high temperature drying did materially effect the absorptive properties of the Fuller's earth. However, the Fuller's earth does not change color at 150° but gets darker when heated to 400°C.

Mr. Clark stated that the trichlor benzene often gave trouble on the corrosion test, due to side chains in the benzene. Trichlor benzene obtained from the Dow Chemical Company was high in water and had to be dried thoroughly here when it was used last summer. Mr. Clark stated that they approved the higher chlorides obtained with Hooker's TCB only because they have to. Recently Dow stepped out of the picture, as to possible supplies of TCB, due to the chlorine shortage. Also, Dow has experienced specification difficulties.

The TCB obtained from Hooker lately has not met the corrosion test. Mr. Clark stated that the trouble went back to traces of toluene in the benzene from which trichlor benzene was manufactured. Mr. Clark would like to see Monsanto make trichlor benzene at Anniston in order to have an additional source of supply for this material. He mentioned that at present although there was a chlorine shortage, General Electric was putting pressure on Solvay in order to obtain a new supplier for trichlor benzene. He emphasized again that he would like to see Anniston manufacture trichlor benzene.

Mr. Clegorn asked whether Mr. Clark thought it would be desirable to be a little on the high side when 1260 is supplied. Mr. Clark stated that now the transformers were O.K., and in the past, trouble with transformers had been a major factor in their setting of the specifications for 1260. He felt that he would hope to go to 1254 instead of the Pyranol 1482 type since these materials were more stable. He thinks that trouble in the old days was due to contamination.

Refractive index was discussed shortly and Mr. Clark promised to send us curves showing the relation of composition to refractive index for Pyranol 1467, which contains 40% trichlorobenzene.

Dielectric strength was shortly discussed. For oils, specifications read 30 KV while for Pyranols the specification is 35. Actually, Pyranols will run between 40 and 45 KV on dielectric strength.

Mr. Clark was asked about his ideas concerning the possible mechanism of the action of tetraphenyl tin as a stabilizer in liquid dielectric. He did not have a full explanation but he thought that some of it did go all the way to tin tetrachloride. He mentioned that General Electric was in touch with the Metal Thermite Company in order to find a supply of the desired stabilizer. Their demand will be between 10 and 12,000 pounds per year. This company has offered them tetrabutyl tin, but only on experimental production scale. This company has a project and a consultant at the Columbia University.

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General Electric is now paying $1.80 per pound for tetraphenyl tin and believes that the price will be going to $2.00 per pound. Hooker is not anxious to keep this business and in order for them to supply General Electric, General Electric gives them a year's order for this material at once, which is then manufactured by Hooker. Mr. Clark is still very much interested in getting dibutyl diphenyl tin as a possible substitute for tetraphenyl tin.

Trichlor diphenyl oxide was discussed at length. Mr. Clark did not have any specifications on electrical properties, except that the dielectric constant, which was the most important property, must be the same as 1476 or should be slightly less. He did not believe that color of the material was a critical factor. This material was used in capacitors. Superior low temperature behavior is exhibited by this material in combination with TCB. Thus, the capacitor containing trichlor diphenyl oxide exhibits 80% of the dielectric constant at 25°C. on cooling to -55°C. This is of great importance in low temperature work and would allow smaller capacitors to be used for low temperatures than are permissible with Aroclor filled capacitors.

Trichlor diphenyl is not a possible substitute in this application for the desired trichlor diphenyl oxide since it cannot meet the desired specification of 80% at -55° for the dielectric constant measured at 25°C.

Mr. Clark also mentioned that they're adding anthraquinone for low temperature use. However, this is only used on DC equipment and not on AC equipment.

The diphenyl derivatives are definitely all right in transformers. Although a small addition of the diphenyl oxide derivative shows some improvement, this is not sufficient to be practical. One advantage of the diphenyl oxide derivative is the lower viscosity.

Hooker has been making some of this material for Lubrizol. Mr. Clark warned that they had experienced explosions with some of their still residues.

Mr. Clark says that General Electric will pay a little more for the diphenyl oxide derivative since a special application was involved. They are interested in obtaining 100,000 pounds per month.

Concerning chlorinated ethyl benzene, General Electric's interest is not for a dielectric but for a hydraulic fluid application. The potential demand for this material is unknown. Du Pont is not making it at present. Mr. Clark believes that Westinghouse is only interested in chlorinated ethyl benzene in order to have their own material different from the trichlor benzene used by General Electric. Dr. Jenkins stated that Monsanto was interested more in chlorinated benzene now since with the acquisition of the new Texas plant from the government, Monsanto has their own ethyl benzene in the company.

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Concerning price, Mr. Clark stated that Du Pont was charging eighteen cents per pound of chlorinated ethyl benzene and that it would definitely not be lower than sixteen cents per pound.

However, in the dielectric field, chlorinated ethyl benzene would compete with TCB, which is six cents per pound, or with chlorinated biphenyl. Chlorinated ethyl benzene is said to exhibit slightly better low temperature characteristics.

Mr. Clark mentioned that toluene gave rise to pyrolysis and also that there was a slightly lower yield and that he did not think so much of chlorinated toluene for this application.

Due to lack of time, isocyanates could be discussed only briefly with Mr. Clark. He showed general interest and requested that he be sent a memorandum on available isocyanates for experimental purposes which should include some of their physical and chemical characteristics.

Mr. Clark also showed an interest in obtaining a reliable source for lacquer-lined drums to be used for Aroclors. This information was kindly furnished him by Mr. Sanderlin.

Edgar E. Hardy

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