5B Ford Engine Test: Equations were developed for predicting sludge and varnish ratings from formulation and engine variables. Equations were modified to assure realistic predictability. Commercial laboratory results were compared with predicted values.

Equations were developed for bench scale settling depth test and these equations applied to engine results to determine if formulation variables behaved in the engine in a similar manner to their behavior in the bench test.

1H Diesel Engine Test: Preliminary studies were run on the effect of formulation on 1H Diesel engine piston ratings.

Exhaust Gas Analyzers: The second chromatographic exhaust gas analyzer, recently installed in the Engine Laboratory, was overhauled and a defective column was replaced. The timer motor was changed to reduce the cycle time from 20 minutes to 12 minutes to produce as rapid analysis as was possible within the limitations of the chromatographic elution times. A third chromatograph has been installed and is being demonstrated.

Temperature Controller: An auxiliary relay unit for a West Temperature Controller was assembled in order to handle the power requirements of the proposed heater load.

PLASTICIZERS

Brabender Tester: Design work is complete and construction has started on a system to provide direct temperature vs. torque plotting from the Brabender (plastics consistency) tester.

Santicizers 141 and 148: Laboratory batches were analyzed for residual sodium content by atomic absorption spectrophotometry. The results showed concentrations in the range of 7-19 ppm and demonstrated that a correlation exists between sodium content of the phosphate ester and control laboratory data from water soluble salt tests.

Isodecyl phosphorodichloridates were analyzed by GC as the dimethylamine derivative. Finished material was analyzed by GC to determine purity.

Santicizer 465 (S-465): Plant batch No. 1 of S-465 (ethylene bis butylphthalate) produced at Everett was found to contain a high boiler. This was isolated by GC and identified by IR as a phthalate ester involving an alkyl group larger than butyl.

Triaryl Phosphates: Chlorophenyl diphenyl phosphate (S-192), cresyl diphenyl phosphate (S-140), tricresyl phosphate (TCP), and triphenyl phosphate (TPP) crude reaction mixtures were analyzed by GC to determine the efficiency of the reaction.

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Santicizer 151: An experimental design was set up to study the effects of various processing conditions on color, acidity, peroxide content and yield of Santicizer 151 laboratory preparation. Samples are being tested in Saflex for edge stability and adhesion. Results will be correlated with processing conditions and Santicizer 151 properties.

Santicizers 160, S-261, DOP, DTDP: We provided an experimental design, developed regression equations and provided consultation on a program to map out the effects of molding and milling conditions on the properties of plasticizer-PVC formulation.

Flame Test: We determined the data correlation between the two foot Monsanto Flame Test and industry standard 100 foot test for flame resistance of plastics containing Monsanto plasticizers and flame retardants. The correlation is sufficiently good to permit replacement of the large scale test by the Monsanto test.

Pigment Systems: Assistance was provided in the study of plasticizer pigment systems for pigment milling applications. Theoretical and empirical equations were fitted. Graphical techniques for formulating to specific customer requirements were devised.

RESIN MATERIALS

Maleic Anhydride: Neutron activation analyses of Monsanto's and National Aniline's maleic anhydride were made in an effort to better resolve the superior heat stability of National Aniline's product. Gulf General Atomic, Inc. made the analyses for Monsanto. Results gave no evidence of a National Aniline heat stability additive. Their product did contain six times more chlorine and twice as much phosphorous but concentrations in both products were below the 1 ppm level. Monsanto's maleic anhydride contained 15 times as much aluminum (8 ppm vs. 0.5 ppm), detectable silicon (3 ppm) and higher sulfur (0.72 vs. 0.26). This suggests the possibility that aluminum and/or silicon may be the cause of the inferior heat stability of Monsanto's maleic anhydride.

Tetrachlorophthalic Anhydride (TCPA): GC analyses indicate that the hexachlorobenzene-TCPA ratio in acetone solution is not altered during evaporation of the acetone solvent on a steam bath.

Phthalic Anhydride: Flow control capillaries were fabricated for a standard sample generator at Chocolate Bayou. The generator is used to calibrate process control instruments on the phthalic anhydride process.

GENERAL CHEMICALS

Phenol: DTA experiments have been run with sodium sulfate and sulfite, using the Perkin-Elmer DSC-1B differential scanning

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Phenol: (Continued)

calorimeter to confirm sulfate transition data and to look for similar transitions in the sulfite. Three distinct changes were observed in the sulfate as predicted by the literature. Several lower energy transitions were also observed in the sulfite but at higher temperatures.

Process samples from the neutralization, evaporation and fusion steps at the WGK Plant were analyzed by XRD as is and after washing with alochol. Only the samples free of alcohol soluble materials have been identified. Thus far, no evidence of crystalline polymorphism has been observed. The samples have been found to contain either $Na_2 SO_4$ (Thenordite) or $Na_2 SO_3$.

Santowax C: Oxygen atmosphere DTA experiments have been completed with six potential insulation materials for the Anniston Santowax C plant. The six materials, in order of best to poorest oxidative stability with Santowax C are: Amosite asbestos, Kaylo-20 silicate, high density mineral wool, foam glass, Carney expanded silica and fibrous glass. Some were much worse than the presently used calcium silicate, which has caused fires at leaking pipe flanges.

Paranitroaniline (Mfg. W.A. 46-67): The determination of physical properties requested for the design of new pNA production facilities has been completed. These include solubilities of pNA in mother liquor compositions, viscosity, density and vapor pressure of saturated solutions, viscosity and density of molten pNA, viscosity and density of pNA saturated mother liquor and the heat of fusion of pNA. The viscosity and density measurements on pNA saturated mother liquid over the temperature range of 90 to 160°C. had to be obtained at pressures sufficient to keep the ammonia and water in solution (150-280 psig). This required the design and fabrication of specialized equipment for measuring viscosity and density under pressure.

Biphenyl: GC analyses have been completed on samples from eleven additional terphenyl suppression runs. Another six runs are scheduled.

Nitrochlorobenzene: An automatic sampling valve was modified for the W.G. Krummrich Plant. It is part of a Beckman Process Control Chromatograph used in the nitrochlorobenzene department.

RUBBER CHEMICALS

N-3 Polyester Cord Dip: A heat of reaction of 57.5 cal/g was obtained with the DSC-1B differential scanning calorimeter for a 10 to 1 molar ratio of resorcinol and triallylcyanurate. This reaction product is an intermediate for a polyester cord dip with a good market potential. The reactants were mixed at the melting point of resorcinol, encapsulated and the heat of reaction run between 200 and 220°C with the DSC-1B.

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4-Nitrodiphenylamine (4-NDPA): A heat of vaporization of 56 calories/gram was measured for 4-NDPA at 230°C. This measurement was made at a pressure of about 5 mm Hg with our Perkin-Elmer DSC-1B. This information was needed for engineering design of distillation equipment at WGK.

PIONEERING RESEARCH

Reprography: Air solubility in SBR polymers Kraton 102 and Solprene 303 was determined by weight increase following prolonged vacuum treatment. This data was needed for calculation of velocity and diffusion constants for absorption of oxygen in the polymer film.

TECHNICAL COMPETENCE

Atlas-MAT (Varian) CH-4B Mass Spectrometer: Testing of the new medium resolution Atlas CH-4B mass spectrometer is continuing. Emphasis is being placed on the gas chromatography rapid scan mass spectrometry (GC/MS) capability of the instrument. Packed columns are presently being used. A new single-stage Biemann helium separator has been ordered to extend our capabilities to capillary column GC.

Heats of Vaporization: A method for measuring heats of vaporization at reduced pressures has been successfully demonstrated with the Perkin-Elmer DSC-1B differential scanning calorimeter. Vacuum operation down to several millimeters of Hg can be achieved so that the $\triangle H_v$ of high boiling materials can be measured directly. Repeatability of results with 4-NDPA was excellent; a further check will be forthcoming when six triaryl phosphate samples have been measured.

Heats of Reaction: A method for measuring the heat of reaction of resorcinol and triallylcyanurate was successfully demonstrated with the Perkin-Elmer DSC-1B differential scanning calorimeter. The reactants were combined in the liquid state (no reaction), encapsulated and quickly raised to the reaction temperature. Experiments run at 200°, 213° and 220°C gave results of 56.3, 59.9 and 56.2 cal/g, respectively. Time of the reactions varied from 15 to 23 minutes. It appears from these data that we can measure heats of reaction with good precision on reactions of this type.

New Gas Chromatographs: Two new F&M 5751B gas chromatographs and an Infotronics CRS-100 electronic digital integrator were received and checked out during the month. These chromatographs which are being readied with support coated capillary columns and "on-column" injection systems will be assigned to laboratory process studies.

Loaner Gas Chromatographs: Approximately 0.2 man-month was expended on diagnosing instrument malfunctions, making minor repairs and adjustments, arranging for major repairs, and general consulting on gas chromatography applications involving loan instruments.

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New Research Job Number System: We set up the conversion of the Organic Division Research job numbers from four to seven digits on January 1st. The new numbering system is designed to provide better cost control and better association of actual work with authorizing documentation. The new job number is composed from the product group, project analysis and charging group leader numbers. New computerized reporting programs are being implemented by the Evaluation Section of MISD.

Statistics Course: A course in "Introduction to Statistical Analysis" was completed by 15 research personnel.

Motor Drives: Three Reliance Compak V-S Motor Drives at the High Pressure Laboratory were modified by the installation of a pilot lamp assembly in each to indicate that the motor has been started.

MISCELLANEOUS

Mercury Inventory: Radiochemical data from the WGK and Anniston mercury inventories have been processed and a Special Study report has been written. The twenty-two DeNora cells at WGK contained a total of 204,500 pounds of mercury and the twenty-nine operational cells at Anniston contained 121,900 pounds ± 1.1% and ± 1.3%, respectively, at the 95% confidence limit.

PERSONNEL

Jimmy G. Converse, from Iowa State University, joined the Instrument Research Group, following completion of work for the Ph.D. degree in chemistry.

William J. Litschgi, from the J. F. Queeny Plant Analytical Laboratory, joined the Analytical Chemistry Group.

Thomas J. Furdek, from the J. F. Queeny Plant Analytical Laboratory, joined the Spectroscopy Group.

George W. Ashworth retired on December 31st after 38 years of service with Monsanto in the Research Department of the Organic Chemicals Division.

PUBLICATIONS

Hinchen, J. D., "Statistics in Analytical Chemistry", Journal of Gas Chromatography, 5, 641 (1967).

Wait, S. C. and Grogan, F. M., "The Electronic Spectrum of Cinnoline Vapor", Journal of Molecular Spectroscopy, 24, 383 (1967).

Keller, R. E., "Adipic Acid", Encyclopedia of Industrial Chemical Analysis, 4, 408 (1967).

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Keller, R. E., "Adipic Acid Derivatives", Encyclopedia of Industrial Chemical Analysis, 4, 423 (1967).

SPEECHES

Mr. J. D. Hinchen presented a talk on "Experimentation in the Factory" before the Peoria, Illinois local section of the American Society of Quality Control on January 10, 1968.

M. W. Dietrich
E. M. Emery
L. Fowler

R. E. Keller

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