Monsanto

W. R. Richard - Research Center - St. Louis, Mo.

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ANNISTON VISIT JULY 6, 1970

File R & D

TO :

J. T. Bell
J. C. Landwehr
J. R. Savage
J. W. Baker
M. W. Farrar
J. E. Smith
R. H. Munch
P. Benignus/J. Bryant
L. Stark/P. Fowlkes
Q. Thompson/R. Weiss

Biphenyl/terphenyl tubular process - T. Bell and Jerry Brown - Unit is built and operating. Acetone, nitrobenzene, cumene are being run as promoters; isopropanol, benzoic acid, azobenzene, methane on list of possibles.

We expected O_2, H_2, (CO?) to take part in the benzene to polyphenyl reactions, based on experience at these temperatures in free radical ethylene polymerization. Parts per million O_2 are critical in the ethylene reaction. Parts per hundred thousand H_2 are chain transfer agents. We have analyses for O_2, good to ppm.

We speculated that Ashland Oil using a dealkylation catalyst (toluene to benzene or methylnaphthalene to naphthalene) had found that toluene over this catalyst was a promoter for biphenyl reaction. T. B. and J. B. are to contact W. Knox for more knowledge of dealkylation catalysts. See also patent review by W. Eberhardt. W. Richard to look up G. Mortimer's work at Texas City on chain transfer agents in ethylene polymerization.

We discussed need for keeping dibenzofuran and dibenzodioxan out of the biphenyl to avoid toxicity problems. This reflects need to watch oxygen in product in benzene and in promoters.

A 2% increase absolute in conversion would be significant; say from 20 to 22%.

Berns Severson discussed use of CS_2 in pilot unit to avoid carbon fouling, vs. plant practice to avoid use of S. The pilot tube reactor has been exposed to "S" and this may or may not be correlatable with plant results. We recommended continued work with present reactor but said we should fabricate another non-sulfur exposed preheater and reactor so that experiments without "S" could be run before committing to plant trials.

We discussed ways of increasing conversion in the biphenyl tube. Catalyst injection at points downstream in the ethylene - polyethylene reactor had increased conversions in that process. Could this work in benzene to biphenyl process? Promoters which made free radicals easily vs. those that took higher temperatures and longer times might give higher conversions. We considered possibility of dual promoters. Nominal dwell times are 7 seconds reactor/1-2 seconds preheater, plus more time in decoking and separator sections. Ethylene polymerization takes 30 seconds. Dwell time in biphenyl reactor is shorter ¼ to ½. Anniston has an automated sample injector on GLC tube to help handle biphenyl reactor analytical samples. Anniston has consulted with L. Fowler-E. Emery for more automation. Problem is getting timely results with limited number of people.

R. F. Brooks and Bob Small wrote a note, 6/30/70, on the dual process for running the biphenyl reactor to produce high terphenyl at reduced costs. The idea was to run for maximum biphenyl rate and then under different reactor conditions run for maximum terphenyl rate. The scheme demands extra product storage according to R. Brooks.

Quaterphenyl suppression - Only suggestion so far was to run H_2 in the reactor or crack Montar #9 in presence of H_2. Can we get more information on hydrocracking catalysts from Hydrocarbons Division?

Hydrogen Compressor - We wondered whether an improved compressor lubricating oil would provide longer operation of Ingersoll Rand compressors. Present oil selected 10 years ago.

HB-40 - clay treatment of Santowax C reduces H_2 catalysts usage - looks OK. Clay treat to reduce color of HB-40 for plasticizers looks too expensive at the moment. Appears "cheaper" to distill Santowax C.

We expect high quaterphenyl content due to need for more terphenyl from the biphenyl reactor. Might have 7-9 M lbs of Montar #9 in '71. Joe Baker outlined set of HB-X runs with high quaterphenyl contents (10, 25, 35 and 50%) to be hydrogenated to 30, 40, 50 and 100% level. T. Bell to send Montar #9 to Joe Baker at St. Louis to distill for increasing quaterphenyl content of Santowax R. Anniston to run hydrogenations to 50% level. Ask QET/J. Wygant to run 100% hydrogenation runs for tractant greases.

Anniston could use another hydrogenation autoclave for the laboratory work.

Aroclor 1242

Problems in Japan because of a Kanagafuchi introduced power factor-heat stability test were mentioned. Ralph Munch will try to tell whether Kanagafuchi has a stabilizer or simply better Aroclor. We know all Aroclor production at Krummrich, Anniston and Newport is not equally good on power factor after exposure to heat or to heat and oxygen.

Anniston-V. Haupt & T. Bell discussed results of color in Aroclor after distillation. They had found a correlation between different batches of lime from Alabaster Lime. "Vertical lime" was better than "horizontal lime." T. Bell to elucidate further in memo. Anniston has issued new specifications on lime.

The Japanese trouble started with an off-color batch of Monsanto Aroclor 1242 and our competition has convinced customers in Japan that Monsanto Aroclor is less stable.

The Krummrich plant is having trouble with sulfuric acid in chlorine.

A Newport recent sample was high in TCC. Trouble could be in sampling. This has happened before.

Aroclor Clean-up - Talked briefly with Allen McCarty about coalescers for separating Aroclor/H_2O. The Fram coalescer on HCl-Aroclor-H_2O stream has fallen apart after 6-9 months in operation. Allen sent literature on coalescer from RPC Division-Midland Ross which uses a polypropylene diaphram instead of rubber. This coalescer of interest to our Aroclor customers as well as to our plants. K. Hale has assignment on coalescers at Anniston.

I. Ransaw was planning to use the plant outflow from the limestone pits as a source of Aroclor saturated water to gain information on adsorption of Aroclor on carbon. He was setting-up 1" diameter carbon absorbers in the field.

Dayton had run into the problem of adsorption of Aroclor on the walls of the stock solution and Anniston experienced similar difficulty. We needed information from Dayton earlier about this problem. Clay, because of outflow acidity, may not be a desirable adsorption material.

Joe Landwehr gave me an updated copy of Aroclor plant clean-up projects. The solids handling problem seems most "up in the air." We seem to visualize (1) an extraction process with solvent extracting clay floor dry material to recover Aroclor or Pydraul; (2) a water displacement process on a steam distillation treatment to recover Aroclor; (3) a rotating kiln to volatilize Aroclor followed by incineration of the volatilized Aroclor or Aroclor/phosphate ester. What happens when we "volatilize" Pydraul from floor dry?

Aroclor 5400 Series - Environmental

Analytical chemists are beginning to track chlorinated terphenyl by its chlorinated biphenyl content. We want to operate the biphenyl/terphenyl column for minimum biphenyl content in terphenyl and suggested target is .1% biphenyl in terphenyl. The Plant has a capacity problem. Also have problems with quaterphenyl/terphenyl split.

Ed Emery has written letter summarizing earlier work on chlorinated biphenyl in chlorinated terphenyl which was originally intended to secure FDA clearance.

Problems are caused by high quaterphenyl content in Santowax R because the chlorinated quaterphenyl content of 5460 won't distill at temperatures and pressures available in the plant. Seem to be getting some HCl. Jim Savage has discussed the possibility of vacuum steam distillation with J. Landwehr and D. Cova. An inert carrier is desired. Questions were raised about the hydrolytic stability of Aroclor 5460 in the presence of steam and lime at high temperatures. We aren't anxious to make phenols or chlorinated furan or dioxane structures. Nor are we anxious to corrode out the steel equipment.

Joe Baker will start the investigation by contacting D. Cova and seeing if the appropriate Montar can be distilled by vacuum steam or vacuum-(benzene?) without decomposition.

Aroclor 5430-5435

This product is desired in Pydraul reformulations to replace chlorinated biphenyl. We want a low viscosity chlorinated terphenyl which is compatible with phosphate esters and does not form insolubles when mixed with hydrocarbon oils and phosphate esters. We want minimum chlorinated biphenyl in the product to avoid "tracing". G. Arnett has worked out some of the controls for plant production of chlorinated terphenyl. R. Weiss is working on isomerization of 5430 to see if lower chlorine content products can be made and still retain total liquidity.

Arobromoclor

We reported making Arobromoclor 1232 from Aroclor 1219-1221 by direct bromination and said we were demonstrating HBr recovery and conversion to Br_2 plus making 30 gallons of product in W-Bldg. Pilot Plant. Product is for Aroclor 1242 replacement in Pydraul.

WRR

W. R. Richard

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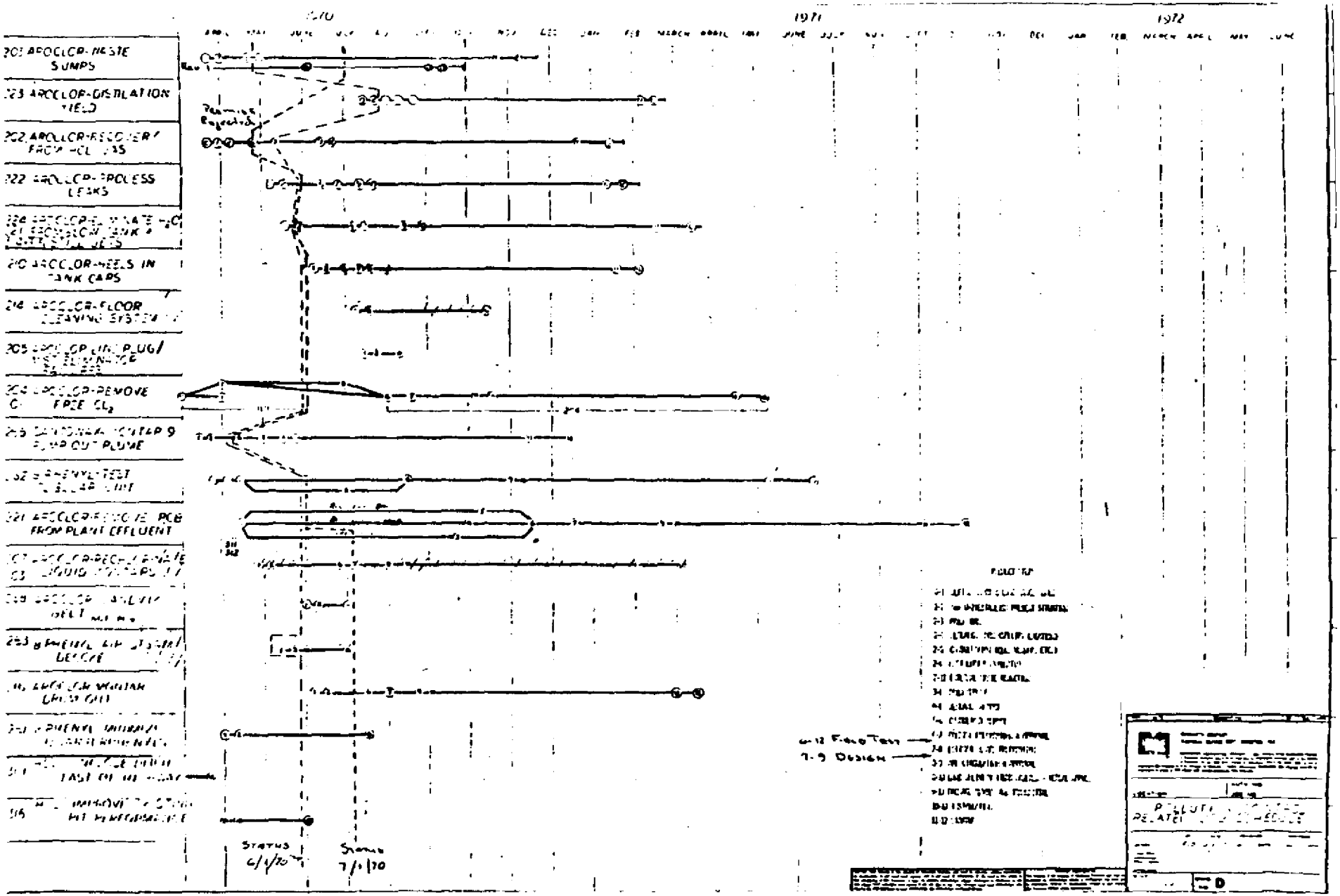
1970
1971
1972
203 AROCLOR-WASTE SUMPS
223 AROCLOR-DISTILATION YIELD
202 AROCLOR-RECOVERY FROM HCL GAS
222 AROCLOR-PROCESS LEAKS
210 AROCLOR-HEELS IN TANK CARS
214 AROCLOR-FLOOR CLEANING SYSTEM
204 AROCLOR-REMOVE FREE CL_2
221 AROCLOR-REMOVE PCB FROM PLANT EFFLUENT
Status 6/1/70
Status 7/1/70
6-12 Field Test
7-9 Design

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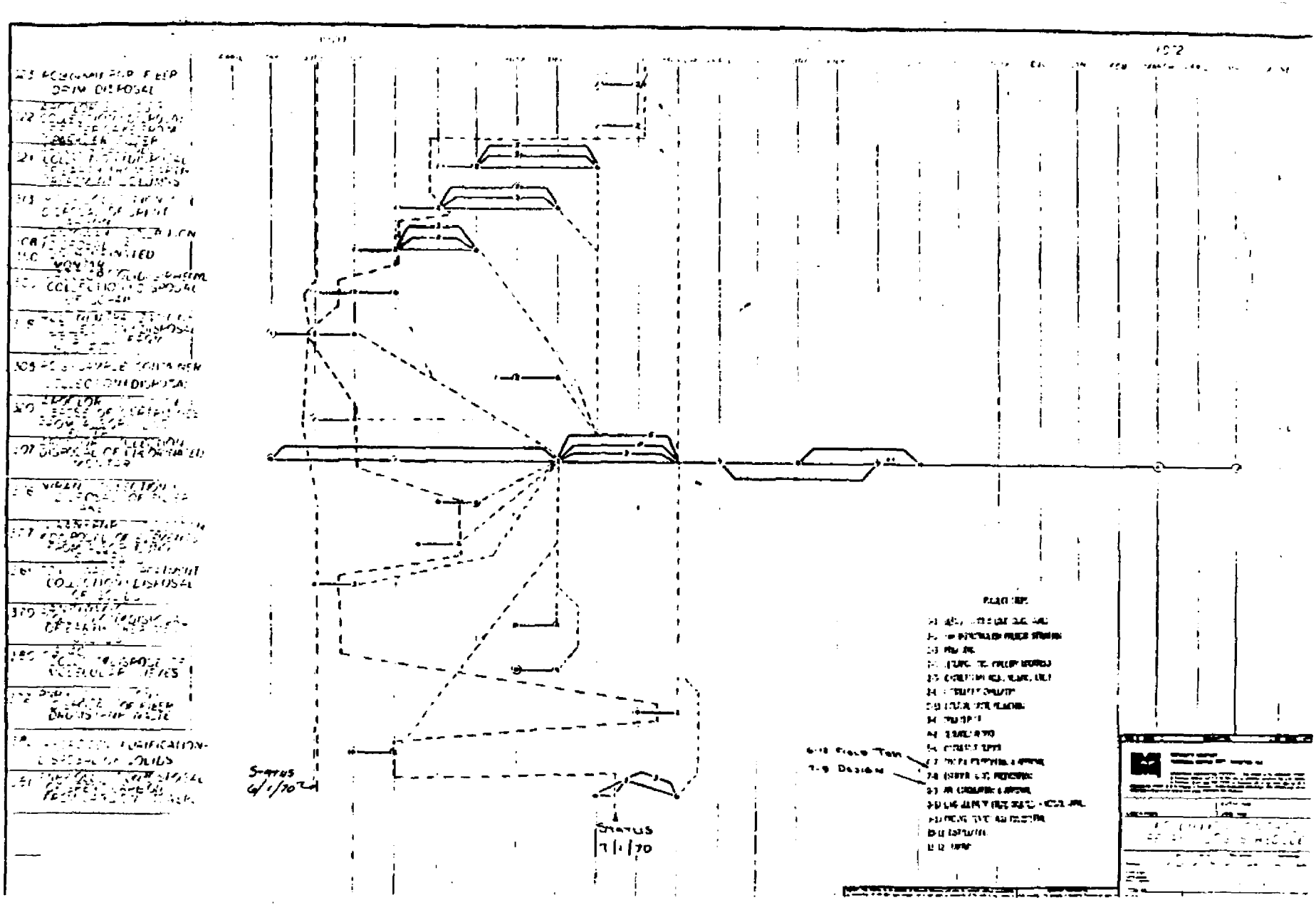

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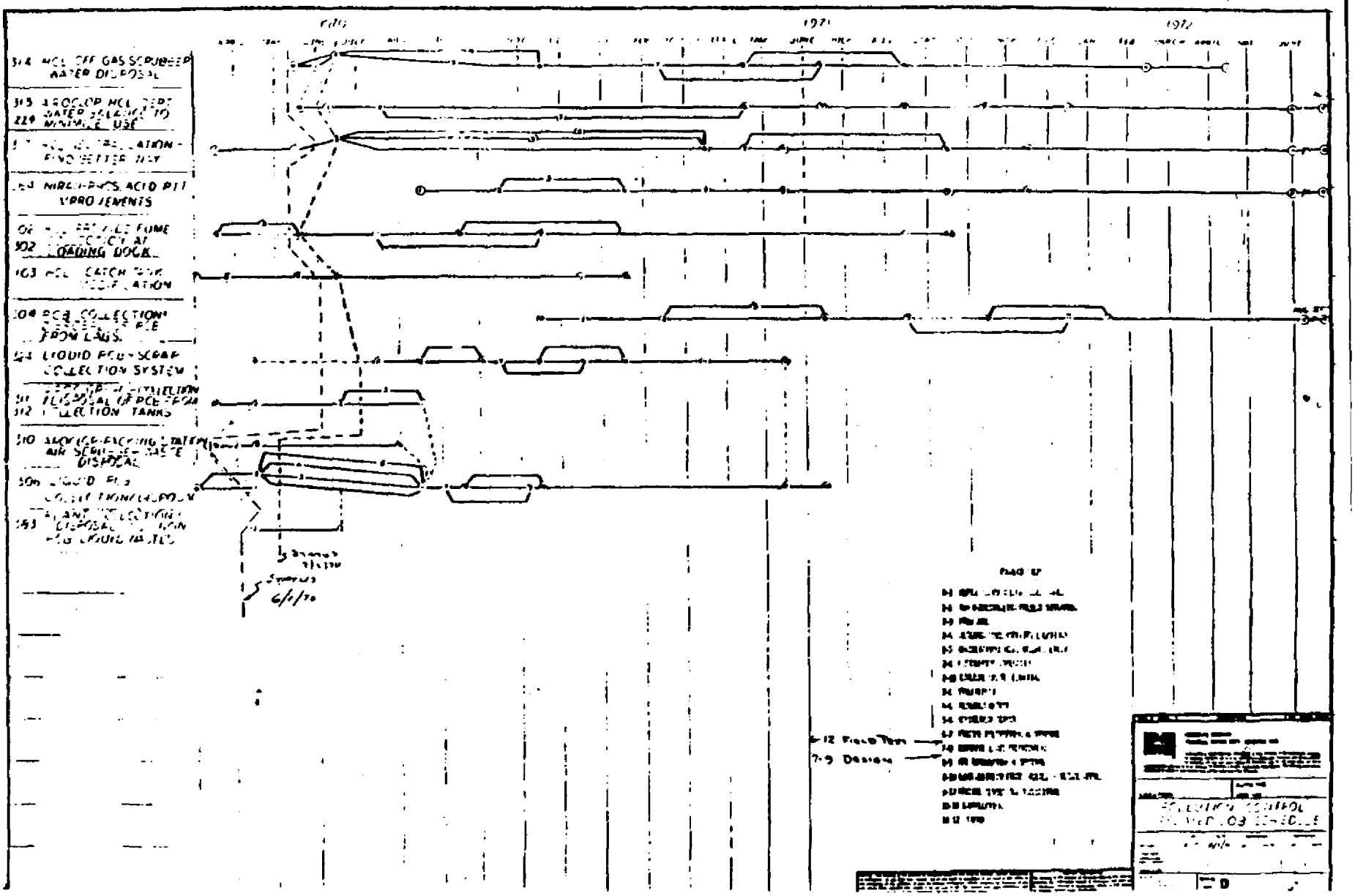

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