APPLIED SCIENCES SECTION

PHYSICAL CHEMISTRY · ANALYTICAL CHEMISTRY · SPECTROSCOPY · INSTRUMENT RESEARCH
COMPUTER TECHNOLOGY · STATISTICS

MONTHLY SUMMARY

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For

Functional Fluids

Distribution

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FUNCTIONAL FLUIDS

AROCLOR - ENVIRONMENTAL PROGRAM

The analysis of rat tissues (fat, kidney, liver and muscle) from the Industrial Bio-Test 30-day tissue collection study have been completed. The samples (72) were analyzed as outlined in Analytical Chemistry Method No. 70-1 and the PCB levels were estimated using the Aroclor fed as the standard. The results agreed very well with what is being observed environmentally, namely that the estimated PCB levels found in all tissues decreased as the degree of chlorination decreased (1260>1254>1242) and increased as the dietary level increased. The isomer distribution of all Aroclors fed were also markedly altered. A more detailed report has been issued.

Several JFQ plant sewer water samples (4) were analyzed for PCB's. The N. ~~trunk~~ sewer contained less than 47 ppb, the S. trunk 147 ppb, a June composite sample from the main sewer effluent 46.0 ppb, and a July composite sample from the main sewer effluent, 43.5 ppb. In all cases, the PCB was identifiable as Aroclor 1248.

A second set of PCB/air samples (6) were analyzed for the Appleton Coated Paper Company. As in the past, the results were forwarded to them for interpretation.

Samples of mold sand used by Ford-Dearborn (2) and GMC-Pontiac (1) were analyzed for Aroclor 1242. The amounts found ranged from 80-1200 ppm.

The analysis of a variety of fish for PCB (31) collected during the semi-annual survey of the Choccolocco Creek have been completed and a report issued. A second set of fish samples (4) from this area are in hand and analysis of these will be completed this week.

PCB's have been observed in a number of carp and catfish (7) taken from the Mississippi river around Kimmswick, Mo. The levels found were, with one exception, in the low ppm range (<10 ppm).

Six (6) water samples from the JARD Company, Inc. have been analyzed for PCB's to establish if they have a significant effluent problem. PCB's were detected in all samples. A report is being written.

EC/GC support at the rate of 10 samples/day is being provided for the semi-continuous activated sludge degradation study of Aroclor 1130, 1242, 1254, and MCS 1016. These studies were started 8-10-70 and will run approximately 30-60 days.

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A study to isolate and identify potentially toxic oxygenated impurities in Aroclors has been initiated. No compounds of this type have been observed in Aroclor 1260, Lot AK-3; detection limit ca 2 ppm. Higher levels of chlorinated naphthalenes have been observed. Aroclor 1242, Aroclor 1254 and the competitive product Pheneclor DP 6 will be investigated by this procedure.

BIODEGRADATION TESTING

Testing of Aroclor 1242, Aroclor 1254, MCS 1016, and fractionated Aroclor 1130 in our semi-continuous activated sludge units is now in progress. These units are being fed at a daily rate of one milligram under identical conditions to permit a comparison between our present Aroclor products and the proposed substitutes. EC/GC analyses for the first weeks of operation have been completed except for calculations. River die-away tests are now being started on these same products.

PYDRAUL AC

Infrared analyses showed that deposits from a Pydraul AC compressor installation at Electric Energy, Inc. were mixtures of phosphate ester and a magnetic metal oxide. No evidence for petroleum oil or other contaminants was found.

DIPHENYL ETHER

Diphenyl ether to be used in a new fluid was produced from phenol. The major impurity in this reaction mixture was identified by GC/MS as dibenzo furan. A compound with a molecular weight of 196 which is most likely a diphenyl propane or an alkyl substituted dibenzo furan was also identified.

ELECTRICAL FLUIDS

The equipment being assembled for life tests of experimental capacitors was sufficiently completed to permit testing of capacitors in one of the two ovens. Preliminary tests showed that the oven connectors used did not have adequate insultation for high voltages. The second oven is being assembled with connectors of higher rating, and the first oven will be converted to these connectors. A recorder for continuous monitoring of the capacitors on test also is under construction.

RECORDING TENSIMETER

This instrument, built for use by NASA at the Lewis Research Center, was delivered, ready for use. The most optimistic time and budget estimates were met.

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MCS 293

A statistical study of correlations between slow 4 ball wear test data, Falex data and other wear tests was made on MCS 293 containing various lubricity additives. Correlations were fair.

TURBINOL 153

A Turbinol 153 (MCS 153) charged compressor at the Texas Eastern's St. Francisville Station has had two failures within one month due to deposit build up. Infrared analysis of deposits collected after each failure showed that they were high in a glycol ether that was not our FH-139 additive and inorganic carbonates, sulfates and magnetic oxides. The sample from the most recent failure contained in addition a hydrocarbon oil. Ionic chlorides are also present in the 2-30% range indicating brine contamination.

SKYLUBE 450

Atomic absorption analyses of an additional six samples of dipentaerythritol phosphite ester showed 1.2 to 1.9% potassium.

NONYL/CUMYL PHENYL DIPHENYL PHOSPHATE (NCPDPP)

Specific heat and thermal conductivity measurements were made on nonyl and cumylphenols and a 2:1 molar blend to provide data for engineering design. Data for the phosphate ester was obtained several months ago for the Pydraul reformulation project. The effect of aliphatic vs aromatic CH structure was very clear in the specific heats of the phenols. Data covered the molten temperature range up to 120°C plus some specific heat data for the supercooled liquids.

Six additional samples of NCPDPP were analyzed for trace magnesium by atomic absorption. These data were needed for process evaluation on crude and finished NCPDPP. Crude and finished material showed a range of 1150 to 1210 and 3 to 9 ppm magnesium respectively.

The vapor pressure curve for nonyl phenol was determined with the Automated Isotenioscope.

PHOSPHATE ESTERS

Vapor pressure curves of candidates for our new phosphate ester fluid line and a competitive material were determined with the Automated Isotenioscope. The samples included MCS-030-E, MCS-050-E, MCS-0115-E, and Fyrquel 550.

SKYDROL LD

Skydrol LD was analyzed for copper by atomic absorption. These data were needed in a test program to reduce formation of soluble cupric salts in Skydrol fluids. Copper levels from 1 to 880 ppm were found.

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Lockheed Aircraft complained of yellow particles in Skydrol LD lot QM-1. Infrared analysis showed that these particles were identical to the drum lining material (drum lining series 46).

BROMINATED BIPHENYLS

An IR method was developed for measurement of 0 to 60 ppm water inbromine with a sensitivity of 0.5 ppm and an estimated accuracy of 1 ppm water. The method is required to check the recycled bromine used for preparing brominated analogs of Aroclor products to minimize corrosion problems. Analysis of the technical bromine used in the first "W" Building run indicated the corrosion experienced with the stainless steel dip pipe was not due to the bromine. The water level was found to be 10 ppm. This is believed to be a safe level.

SKYDROL 500

IR analysis of a deposit from the hydraulic system of Mohawk Airlines A/C 32J showed that they arise from the thermal stressing of the fluid. About 33% of the deposit is residual Skydrol 500 containing some free phenol. The solids are metal butylphenyl phosphate salts.

A solid was observed in a fluid at United Air Lines after removal from a pump after 236 hours use. It was identified by IR as the thermally stressed reaction product of Skydrol 500; i.e., a metal salt of butylphenyl phosphoric acid. Emission analysis indicated that iron and phosphorus were the major metallic elements present. Minor quantities of chromium, manganese and nickel were also detected.

A solid contaminant formed in Skydrol 500B at the NYCO blending facility and halted production. It was identified by IR as an amorphorous silica such as a diatomaceous earth.

A gel plugging the 0.45 μ filters in the J. F. Queeny Plant small canning operation of Skydrol 500B was found by IR to be a mixture of a styrene-acrylic copolymer and Skydrol 500 base stock. Our reference polymer spectra indicate that Rohm and Haas makes at least five styrene-acrylic copolymers suggesting that this contaminant was introduced in their HF-412 operation.

AROCLOR DIELECTRICS

Eleven lots of Aroclor 1242, 1242B, 1254 and Kaneclor KC-300 were analyzed by atomic absorption for trace iron. None was found (<5 ppm). Iron was suspected as the cause of poor electrical properties of Japanese product.

Seven distillation fractions of Aroclor 1242 were analyzed by IR. Significant differences in the spectra were observed due to a varying isomer distribution. The data indicates that a mixing tank should be included in the plant design. If not, customers who use IR for quality control may object or complain about variations in product quality.

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A large number of Aroclor 1121, 1130 and 1142 distillation fractions were analyzed by GC for isomer distribution to compile data for plant design. The chlorinated biphenyl isomer separation has been improved by use of a 100 foot SCOT (support-coated open tubular) capillary column coated with FFAP liquid phase.

A study to isolate and identify potentially toxic oxygenated impurities in Aroclor products has been initiated. No compounds of this type have been observed in Aroclor 1260, lot AK-31. The detection limit is ca 2 ppm for oxygenated components. Higher levels of chlorinated naphthalenes have been observed. Aroclor 1242, Aroclor 1254 and the competitive product Pheneclor DP6 will be investigated by this procedure.

A solid which appeared in a distillation fraction of Aroclor 1242 was shown by IR to not be the suspected 4,4'- or 2,4-dichloro-biphenyl isomers. The IR spectrum suggested this was a higher chlorinated Aroclor. Mass spectrographic analysis showed that the material contained tri, tetra and pentachloro isomers with about one-half the sample being tetrachloro isomers.

Differential IR analysis of an Aroclor 1242 sample returned for reclamation showed the mineral oil level was 0.018% by volume. A second lot showed <0.01% contamination.

PYDRAULS

The German Mine Authority has set a maximum limit of 4% petroleum oil contamination in Pydraul fluids. They have also asked Monsanto to provide methods for verifying contamination is below this level. Differential IR methods were developed for Pydraul A-200, Pydraul 280, Pydraul 281, Pydraul 625, and Pydraul 625A to measure 0 to 4.5% oil in the fluids with a sensitivity of 0.1%. Experiments have shown that rather large compositional variations in the formulations would have only minor effects on the accuracy of the methods. For TCP in Pydraul 280 (21.8% level) and in Pydraul 625 or Pydraul 625A (50% level), 5% TCP variation would be equivalent to 0.2% oil. For Acryloid 732 in Pydraul 281 (3% level), 0.3% Acryloid 732 would be equivalent to 0.27% oil.

PYDRAUL REFORMULATIONS

The comparison study of Pydraul blends and base stock specific heats, thermal conductivities and densities is completed. Results look encouraging. Linear calculation of blend specific heats and thermal conductivities looked good in the two and four component blends studied (Pydrauls AC-A and 540A). Measured specific heats were within 2 and 6% of the calculated values and when corrected by a blend density factor were in the 1 to 2% range. Measured thermal conductivities were within 1% of calculated values. These comparisons covered the temperature range from 25 to 150°C.

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TRACTANT FLUID

A thermal stability experiment was run with dicyclohexyl-1,2-cyclohexane dicarboxylate to provide data for further testing of the compound. An endothermic reaction with gas evolution was observed above 225°C. The total condensation of decomposition vapors on cooling plus IR analyses identified cyclohexene as the gaseous decomposition product.

PYDRAUL 312

The major components of a recently introduced competitive fluid, Dasco Fr 300, have been identified. The fluid was separated into three major components by gel permeation chromatography: a phosphate ester, hydrocarbon oil, and fatty acid ester of a glycol. The first two are present in about equal amounts. The fatty acid ester makes up 5 to 10% of the fluid. The phosphate ester is a mixed ester of phenol, cresol, and alkyl phenols such as ethyl phenol and dimethyl phenol. The hydrocarbon oil is aliphatic and has a number average molecular weight of 450. The fatty acid ester is probably a natural product. Work is continuing to identify the glycol from the fatty acid ester, to quantify results, and to identify low level components of the fluid.

Another competitive fluid, Chem Trend HF-30, was analyzed by GC and found to contain 3.5% low boiler, 69% Aroclor 1254 and 27% hydrocarbon oil.

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