COMPANY CONFIDENTIAL

MONSANTO COMPANY
ANNISTON ALABAMA

December 1971

Distribution:

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J. C. Landwehr
TSD/Lab

Ag Division

Winterization for Methyl production during cold weather was completed except for the final items requiring plant shutdown. These will be completed during the late January-early February turnaround.

Design completed for conversion of P_2S_5 therminol system to Therminol 66.

Preliminary design completed for addition of three Dorr-clones for removal of waste treatment plant solids. Decision to move was based on the excellent performance of the existing Dorr-clone in solids concentration.

Vendors were contacted and quotes requested on equipment for recycle of Niran sniff chlorine vapor to tank cars as unloading pad.

Detail estimates were made for PNP expansion by 2.0$\overline{M}$ lbs. Project will be prepared in early January.

Polyphenyls

The Santowax Q storage tank arrived. CED drawings received and being modified to permit installation of only tank and pump. Some materials (E/I) on hand and transferred from CED construction to storeroom. Other items being ordered and expedited. The location of the tank was changed due to potential blocking of a fire lane (alternate route will be blocked more than originally thought). Complete project package will be transmitted by 15 Jan. '72.

HB-40

Issued progress report No. 3, 105-1161 on 17 Dec. '71 on Hydrogen Purification Unit Revamp. Design capability upgrade from 65% to 70% recovery.

TSD is investigating the cost of a 500 psi hydrogen compressor to allow shorter WCM autoclave cycles and higher recovery of available hydrogen.

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Solid Aroclor

Scheduled shutdown and abandonment of these products by mid-1972 has caused a wave of uncertainty concerning the future of the Polyphenyl business!

Standby Propane System

Ignition system for remote lighting of flare installed and operating. Through Dec. 31, 1971 the unit has operated for 207 hours during curtailment periods. Some minor work still remains before project completion.

Fire Alarm System

Alarm system was modified and successfully tested. The alarm is now "locked in" when the code is dialed, i.e. the phone is "free" to make other calls as soon as the alarm starts.

HB-30 Crystals Study

Twenty samples of HB-30 were prepared to evaluate the effect of hydrogenation level, quaterphenyl level, and blending on the formation of crystals in the freeze-thaw test. Results showed that all samples made within the present hydrogenation level specification limit passed the freeze-thaw test. However, samples made by blending material with a hydrogenation level lower than specified with material with a higher hydrogenation level did not pass the test. It is, therefore, critical that each batch be hydrogenated within spec limits.

J. C. Landwehr

/dp

PLANT LABORATORIES

ANALYTICAL AND QUALITY CONTROL - S. O. Kemp

Complaint Report

Quality

Number	Material	Customer	Reason
ATL 24-P	S-140 & S-981	Pandel Chemicals	S-140 off color but material was accepted; S-981 - temperature 110°F too high

YTD Complaints/100 Shipments 0.27

Target - 0.20

1970 Manpower/sample	0.63
1971 YTD Manpower/sample	0.39
1970 $/manhour	13.18
1971 $/manhour	8.80

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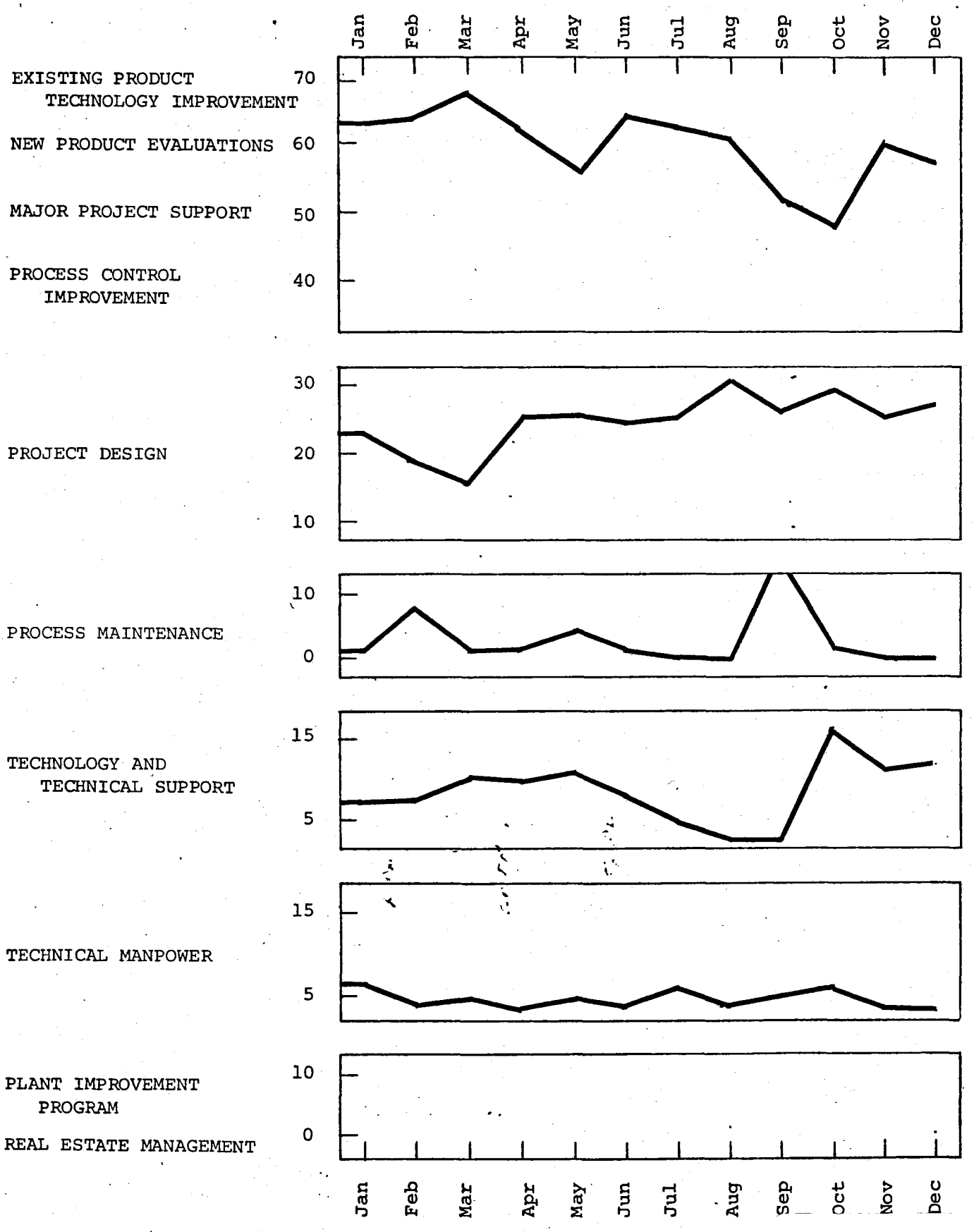
1971 DISTRIBUTION OF TIME BY TECHNICAL SERVICE



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C O N F I D E N T I A L

THIS POLLUTION CONTROL SECTION IS FOR IMMEDIATE INFORMATION PURPOSES ONLY. NOT TO BE FILED.

Pollution Control Summary for December 1971 - E. G. Wright, M. D. Turner (J. T. Bell)

Environmental Control Log Book Information

Nothing during December.

Air Pollution

1. Three air pollution complaints were received from plant neighbors during December. All were investigated and attributed to SO_2. Weather conditions during the month hampered the dispersion of SO_2.

2. Monsanto Research Corporation was contacted in December for a quote on using their math model for predicting SO_2 seasonal concentration isopleths. This data will be used to determine the optimum location for an SO_2 monitoring station. This station will be necessary for satisfying O.S.H.A. and Alabama Air Pollution Commission rules governing SO_2 ambient air concentrations.

3. Considerable efforts in December were expended in preparing a statement of Monsanto's position on the proposed rules and regulations by the Alabama Air Pollution Commission. This statement will be submitted at the Commission's public hearings in January if deemed appropriate.

Waste Pollution

1. Drs. Gunning and Suttkus (Biological Consultants) collected the December set of fishes from Choccolocco Creek on Dec. 6, 7, 8, and 9. This is the first quarterly trip of the second year in the P.C.B. residue studies.

2. During December 132 samples were analyzed for trace P.C.B.'s. Fifty-three of these were water samples which included 18 final lots of HCl for shipment. Seventy-nine samples were analyzed for the Nitro Plant in a continuing effort to define their P.C.B. problems.

MID-MONTH REPORT
PR&D LABORATORY
ANNISTON PLANT

PROJECT TITLE: Methyl PCT Analytical Methodology

PERIOD ENDING: December 15, 1971

CHEMIST: Jerry L. Brown

Objective:

To determine differences between Anniston and St. Louis GLC methodology for PCT assay and isomer analysis.

Results and Discussion:

A sample of Methyl PCT produced from the improved PCT process described in Ag Research Progress Report 2104 was analyzed by Anniston and St. Louis. The results of these analyses, as previously reported, are as follows:

	Anniston Method 044	St. Louis Method
% Me PCT	91.9	94.5
% "C" Isomer	2.47	2.04
% "G" Isomer	4.02	0.3

This sample was then reanalyzed during the above time by the St. Louis method. The results were as follows:

% Me PCT	91.62
% "C" Isomer	2.46
% "G" Isomer	2.28

The GLC was then converted to conditions, including columns, employed by the Anniston procedure. The sample was again analyzed in duplicate resulting in the following:

% Me PCT	94.4
% "C" Isomer	1.41
% "G" Isomer	0.71

A close examination of the St. Louis instrument revealed that the major difference from the Anniston instrument was injection port design. Sample injected into the former instrument had to

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travel a much longer path through a metal port than the latter, which utilized on-the-column injection. It was decided to try a third instrument which was equipped with glass columns and on-the-column injection. Results from this test are:

% Me PCT	91.66
% "C" Isomer	2.95
% "G" Isomer	2.65

It was decided that this procedure utilized the most thermally-stable conditions possible and should, therefore, yield the best results.

In addition to the above, a sample of Methyl PCT prepared by the recycle process was analyzed on this GLC resulting in the following:

% Me PCT	96.25
% "C" Isomer	0.83
% "G" Isomer	1.93

Conclusions:

1. It was concluded that two phenomena can occur in the GLC that can cause the discrepancies that have been noted. These are (a) isomerization of PCT by S_2Cl_2 to result in an abnormally high content of "G" isomer and (b) thermal degradation of "G" isomer to result in an abnormally low amount of "G" isomer. It would be possible for either phenomena to occur separately or at the same time at some GLC conditions. Therefore, to minimize these discrepancies, it was decided that a GLC procedure be adopted which utilized glass columns, on-the-column injection, and the lowest operating temperature possible.

2. GLC is at best a poor tool for analyzing these thermally unstable compounds. The most satisfactory analytical tool known is phosphorus NMR which is a non-destructive method.

Jerry L. Brown

Jerry L. Brown

/dp

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MID-MONTH REPORT
PR&D LABORATORY
ANNISTON PLANT

PROJECT TITLE: Differential Thermal Analysis (DTA) Study of Improved Methyl PCT Process Described in AG Research Progress Report 2104 (1)

PERIOD ENDING: December 15, 1971

CHEMISTS: G. L. Arnett and I. Ransaw
Written by I. Ransaw

AG Research Report 2104 (1) by A. E. Lippman describes a process in which the yield of distilled Methyl PCT is increased 7 - 9% by increasing the chlorination level from 105 to 112%. This process involves the following basic steps:

1. Chlorination - A batch of 1500 grams of thio acid is chlorinated using 690 grams of chlorine @ 50 - 55°C over 6 hours, with N_2 added to the chlorine at a rate of 240 ml./min. after 520 grams of chlorine is in.

2. Sulfur Removal - Part of the sulfur (liquid) is allowed to settle from the chlorination mass over a period of 2 - 3 hours at 45 - 50°C while the remainder of the sulfur is removed after holding the mass for an approximate 12 hour period at room temperature.

3. Distillation - The chlorinated mass (without sulfur) is distilled at 10 mm to a pot temperature of 80°C.

To provide thermal stability data for this process, two Methyl PCT batches were prepared according to this process for DTA analysis. For comparison a batch was chlorinated to 105% (typical for plant).

DTA results for the chlorinated batches (before distillation) were within the normal DTA limits:

@ 80°C -- 50 cal./kg./min.

@ 100°C -- 150 cal./kg./min.

DTA's for the still residues of the two (proposed process) batches and comparison batch were as follows:

Process Run #1

@ 80°C -- 72.7 cal./kg./min.

@ 100°C -- 403.3 cal./kg./min.

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Process Run #2

@ 80°C -- 72.7 cal./kg./min.

@ 95°C -- 403.3 cal./kg./min.

@ 100°C -- Off chart

Comparison Batch

@ 80°C -- Nil

@ 100°C -- 63.5 cal./kg./min.

These results indicate that the still residues for the proposed process were abnormally high (unstable) in comparison to typical plant still residues.

In an effort to stabilize or find the cause for instability in the still residues, changes were made in the process following the chlorination step or sulfur removal step. The procedure and results of the changes are described in the following tests:

Run #3

After chlorination the chlorinated mass (no sulfur removed) was allowed to set at room temperature overnight and then distilled at 10 mm to a pot temperature of 80°C. The still residue was allowed to cool to 45 - 50°C. Then a residue sample (containing approximately 1:1 ratio by volume of organic to the sulfur mass) was transferred immediately to a DTA cell. The DTA results are as follows:

@ 80°C -- 87.8 cal./kg./min.

@ 100°C -- 313.7 cal./kg./min.

Run #4

After the sulfur removal, 205.3 grams (wt. equiv. to sulfur removed) of No. 2 diesel fuel was added to the chlorinated mass before distillation. The distillation was carried out under normal conditions. The still residue had the following DTA:

@ 80°C -- 61.2 cal./kg./min.

@ 100°C -- Exotherming too badly to complete DTA

Neither of the process changes resulted in a more stable residue.

I. Ransaw

/dp

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