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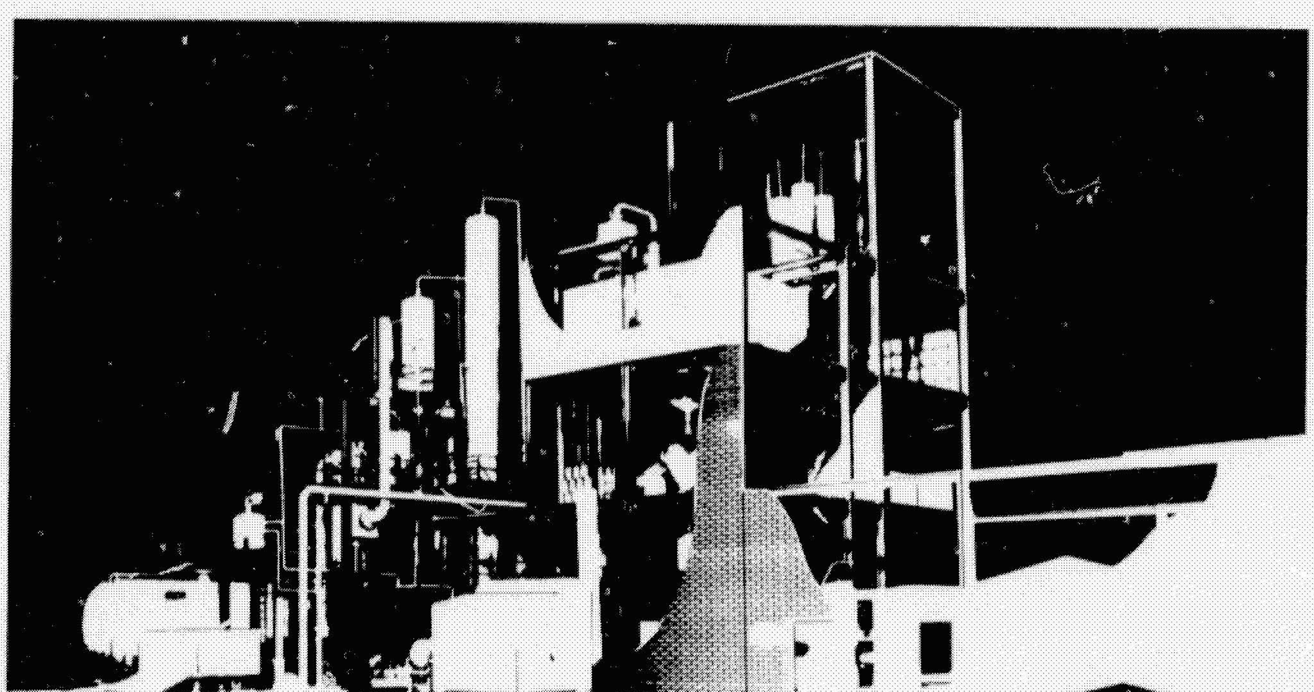
QUARTERLY PROGRESS REPORT

JAN - MAR 1980

low cost solar array project

EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE SILANE-TO SILICON PROCESS



UNION CARBIDE CORPORATION

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ABSTRACT

This report covers the work performed in January, February and March, 1980 on JPL/DOE Contract 954334, Phase III. This phase consists of the engineering design, fabrication, assembly, operation, economic analysis, and process support R&D for an Experimental Process System Development Unit (ESPDU).

With completion of the design for the hydrogenation reactor and four distillation columns, only the free-space reactor and the melting/consolidation system design remain to be completed. The waste-burner test was completed this quarter, so the waste treatment system for the EPSDU can now be specified.

The facility layout drawings were completed and are ready for review. These will form the basis for the installation design activities. The drawings incorporate the revised waste treatment system, environmental considerations, and personnel safety.

According to the detailed cost estimate just completed, cost of the construction sub-contract is much higher than planned. Fortunately, recent process design changes, which are not reflected in the estimate, will have a sizable impact in lowering the estimate. The cost-impact study will be carried out in April.

Equipment procurement activities are just starting. Purchase orders for most of the equipment are expected to be issued in the next quarter.

The free-space reactor PDU was successfully started up. After some equipment modifications, the unit was run for 24 hours at the design throughput. Powder purity and longevity of the quartz liner will have to be addressed next.

The melter sub-contract was signed with Kayex Corporation of Rochester, New York, and work was started on March 1, 1980. Theoretical models for shot formation and shot cooling, from a bottom-apertured crucible, are being developed by Union Carbide, in support of the contract.

In the fluid-bed development area, the following tasks have been completed:

- All fixed-bed experiments were completed, and correlations for the silicon deposition rate and a critical silane concentration curve were developed.

- Particle separation experiments were completed which revealed positively that large particles can be selectively removed from a fluid bed.

- Capacitive heating experiments showed that a fluid bed can effectively be heated without heating the bed wall.

- Design of the fluid-bed PDU is underway, and the P&I diagram was completed.

A chromatographic technique has been developed for determining trace levels of phosphine in silane down to a few parts-per-billion. The slim-rod reactor and epitaxy reactor are being assembled and both should be ready for testing in the next quarter.

Overall, the EPSDU engineering function is progressing satisfactorily but is slightly behind schedule in the P&I and equipment procurement area. This is not expected to impact the overall schedule. The process support R&D activity proceeded on schedule until the free-space reactor testing was interrupted by the powder scraper malfunctioning and breakage of quartz liner. A concentrated effort in these areas during the next quarter should solve these problems and put this part of the program back on schedule.

SECTION I. INTRODUCTION

This report covers the work performed in January, February and March, 1980 on the JPL/DOE Contract 954334, Phase III.

The overall objective of the LSA Silicon Material Task is to establish a chemical process for producing silicon at a rate and price commensurate with the production goals of the LSA project for solar-cell modules. This material must be suitable for utilization in the large-area sheet process and in the automated process for the fabrication of solar cells having satisfactory physical and electrical performance characteristics.

As part of the overall Silicon Material Task, Union Carbide developed the silane-silicon process and advanced the technology to the point where it has a definite potential for providing high-purity polysilicon on a commercial scale at a price of $14/kg by 1986 (1980 dollars). This work, completed under Phases I and II of the contract, provided a firm base for the Phase III program (initiated in April 1979) aimed at establishing the practicality of the process by pursuing the following specific objectives:

- Design, fabricate, install, and operate an Experimental Process System Development Unit (EPSDU) sized for 100 MT/Yr to obtain extensive performance data to establish the data base for the design of commercial facilities.

- Perform support research and development to provide an information base usable for the EPSDU and for technological design and economic analysis for potential scale-up of the process.

- Perform iterative economic analyses of the estimated product cost for the production of semiconductor-grade silicon in a facility capable of producing 1000 MT/Yr.

This process for preparing semiconductor-grade silicon in the EPSDU from metallurgical-grade (M-G) silicon is based on a well-integrated arrangement of purification steps that provides a cost-effective process system.

The three basic steps entail converting M-G silicon to trichlorosilane, redistributing the trichlorosilane to produce silane, and thermally decomposing the silane to form polycrystalline silicon powder. The powder is then melted and the molten silicon is cast for subsequent fabrication of solar cells.

The technical progress presented in this report is arranged according to the Work Breakdown Structure (WBS) shown in Table I.

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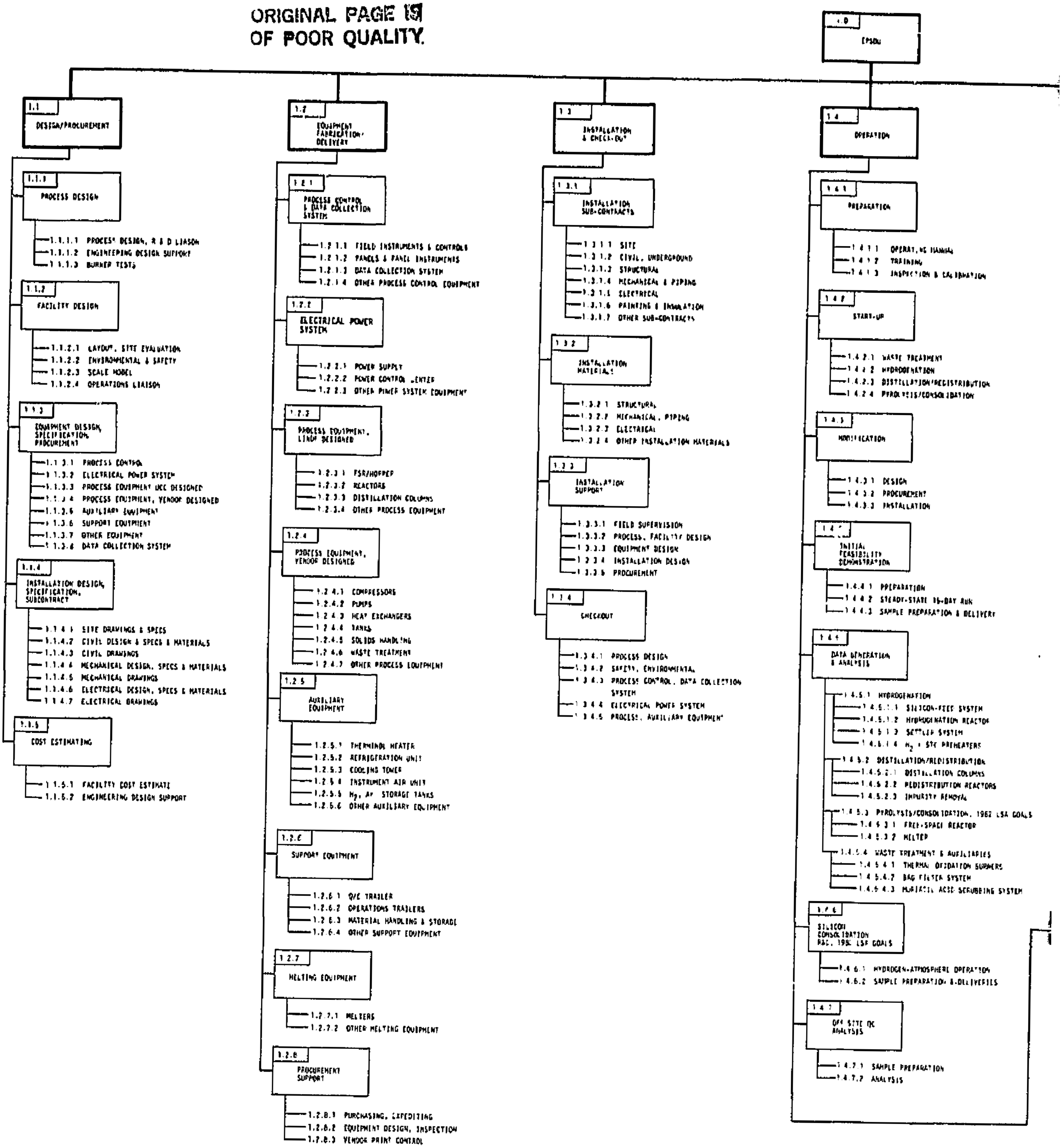

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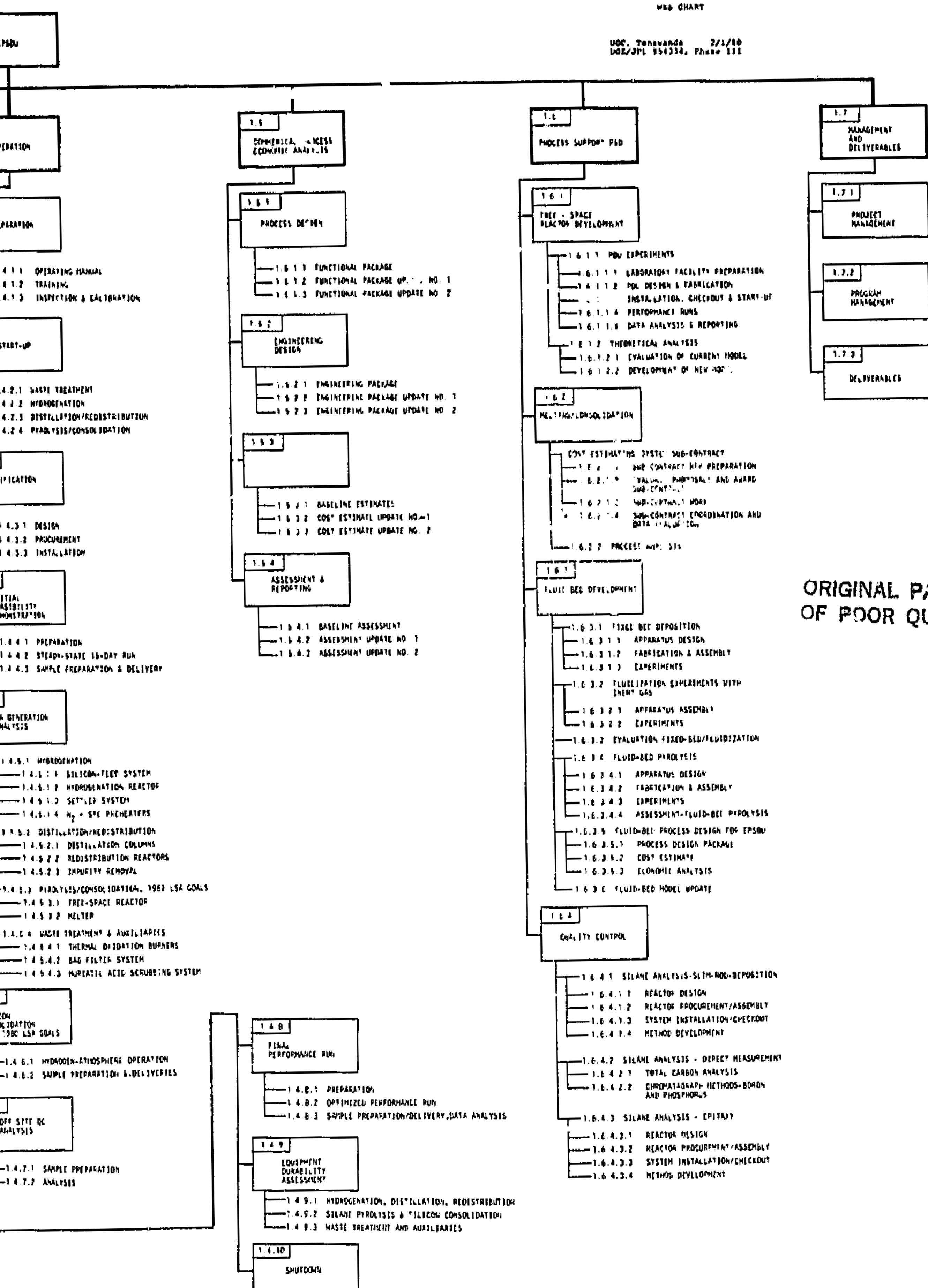
TABLE I

WORK BREAKDOWN STRUCTURE



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SECTION II. TECHNICAL ACTIVITIES (BY WBS NUMBER)

1. EPSDU PROGRAM

As illustrated in Table I, the current Phase III program consists of seven primary (WBS level 2) divisions of effort:

- EPSDU Design and Procurement
- EPSDU Equipment Fabrication and Delivery
- EPSDU Installation and Checkout
- EPSDU Operation
- Commercial (1000 MT/Yr) Process Economic Analysis
- Process R&D to Support EPSDU Design and Commercial Analysis
- Program Management

Collectively, these activities encompass all effort required to attain the program objectives. The subdivisions (WBS levels 3, 4 and 5) define the individual work items that must be performed. The progress for this quarter, documented in this section, is reported at the work-item level. Only work items that are currently in work or were recently completed are included.

1.1 EPSDU DESIGN/PROCUREMENT

This effort includes all engineering, design, and procurement activities necessary to transform the process design, developed during the Phase II program, into a complete installation-drawing package for EPSDU. The major tasks include process design updates, facility design, equipment design and procurement, installation design, and cost estimating support.

1.1.1 Process Design

The process design effort is geared toward using the most recent information available to provide the most practicable integration of process subsystems for attaining the EPSDU program objectives. The process design package consists of a heat/mass balance, process description, process flow diagram, and functional specifications for process equipment.

The original package, issued in June 1979, served as the basis for all subsequent engineering effort. Beneficial data from the Supporting R&D effort and other process-related analyses and experiments is being used to update the original package. Process engineers, using information available from the Phase I and Phase II programs, provide direct support to the facility and equipment design efforts.

1.1.1.1 Process Design Update

The process design is continually being reviewed to identify refinements that have the potential for lowering the cost, reducing the requirements for specialized equipment, or alleviating problems that could arise during operation. The effort for this quarter was concentrated primarily on the following:

- M-G silicon conveyor system
- Hydrogenation reactor
- Waste treatment
- Silane distillation column
- Equipment drying procedure

The conveyor system for moving the M-G silicon feed for the hydrogenation reactor from the storage bin to the lock hopper was re-examined to ascertain if the required size was commercially available. Commercial dense-phase pneumatic conveying systems are larger than that required for EPSDU and would not be cost effective. Flexible-screw conveyor systems are available which are ideal for this application. This type of conveyor has a bearing housing at the motor-end only; therefore, the conveying spiral, made of spring steel in a non-rigid plastic tube, can be cut to size. The manufacturer has demonstrated the capability to transport similar materials successfully at any angle. The new conveying scheme is to nitrogen-feed the copper-blended silicon to the conveyor which transports it to a lock hopper elevated slightly above the bottom section of the reactor. This will provide a simple, low cost, and reliable system.

The hydrogenation reactor was modified to include a pair of perforated-pipe gas distributors. This mechanically simplifies the reactor, and provides for easier maintenance and flexibility in gas injection to the fluid bed.

The waste treatment process was changed so that the muriatic acid byproduct is now neutralized and disposed of in the sewer as a dilute solution, instead of being concentrated to a nominal 20% acid. The new scheme will be much simpler and less expensive for EPSDU; however, the original method of producing a salable muriatic acid by-product is still attractive for a large commercial facility. In the new neutralization scheme, the neutralizing tank will be centrally located to process the acid wastes scrubbed from the combusted waste-gas and the plant wastewater. An estimated 30 to 50 gpm of wastewater will come from the acid gas scrubber and up to 50 gpm could be generated from curbed and other areas where process spills, if they occur, will be isolated. The neutralizing tank will be designed to process up to 100 gpm of wastewater. The effluent will have a pH of 6 to 9 and will flow to the municipal sewer.

Functional design of all four distillation columns was completed. The work involved specifying mechanical details, materials of construction, process connections, column internals, column supports, and special cleaning procedures. The silane distillation column was changed from a trayed-design to a packed-design so that cleanliness standards can be maintained. The tray-column is not viable because, in this small size, the trays would have to be welded to the shell to minimize fluid by-pass. The column would consist of about two dozen fabricated parts that would be welded together. It would be nearly impossible to clean the column to electronic standards and it could not readily be field disassembled to remove tramp materials. The packed column is smaller and simpler. However, for a commercial plant, either a packed or cleanable trayed column could be used for sizes up to about 5000 MT/Y; a trayed design would be clearly preferred for larger sizes.

The equipment drying procedure was checked to make sure that sufficient nitrogen will be available at the EPSDU site. A functional specification for the dry-out heater was written. The heater would be located at the "gantry" and deliver 250°F nitrogen to all lines and equipment to be dried prior to plant start up.

1.1.1.2 Engineering Design Support

Because of the special process considerations required for EPSDU, process design personnel, using knowledge of the Phases I and II development effort and relevant chlorosilane work at other UCC locations, assist in the detail design of the facility and equipment. Although this participation encompasses many design activities, the effort for this period was involved primarily with environmental and safety considerations and, to a lesser extent, equipment finishes and cleaning.

In support of plant layout activities, a methodology for spill and fire control was developed in conjunction with environmental and safety consultants. The major concern was to provide proper drainage, water deluge, and fume control for chlorosilane spills. Data supplied by Southwest Research Institute, under an industry-wide contract, and consultation with the fire safety engineer at the UCC Sistersville plant indicate that hydrochloric-acid fumes and fire are both major hazards associated with chlorosilane spills.

Virtually all EPSDU process streams are flammable due to the presence of hydrochlorosilanes. The most effective fume-control agent appears to be a high-expansion water-based foam which suppresses fire and cools adjacent equipment. Hydrochlorosilane fires cannot be extinguished; they must be allowed to burn out. Based on this, the chlorosilane tankage and process areas will be diked or curbed to contain spills. A portable high-expansion foam generator will be provided to blanket large spills using water from strategically located hydrants. (Two vendors of foam generators have been identified.) Small spills will be flushed with water only.

Sluice valves in the containment areas can be regulated to allow a controlled discharge of diked liquid to the process sewer. Copious quantities of water will be added to the sewerage to hydrolyze the liquids and move the resulting solids. The wastewater will then drain to the central collector where it will be neutralized with caustic soda to adjust pH to permissible levels (6-9). As a b k-up fire-control measure, a water-spray deluge system will be used to protect the support steel in the process area. Chlorosilane storage tanks will not have an in-place deluge system, but will be insulated to protect them from external heat. However, hydrant-supplied water will be available if it is needed.

Specifications were prepared for primer painting and process equipment cleaning. These specification packages included recommendations of UCC experts at Sistersville. The painting specifications are important because the specified primers will reduce the possibility of stress-crack corrosion in stainless equipment, and external corrosion of steel equipment resulting from chlorosilane leakage. The planned operation schedule for EPSDU is tight and proper process equipment cleaning specifications should enable a rapid, less troublesome startup.

1.1.1.3 Burner Tests

An analytical and experimental development program was conducted to provide design data for an economically and technically viable waste treatment system for EPSDU. The tests were conducted to demonstrate operation of a burner system to flame hydrolyze and neutralize waste chlorosilanes. The tests were started in late 1979 and are essentially complete. The effort for this report period consisted basically of burner operating variations on representative chlorosilane wastes.

Although the burner had operated successfully with silicon tetrachloride (STC) vapor, the initial test with liquid STC was unsuccessful. The equipment was modified using a commercial fuel-oil atomizer and in-house designed atomizers. The commercial atomizer was not successful, but one in-house designed unit worked well in combination with a vendor-suggested modification to the burner flame holder. This phase of the work was completed with a successful long-duration test with liquid STC.

Our final task was to flame-hydrolize concentrated chlorosilane sludge obtained from our Sistersville facility. Two types of peristaltic pumps (borrowed from other UCC projects) were tried but neither could deliver a constant flow. This was probably caused by the thin, non-viscous nature of the sludge. An acceptable flow was attained by pressurizing the sludge tank with nitrogen. The initial attempt to meter the opaque sludge into the burner did not work. A rotameter with a magnetic pickup was then tried and it appeared to work satisfactorily. The valve used to meter the slurry was troublesome because the valve characteristics changed as solids built up around the seat. This was solved by attaching a vibrator to the valve body. After the metering system was reworked, a short test was tried using the Sistersville chlorosilane sludge. Although the atomizer did an adequate job of breaking up the liquid into a low-velocity droplet mist, a solids buildup kept occurring on the atomizer tip. The buildup rate was roughly constant for atomization with air, nitrogen, and natural gas; consequently, the problem appeared to be caused by a rapid solvent evaporation at the hot tip. Attempts to withdraw the atomizer from the flame front to reduce the tip temperature resulted in unstable burner operation. Withdrawal of the tip apparently reduced the combustion air flow due to the venturi-effect so that the flame was extinguished on three successive tries.

The chlorosilane sludge obtained from Sistersville could not be completely flame-hydrolized. It was decided that further investigations at this point, on the mini-burner scale, would not be worthwhile since the polychlorosilane content of the available Sistersville sludge is far larger than that expected at EPSDU (perhaps by over an order of magnitude). A mixture of 80% STC and 20% TSC (simulated EPSDU sludge, less solid) was tried next. Although the exhaust plugged after one hour of operation, the mixture burned properly with flame inside the flame holder. The solids content does not appear to present an operational problem if an atomizer with wide chlorosilane passages is used and a "rapper" mechanism is provided to prevent friable deposits from building up on the atomizer.

1.1.2 Facility Design

Facility design consists primarily of the effort required to translate the process design functional requirements into specific plans regarding site, physical arrangement, human factors, and safety and environmental considerations. Personnel who will operate the facility participate to provide human factor inputs and to become familiar with the process.

1.1.2.1 Layout, Site Evaluation

This effort includes preparation of layout, plot-plan, and elevation drawings to establish the spatial relationship of equipment and structures and provide the design basis for detail piping, structural steel, foundations, and electrical systems. Site evaluation includes definition of battery limits and utility connections, topographical surveys, and soil testing.

The site plan was redrawn to provide additional information and changes from the previous plan. The update included relocations required to provide roadway access to the EPSDU site, fencing relocations, existing roadway areas, source points on Kennedy Avenue for electrical power and natural gas, location of the cryogenic hydrogen storage tank, and site-grading features.

The possibility of obtaining additional supplies of natural gas through the Northern Indiana Power Service Company was discussed with NIPSCO personnel. A formal letter outlining the natural gas requirements was sent to NIPSCO so that they could respond with a supply proposal. NIPSCO responded verbally with information that a supply of 40,000 CFD natural gas will be available on a firm, non-interruptable basis, and 100,000 CFD on an interruptable basis. This additional supply of natural gas will permit a substantial reduction in the use of fuel oil for process heat and, in turn, a reduction in the size of the fuel oil storage tank.

1.1.2.2 Environmental and Safety

This environmental effort includes assessment of all environmental considerations associated with the process for regulatory compliance preparation of standards, and obtaining appropriate approvals and permits. The safety aspects include evaluation of the process and detail design and monitoring of installation and operation to ensure that all features necessary for public and staff safety are included and proper procedures are used.

Environmental and design features were reviewed as part of the facility layout meetings. Process wastes and spilled chlorosilane liquids will be neutralized in a common treatment tank with pH control prior to disposal in the sanitary sewer system. Potential liquid spills will be contained using a diked area around the STC and TCS storage tanks. Drainage curbs and trenches around pumps and process equipment will direct occasional spills or leaks to the neutralization tank.

The use of a portable, high-expansion foam system is now favored over a water-deluge system for HCl vapor and fire control in the event of a large chlorosilane spill in the STC/TCS storage tank are . Fire hydrants have been relocated for greater accessibility and effectiveness.

The revised waste treatment system requires greater use of caustic and process water for waste scrubbing and neutralization. Higher dissolved/suspended solid concentrations and higher wastewater flowrates into the East Chicago sanitary sewer will result. Updated environmental permit information will be transmitted to the appropriate agencies in April. The revised wastewater discharges should not adversely affect permit approval.

1.1.2.4 Operations Liaison

Operating personnel participate in the facility design effort to provide inputs and obtain process familiarity.

Operations representatives advised that a portion of the present sanitary sewer line at the East Chicago site may be too small to handle the now-increased EPSDU wastewater flows. An alternate "tie-in" design was proposed which will be evaluated in April.

A fire-water loop has been installed at the Union Carbide plant at East Chicago to supply the fire-water and potable-water systems for EPSDU. A drawing showing the actual locations of the installed fire-water loop at the EPSDU tap-in point was completed.

1.1.3 Equipment Design, Specification, Procurement

The equipment related effort includes development of the control system, preparation of the piping and instrumentation diagram, preparation of wiring schematics and control panel drawings, and the design of equipment. The specification activity includes definition of specific requirements for each item of equipment, preparation of bid packages, evaluation of vendor quotation, and preparation of final specifications and drawings. Procurement includes the issuance of procurement packages to selected vendors and obtaining comprehensive design information necessary for preparing installation drawings.

The design and procurement of each item of equipment is accomplished through the combined efforts of process engineers, equipment engineers, and purchasing agents. These efforts produce a series of documents that evolve, ultimately, into a complete, definitive procurement package. The individual documents (representative samples are exhibited in Appendix A) and their respective uses are as follows:

- Functional Specifications: This specification (Exhibit A) is developed by the process engineer based on process requirements reflected in the process flow diagram, heat/mass balance, and process control scheme. It defines the duty that this item of equipment must perform for the overall process system to operate.

- Engineering Specification: Using the Functional Specification as a basis, the equipment engineer determines the specific type of equipment necessary to satisfy the process requirements. This translation of process requirements into hardware-specific information is deliniated in the engineering specification (Exhibit B).

- Request for Quotation (RFQ): Request for Quotation form PUR 201 (Exhibit C), prepared by the equipment engineer, summarizes the equipment requirements, identifies vendors to be contacted, and defines the bidding instructions. This form plus the engineering specification constitutes the RFQ package submitted to the purchasing department for transmittal to potential vendors. Vendor quotations are reviewed by the equipment engineer and, based on a technical, cost, and schedule evaluation, a vendor is selected.

- Request for Requisition (RFR): The Request for Requisition, prepared by the equipment engineer, consists of the RFQ package plus the Bid Evaluation Report that identifies the selected vendor and the specific equipment model to be purchased. The RFR is submitted to purchasing and serves as the technical basis for the purchase order.

- Purchase Order (PO): Purchase Order form L334-31D (Exhibit D), prepared by the purchasing agent, definitizes the terms and conditions, delivery requirements, and billing instructions applicable to the particular equipment and vendor. This form supplements the technical information contained in the RFR to provide a complete procurement package. When the internal review cycle is completed and the appropriate approvals have been obtained, the validated Purchase Order is issued to the vendor.

- Procurement Status Report (PSR): After the order is placed, technical performance is monitored by engineering personnel and contractual performance is monitored by purchasing personnel. Status is reflected in the Procurement Status Report. (The PSR for March, presented in Appendix B, reflects the current procurement status of all EPSDU equipment.)

These six documents serve as milestones for measuring performance of the procurement cycle for each item of equipment.

1.1.3.1 Process Control

The controls systems engineering effort includes all activities associated with developing the P&I diagram, designing process control loops and control panels, specifying valves and instrumentation, and preparing control wiring and pneumatic tubing diagrams.

The design effort is basically complete and the procurement cycle has been started for all instrumentation except that for the waste treatment section. RFQ's were issued for a major portion of the process instrumentation as identified on the current version of the process and instrumentation (P&I) diagram released February 15, 1980. The following specifications formed a portion of the RFQ's:

- The "instrument equipment list" (A-2145987) provides specifications for 188 different instrumentation items for temperature, flow, pressure, and liquid level measurement/signal transmission. These items will be located on equipment and piping throughout the process.

- The "main instrument panel specification" (A-2145988) identifies over 20 instrumentation items to be mounted in the main control panel. These include temperature, flow, pressure and liquid-level controllers/recorders.

- The "automatic control valve specification" (A-2145970) lists 45 automatic valves required for process control.

Relay logic diagrams are being developed to enable specification of the relay circuit equipment, and development of the schematic wiring diagram.

1.1.3.2 Electric Power System

This effort includes all electrical engineering activity required to develop the power control and distribution systems, prepare the definitive electrical one-line diagram, and specify electrical equipment.

A technical and economic comparison of a direct Northern Indiana Public Service Company (NIPSCO) power-supply arrangement vs the currently proposed UCC/Linde in-plant supply arrangement favors NIPSCO supply. Alternates for a secondary source of emergency power were also evaluated and the most reliable source still appears to be a diesel-powered emergency generator at the EPSDU site. If primary power is lost, the emergency generator will automatically cut in and provide a level of power sufficient to operate the refrigeration system, some ventilation systems, and some lighting systems.

The electrical power equipment specifications were issued. Based on the specifications, all RFR's were issued including those for a 1500 KVA primary transformer, two 480V motor-control centers, and an emergency generator.

The 11.5 KV primary power will be supplied by NIPSCO and is reflected in the issued electrical one-line diagram. This diagram delineates, through single lines and simplified symbols, component devices and circuits that comprise the electrical system. It depicts the path of energy transfer and identifies circuit components and their ratings. Thus, the one-line diagram is a complete representation of the EPSDU electrical system from the receiving point to the equipment. Typical information includes:

- Power Supply - Incoming voltage, frequency and short circuit.

EPSDU incoming voltage is 11.5 KV, maximum short circuit is 36.4 MVA, and minimum short circuit is 24.7 MVA.

- Transformers - EPSDU includes a 1500 KVA, 11.5 KV/480 V power transformer with an impedence of 8.0%. Four 480 V to 120/280 V transformers are distributed throughout the plant for lighting, HVAC, and other general uses.

- Power Distribution Cable and Bus Ducts - size and capacity.

- Bracing - all equipment must be braced for short circuit.

- Switch gear - with protective relaying and equipment to control and protect large motors and transformers.

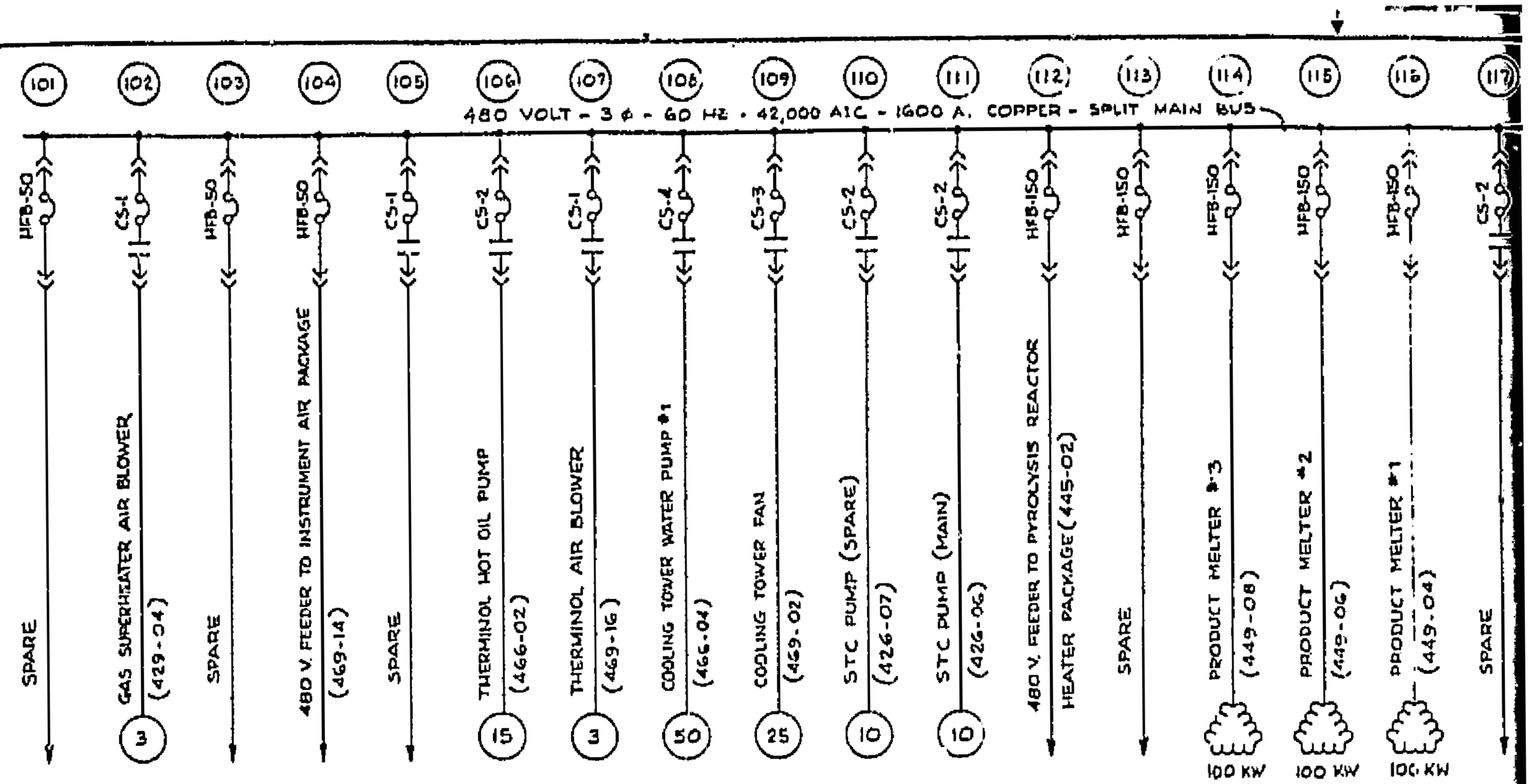
- Motor Control Center - with prot tive devices to control and protect smaller size equipment. EPSDU utilizes two motor control centers (MCC); however, MCC #2 is used to distribute power only to those items of equipment that must be powered during a power failure. A 300 kw generator is used for emergency power.

- Motors & Heaters - horsepower and kw. EPSDU includes 33 motors ranging in size from 1 HP to 75 HP and three 100 kw heaters.

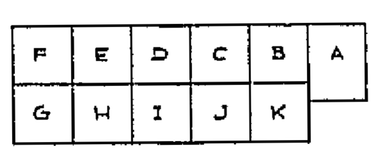
The one-line diagram serves as the basis for specifying the electrical equipment. Figures 1 and 2 (drawing D-2145548) show the electrical one-line diagram for the EPSDU project.

1.1.3.3 Process Equipment - UCC Designed

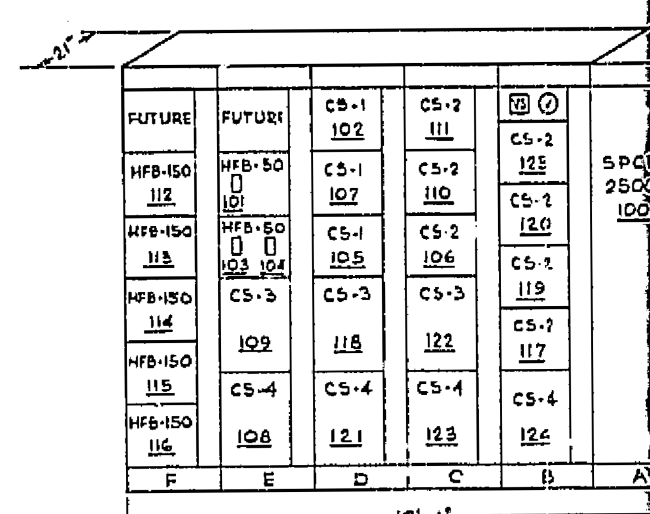
This includes the in-house effort necessary to develop and prepare the complete design for specialized process equipment such as the hydrogenation and free-space reactors and distillation columns.



480 VOLT MOTOR CONTROL CENTER #1 – SIDE A – NEMA 1



480 VOLT MOTOR CONTROL CENTER #1
FLOOR PLAN



480 VOLT MOTOR CONTROL CENTER #1
SIDE A – FRONT ELEVATION

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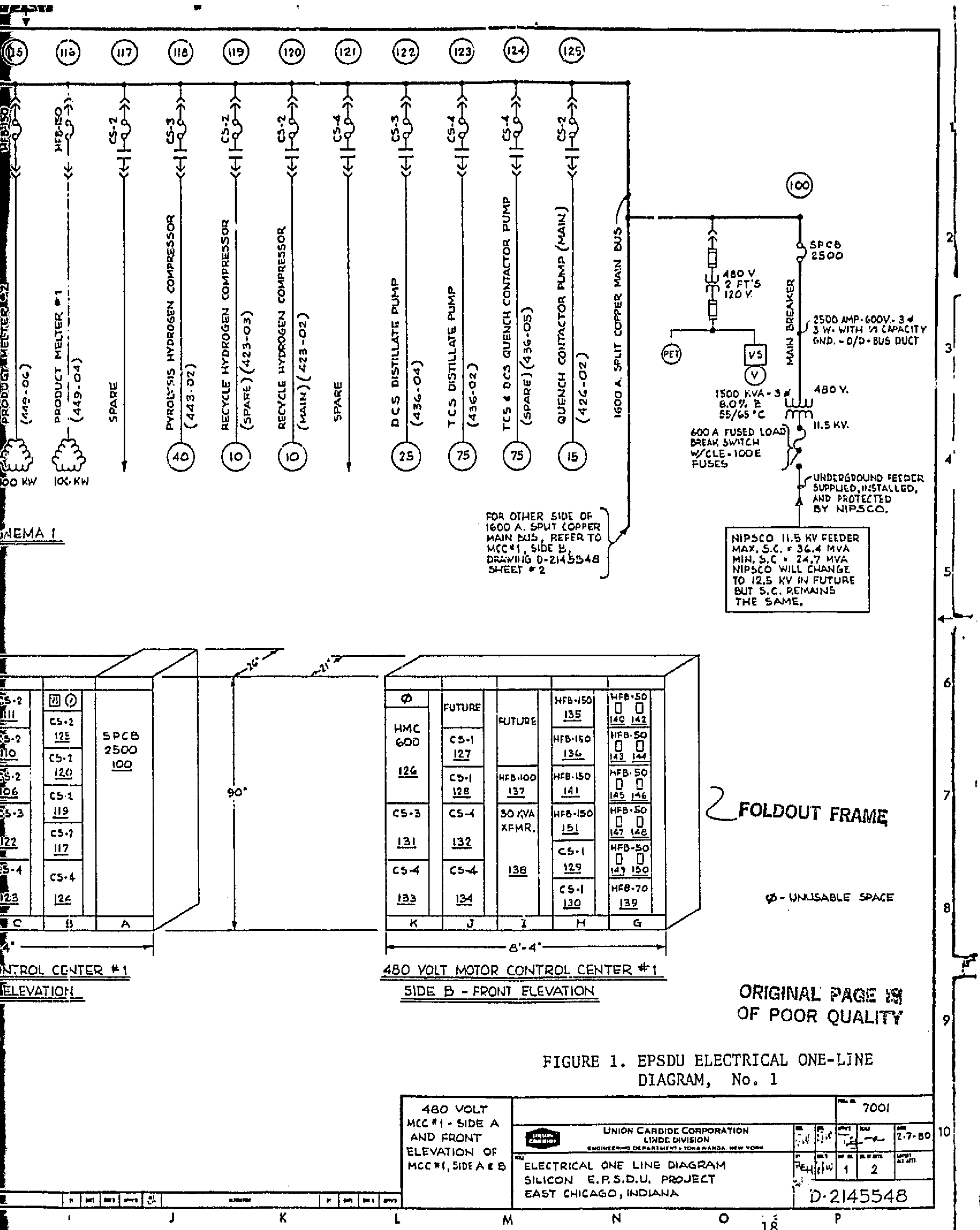
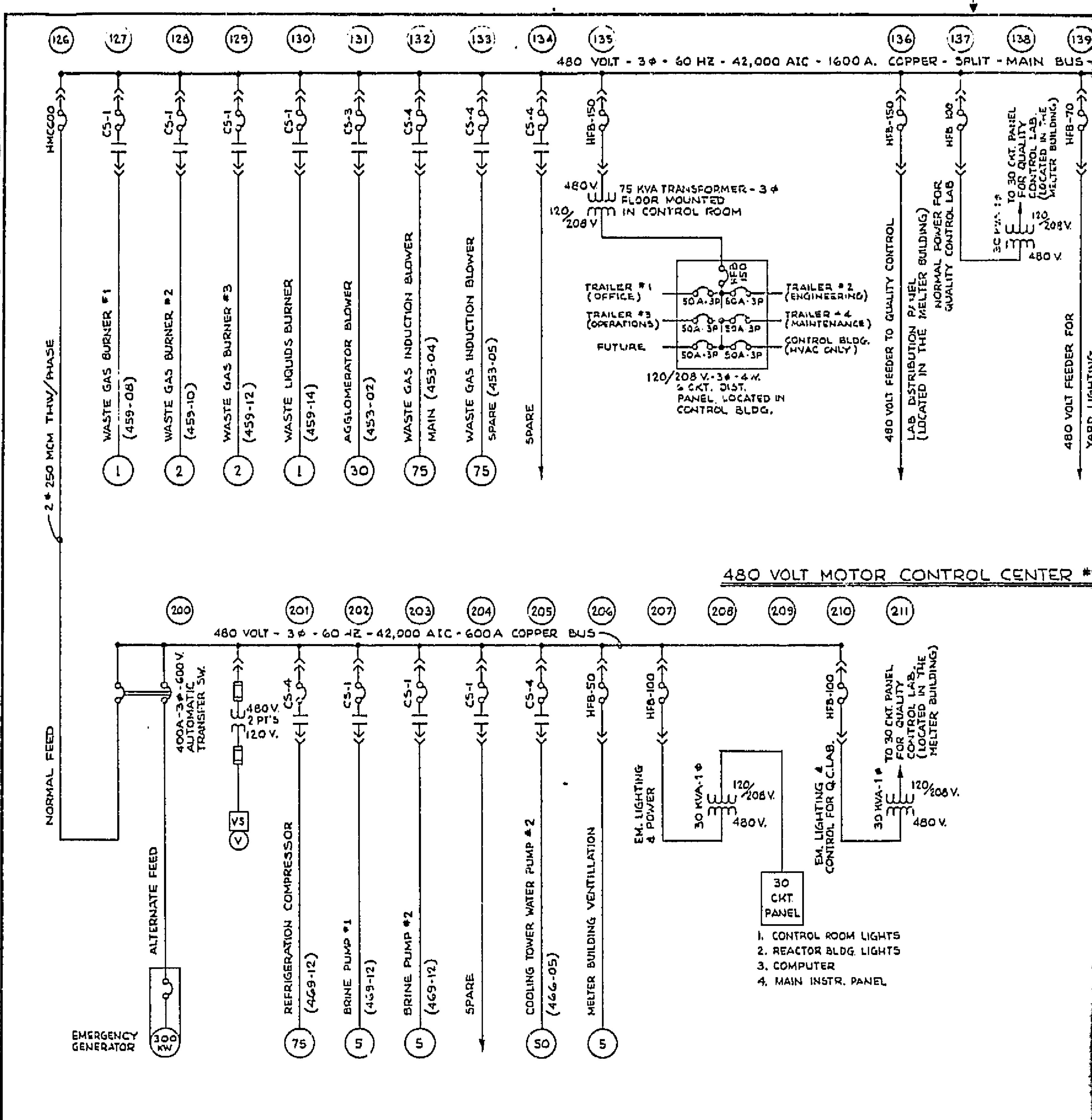


FIGURE 1. EPSDU ELECTRICAL ONE-LINE DIAGRAM, No. 1



480 VOLT MOTOR CONTROL CENTER #2 - NEMA 1

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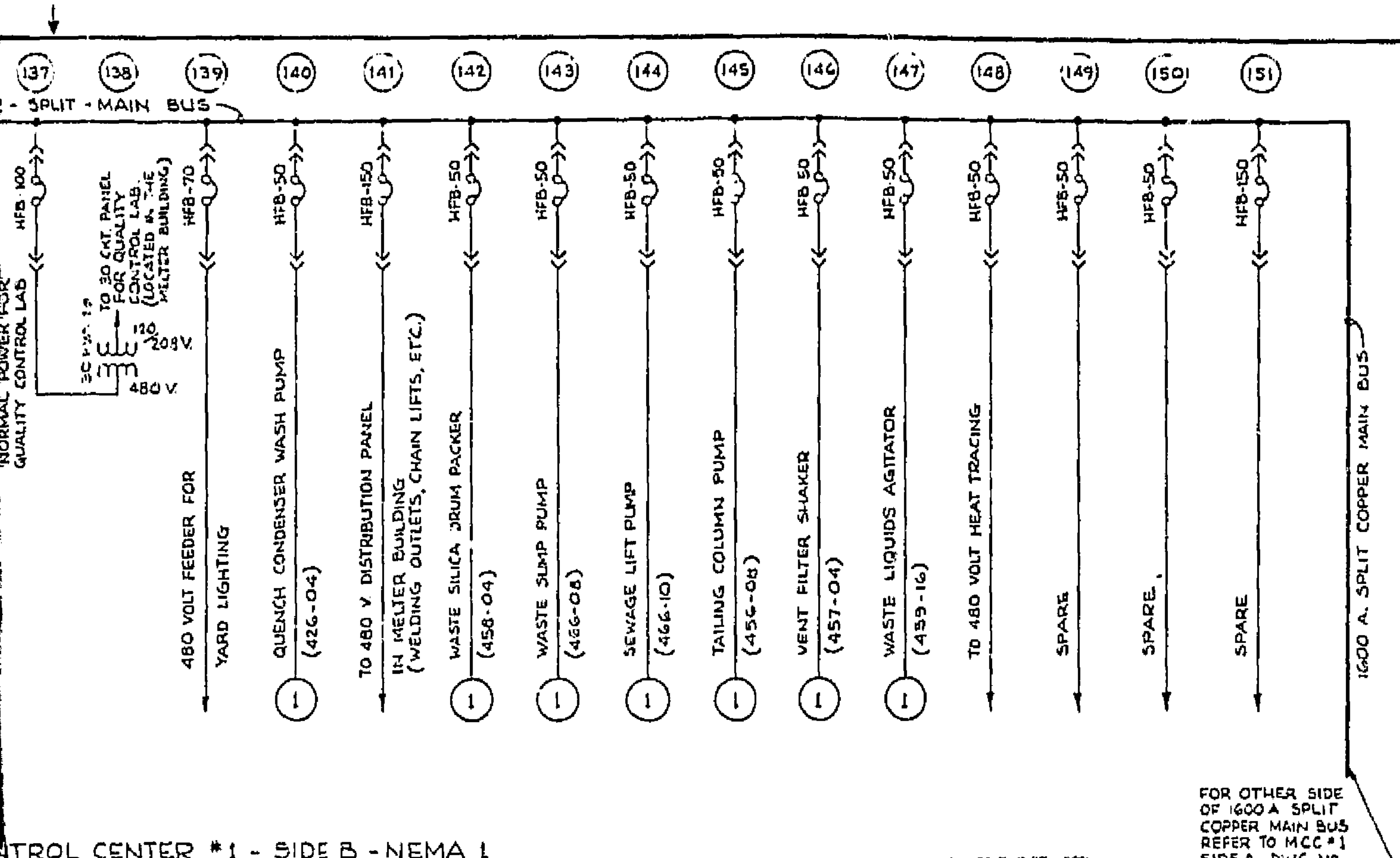

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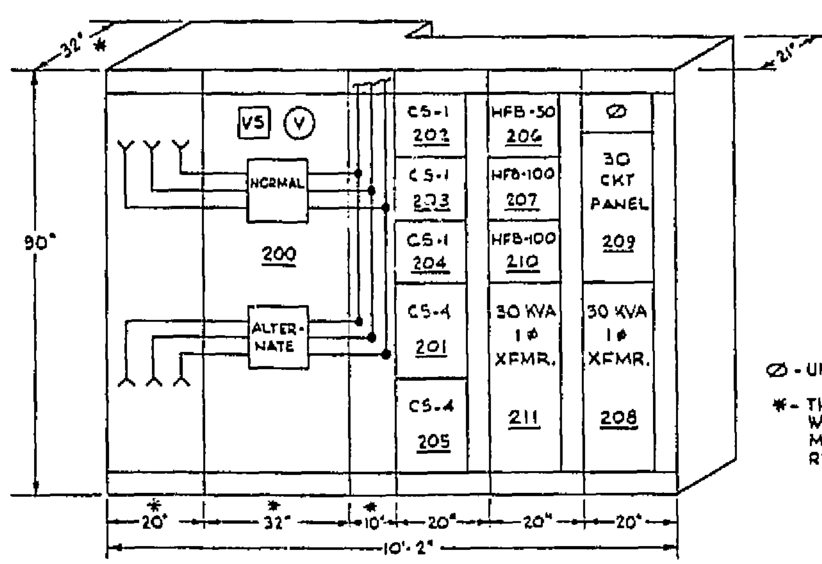

480 VOLT MOTOR CONTROL CENTER #2 FRONT ELEVATION

Ø - UNUSABLE SPACE

* - THESE DIMENSIONS WILL VARY WITH THE MANUFACTURERS REQUIREMENTS.

FOLDOUT FRAME

FIGURE 2 EPSDU ELECTRICAL ONE-LINE DIAGRAM, NO. 2

480 VOLT MCC #1 - SIDE B AND MCC #2, AND FRONT ELEVATION OF MCC #2

UNION CARBIDE CORPORATION
LINDE DIVISION

ELECTRICAL ONE LINE DIAGRAM
SILICON E.P.S.D.U. PROJECT
EAST CHICAGO, INDIANA

7001 — 2-11-80 — 2 — 2

D-2145548

I J K L M N O P

ASME code calculations, lifting/support lug designs, layout drawings, and welding procedures were completed for the stripper, TCS, and DCS distillation columns. The fabrication drawings are in work. Engineering design of the packed-bed silane column was started and RFR's were issued to the Linde/Tonawanda Fabrication Department for the stripper, TSC, and DCS columns.

1.1.3.4 Process Equipment - Vendor Designed

This activity includes the engineering effort associated with specifying and selecting process equipment such as compressors, pumps, and tanks that will be designed and fabricated by commercial suppliers.

Equipment cleaning procedure "SGS-1" was issued. This procedure, specific to fabricated equipment for the silane portion of the process (e.g. silane column, condenser, storage tanks), will be used by equipment suppliers as a final fabrication specification. For chlorosilane service up to the silane column, an existing specification (GS-40) was modified to cover all relevant equipment.

Process equipment specifications, RFQ's, and RFR's were issued for four silane tanks, twelve process tanks, two redistribution reactors, one hydrogenation reactor, three silicon bins, one process pump, one therminol pump, and two cooling-water pumps. Process equipment specifications and RFQ's were issued for twelve heat exchangers. The hydrogenation reactor, made of Incoloy 800-H, will cost approximately $100,000 and represents the most expensive purchase.

1.1.3.5 Auxiliary Equipment

This includes the engineering activities associated with specifying and selecting auxiliary equipment such as heating and refrigeration systems, instrument air unit, and cooling tower that will be designed and fabricated by commercial suppliers.

Equipment specifications and RFR's were issued for the cooling tower and Therminol® heater. An RFQ was issued for the cooling water treatment system. A proposal was received for leasing one TLH-9000 liquid-hydrogen storage tank and one TM-6000 liquid-argon storage tank.

1.1.3.6 Support Equipment

This effort includes the engineering activities associated with specifying and selecting commercially-supplied equipment such as trailers and materials-handling systems.

Work was started on the specification for the quality control laboratory. The initial bid package was revised after discussions with several potential vendors as to their capabilities and recommended practices. The RFQ for the modular laboratory will be issued soon for a unit 56 feet long x 12 feet wide equipped with appropriate work surfaces, cabinets, fume hoods, and electrical and plumbing fixtures. The sample line termination panel will be bid separately as will the analytical equipment. All components will be shipped to the UCC East Chicago facility for assembly and checkout prior to final connections at the adjacent EPSDU site. This flow of work should minimize overall costs. Numerous vendor contracts were made to help select laboratory furnishings which are appropriate for the type of work to be performed and the short-term nature of the facility.

A preliminary layout of the sample line termination panel has been developed. The panel face, 5 feet high x 6 feet wide, will allow display of sample flow, pressure, and manual sample-stream selection for the 47 process sample points. The panel will be designed to be maintained at 165°F to assure that all samples remain in the vapor phase for chromatographic analysis. Special consideration was given to safety and environmental concerns. A unique panel face ventillation arrangement was designed to vent any small leaks directly outdoors, and sandwich-type-construction insulation will keep the panel face cool while maintaining 165°F inside. The detailed panel layout drawings and bill of materials will be prepared for the bid package.

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1.1.3.7 Other Equipment

The silane free-space reactor and the melting/consolidating system are being developed as part of the Supporting R&D and are described under Tasks 1.6.1 and 1.6.2, respectively.

1.1.3.8 Data Collection System

This work item covers all effort required for tailoring and specifying a computerized data-collection system for EPSDU.

A meeting was held to review planned activities. Work scheduled to start in February will be deferred until May, 1980, to permit completion of additional work on the Process Control System. The main data-acquisition computer will be purchased in June 1980 for delivery to Tonawanda in January 1981 for checkout prior to reshipment to the EPSDU site. Specification and ordering of input/output equipment and peripheral equipment may be deferr'd until early 1981. Designs for these items are being continually refined by suppliers, and ordering too early might result in less cost-effective equipment.

1.1.4 Installation Design, Specification, Subcontract

This design effort includes development of separate installation drawing packages for the site, civil, mechanical, and electrical specialties based on the engineering design effort and vendor-supplied information. Specification activity includes definition of specific requirements for performing all installation functions. Subcontracting includes the preparation of bid packages, evaluation of quotes, subcontractor selection and contract negotiation.

1.1.4.1 Site Drawing & Specs

This work item includes preparation of all drawings and specs necessary for rough grading, establishing drainage patterns, installing fencing, providing base for parking lots and roads, and, if necessary, obtaining temporary construction utilities.

Preliminary work has been started on the site drawings.

1.1.5 Cost Estimating

Cost engineers support the design effort by providing cost estimates and controls for procuring equipment and installing the EPSDU.

1.1.5.1 Facility Cost Estimate

The major task was to prepare a detailed cost estimate from information generated during the engineering design and equipment specification activities for use in budgeting and monitoring and controlling performance.

The detailed facility cost estimate was prepared based on the design definition for EPSDU developed during the June-December 1979 "pre-engineering" phase. The cost estimate will undergo further refinement as detailed engineering and equipment purchasing occur under WBS items 1.1,1.2 and 1.3.

Costs associated with Design/Procurement and Equipment Fabrication/Delivery agree quite well with the baseline estimates. However, the cost associated with Installation/Check-out increased almost 30 percent from the baseline cost. Most of these costs are for the construction sub-contracts, and an accurate cost will not be known until the sub-contract bids are returned in the fall of 1980.

1.1.5.2 Engineering Design Support

Cost engineering also provides support for individual design efforts, monitors costs, and prepares periodic cost evaluations of engineering design activities.

A "first-round" cost improvement review was initiated to identify the cost impact of all facility design changes that have occurred since the design was baselined in December, 1979. Substantial cost reductions in several areas are expected.

1.2 EQUIPMENT FABRICATION/DELIVERY

This report item includes all in-house and outside activity associated with fabrication, delivery, and vendor coordination for all items of equipment.

Appropriate JPL contract pass-down provisions, Union Carbide procurement provisions, and equipment cleanliness specifications have been prepared. Some purchase orders have been issued, but the bulk of the procurement activities is scheduled for the next quarter. The equipment procurement status is presented in Appendix B.

1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS

This report item includes all process engineering, design, and estimating activities necessary to provide a detailed baseline investment, operating, and product cost for a 1000 MT/yr commercial facility based on EPSDU engineering design information, and to prepare subsequent updates based on information from supporting R&D and EPSDU operation.

1.5.1 Process Design

This effort consists of preparing a process design package for a 1000 MT/yr facility optimized for commercial production.

1.5.1.1 Functional Package - Baseline

The initial process design package is based on data available from the EPSDU design effort. This work has been started and will parallel the remaining EPSDU design effort.

An examination of potential process improvements for the large facility was started with the goal of reducing product silicon cost. One beneficial improvement was the reduction in DCS column pressure from 320 psia to about 170 psia. Another improvement was in the interchanging of process heat from the quench condenser to either partially boil TCS in the TCS reboiler or preheat the STC recycle to hydrogenation. While the potential for cost improvement is better for the TCS reboiler approach, it introduces some possible instabilities. Therefore, preheating of the STC recycle will be used.

1.6 PROCESS SUPPORT R & D

The supporting R & D program is separate from the mainstream design effort and includes all activities associated with analytical and experimental development of the free-space reactor, melting/consolidation system, fluid-bed reactor system, and quality control techniques and procedures. Information generated on this program will be used for the EPSDU effort and the commercial facility economic analysis.

1.6.1 Free-Space Reactor Development

This development includes all experiments and analysis necessary to verify design data for the free-space pyrolysis reactor and to develop a new reactor model.

1.6.1.1 PDU Experiments

The process development unit was originally installed at UCC Parma and was used during the Phases I and II program. It was transferred to Tonawanda, modified, installed, and is being used to conduct experimental tests to demonstrate steady-state operation and provide design data.

The free-space reactor PDU was assembled and a 1-hour shakedown run at 1.2 lb/hr was made. Free-flowing dark-grey powder with a bulk density of 4.7 lb/ft^3 was produced.

Experience at Parma had indicated that a mechanical scraper was required to prevent plugging of the reactor by powder bridges. The initial scraper design for the Tonawanda PDU performed poorly during pre-startup tests and a number of modifications were made to improve operation. These changes had not been implemented at the time of the shakedown run and, as a result, the scraper was not used.

During flow calibration and startup, diaphragm leaks occured in both silane regulators. The manufacturer has not been able to explain these incidents; however, it seems likely that the regulators were not designed to cope with the intense Joule-Thompson cooling effect of large silane flows. Several major changes have been made to improve regulator reliability:

- Linde single-stage silane regulators have been installed. These offer several design advantages over the Matheson high-purity regulators used initially, but were not available until recently.

- A preheater has been fabricated and installed to raise the minimum temperature experienced by the regulators.

- The silane purge system has been completely revamped to eliminate dead-ended sections of tubing.

These modifications eliminated the silane regulator problem.

After these improvements were made, and the scraper was installed, two experimental runs were conducted with the free-space reactor PDU. The first run lasted approximately 8 hours and the second 24 hours. The 24-hour run marks the fulfillment of the first major milestone for the PDU work. A brief summary of the two experimental runs is given below.

The first run, on March 4, 1980, proceeded smoothly for the first five hours of operation. A thermal conductivity cell was used to aid in determinimg off-gas composition. Although a long "tail" of argon in the off-gas made interpretation of the data difficult, qualitative observation of the flame color of the vent from the conductivity cell showed a very high conversion efficiency (>99%). This conclusion is based on qualitative criteria developed previously in Parma during the Phase I work. Run conditions were as follows:

Silane flow:	4.96 lbm/hr
Wall temperature:	$1560^{o}F$ ($850^{o}C$)
Pressure:	6.0 psig

After five hours, scraper operation became erratic and slow. Air pressure to the actuator was increased to the maximum available (100 psig), but after 7.5 hours the scraper jammed in an upstroke position. All attempts to free the scraper by remote control failed and the run was terminated. Approximately 0.5 hours later, when the reactor had cooled to $1200^{o}F$, the scraper suddenly freed itself and moved to the top of the reactor.

Attempst to convey the product powder from the hopper into 55-gallon drums were hindered by the poor performance of the initial drumming system. The system was restructured and conveying was resumed with only fair success. Examination of powder removed from the system revealed numerous pieces of quartz. Inspection of the upper section of the reactor, using a light-pipe inserted through the spare scraper port, revealed that the quartz reactor liner was shattered and that pieces of quartz were probably blocking the outlet of the fluidizing cone. The manway was then opened and the remaining powder was vacuumed into a fifty-five gallon drum. Powder remaining after vacuuming was flushed from the reactor and sent to the sewer. The bulk density of silicon powder from this run was 0.26 gm/cc.

Following a thorough inspection of the reactor and pieces of the broken liner, it was concluded that the scraper jam had occurred primarily because the silane had been turned off each time the scraper was used. This probably disrupted the flow pattern in the reactor and could have led to excessive silicon deposition on the liner walls. This caused the formation of mechanically strong "glued" powder deposits that the scraper could not remove. The liner probably broke when the scraper finally jumped free and slammed against the reactor head.

The scraper was redesigned to closely model the unit used in Parma. The specific modifications were as follows:

- Replacement of the Grafoil packing with high-purity teflon rings. The Grafoil was apparently corroding the scraper shaft and did not appear to be compatible with the bearing material used in the lantern ring. The new packing greatly reduced friction in the stuffing box.

- Installation of a modified lantern ring with filled teflon sleeve bearings press-fit on the inside diameter. This helped to locate the shaft and eliminate the alignment and galling problems encountered during January tests.

- Re-machining of the scraper outside diameter to reduce interference with the reactor lining. Boroscope examination of the reactor through the unused scraper port revealed several points of interference.

- Addition of side braces to increase the rigidity of the air cylinder. This helped to reduce side loads on the shaft and lantern ring/bearing.

The reactor was bored out to accommodate the new liners which were somewhat larger than the original, and the reactor was reassembled.

The second run, on March 20, ran continuously for the scheduled 24 hours. Two problems were encountered. Failure of a square root extractor and partial blockage of the orifice valve crippled the silane flow-control system early in the run. This limited the flow to 4.3 lbm/hr, slightly below the original target level of 5.0 lbm/hr. After 11 hours of operation, the scraper jammed; however, it was eventually freed by reversing its direction several times. A recurrance, 22 hours into the run, was also overcome. The conditions for the run were:

Silane flow:	4.3 lbm/hr
Temperature:	1600°F (870°C)
Pressure:	6.0 psig

The on-line chromatograph was operational throughout the run and gave excellent separation of the silane and inert peaks. Silane conversion as measured on the chromatograph was over 99.7%. The bulk density of the silicon product powder from the 24-hour run was 0.27 gm/sec.

Although the condition of the reactor liner and scraper after the long run has not yet been examined, successful operation of the PDU for 24 hours demonstrates that sustained operation at high silane feed rates is feasible.

1.6.1.2 Theoretical Analysis

The computer model of the free-space reactor has been modified to include the effects of compressibility of the silane gas. Silane is treated as an ideal gas whose density depends on temperature and pressure. This couples the conservation equations for momentum with the energy equation, and makes solution of the system of equations much more difficult. Substantial under-relaxation of the coefficients in the finite difference equations must be used to prevent divergence of the computer solution. This is a result of the way the numerical scheme corrects the velocity and pressure fields to satisfy continuity. As a result, convergence of the solution is extremely slow. Recent literature reports that thousands of iterations requiring one hour of computer time (representing a cost of roughly $1200 on the IBM 370) have been required to obtain a converged result for problems involving compressible, recirculating, chemically reacting flows. Although work is currently underway to sharply reduce run time, it appears unlikely that converged solutions for compressible flow in a free-space reactor simulation with R/d $\sim$20 will be obtainable with the current (TEACHT based) program due to excessive cost. Convergence of the solution is much faster if the density is held constant.

Convergent solutions to the system of equations describing the free-space reactor have finally been achieved for an R/d ratio equal to 20. Constant density flow had to be assumed to keep the cost of running the computer program from becoming prohibitively expensive. Initially, a 22 x 22 grid was used, but the solutions generated were not at all grid independent. Altering the spacing of the grid points caused considerable change in the solution. Currently a 30 x 30 grid system is being used. Solutions for this number of grid points are less sensitive to changes in the grid spacing, and the results obtained (particularly for the reattachment length) appear more physically reasonable. Since a run of the program with a 30 x 30 grid costs about $70 and the run-cost increases more than proportionately to an increase in the number of grid points, using a finer grid doesn't seem worthwhile. Little increase in accuracy could be expected, but the run-cost would increase dramatically.

The degree of silane conversion predicted by the model is strongly affected by the reaction rate constants and the value of the heat capacity chosen to be representative of the silane-hydrogen-silicon dust mixture. Simulation runs indicate that the rate constants reported by Joyce and Bradley appear to be reasonable. The heat capacity of hydrogen is roughly an order of magnitude higher than the heat capacity of silane or silicon. Since the convection and reaction terms are the most important in the energy equation, the heat capacity value largely determines the temperature increase given a certain amount of heat release by the chemical reaction. Using the heat capacity of silane in the model yields high temperatures and overpredicts the reaction conversion. Using the heat capacity of hydrogen yields low temperatures and underpredicts reaction conversion. Following the suggestion of Galloway, the recirculating fluid is assumed to be a stoichiometric mixture of hydrogen and silicon dust. The heat capacity of such a mixture appears representative of what should actually exist in the reactor, and simulation runs made with such a value show good agreement with experimental results. With R/d = 20 and a silane throughput of 5 lbm/hr, runs were made at wall temperatures of 800°K, 1000°K, and 1200°K. With a wall temperature of 800°K, the silane passed through the reactor completely unreacted. At 1000°K, the reaction took place about half-way down the reactor and went to completion, but high concentrations of silane were present along the reactor wall in the top half of the reactor. At 1200°K, the reaction took place very near the jet nozzle and also went to completion. Deposition on the reactor nozzle is a strong possibility under these conditions. Runs made at R/d = 10 indicated that increasing the silane throughput should have little effect on the reaction conversion provided the wall temperature is high enough. If this proves to be true, a reactor of the present size may be capable of much higher throughputs than previously anticipated.

Increased fluid recirculation appears effective in promoting greater particle growth. The model predicts increased recirculation at high throughputs. When the silane is injected upward into the bottom of the reactor, simulating the Parma operating conditions, larger particle sizes are predicted. The model confirms that powder buildup on the reactor walls is due to inertial impaction of particles unable to follow the fluid streamlines in regions of strongly turning flow. Operating the reactor at higher pressures increases the silane concentrations and leads to more rapid completion of the reaction. The heat released by the pyrolysis reaction is primarily responsible for heating the incoming silane to the reacting temperature. Conversion is poor upon startup because the incoming silane is heated solely by the reactor wall until the reaction becomes established.

Future work on the free-space reactor model should concentrate on investigating the influence of the injector position on the flow field and reaction conversion, and on incorporating particle effects into the model to provide a better capability for analyzing particle growth and powder build-up. Once the maximum throughput of the experimental reactor has been determined, the rate constants in the model should be adjusted to match the model prediction to the experimental result. The model can then be used to size the proposed EPSDU reactor.

1.6.2 Melting/Consolidation

The design and development effort necessary to obtain a reliable melter for EPSDU involves UCC and sub-contractor effort.

1.6.2.1 Melter System Subcontract

The in-house effort involves establishing and managing the sub-contract and sub-contractor development, design, and fabrication efforts.

A sub-contract was signed with Kayex Corporation for the development of a silicon melter system for EPSDU. The silicon consolidation scheme is based on melting the powder in a quartz crucible and dropping molten silicon shot from the crucible bottom into a cooling tower where the shot is solidified.

Sub-contract work was started on March 1, 1980 and will be completed in August 1981. The goal of the project is to design and build a melting/consolidating system suitable for installation in the EPSDU. Kayex spent the first month developing a program plan and calculating the approximate tower height required to solidify silicon shot.

1.6.2.2 Process Analysis

UCC is also performing consolidation process analyses geared toward supporting the melter sub-contract effort and EPSDU design activities.

A theoretical model for silicon shot cooling was developed at UCC and the results were discussed with Kayex personnel in Tonawanda on March 26.

Several aspects of shot casting were investigated. The process of droplet formation through an orifice by the formation of individual drops and by the breakup of a liquid jet were investigated to determine how to control shot size and the rate of shot production. The solidification of a spherical droplet falling in a cooling tower was studied to predict the height of the cooling tower required to completely solidify the droplets formed. The dynamic response of the shotting system to changes in the various design and control variables was investigated to determine the best means of controlling and monitoring the system operation. The requirements of the cooling system for the shotting tower were also calculated.

Calculations show that shot larger than 2 mm in diameter cannot be completely solidified in a 20-foot high cooling tower. Shot of the desired size (1-2 mm diameter) can be formed at rates comparable to the proposed EPSDU throughput by forcing molten silicon through a 1 mm orifice at velocities corresponding to the varicose region, where drops form by jet breakup. Droplets formed at low flow velocities through orifices of reasonable size are too large to solidify completely. Hydrogen or helium should be used as the cooling gas in the shotting tower, since their high thermal conductivities enhance convective heat transfer significantly and result in a faster solidification of the droplets.

The controllability of the shotting system depends on the ratio of the area of the crucible bottom to the cross-sectional area of the orifice. Use of a crucible with a small diameter increases the sensitivity of the melt height and flow velocity through the nozzle to changes in the powder feed rate, pressure differential across the melt, and orifice diameter. The possible problem of orifice plugging or erosion can be most effectively monitored by observing the shot size and throughput through the system. The cooling requirements for the shotting tower are minimal.

The results obtained from the theoretical model will be used by Kayex to guide preliminary designs for the shotting system. The analysis suggests that experiments should be conducted to determine the severity of the problems of orifice plugging or erosion, and to determine the ability of partially solidified droplets to survive impact at the bottom of the tower. Liquid silicon expands as it solidifies. Since the droplet expands from the outside surface inward, significant thermal stresses can develop that could fracture the droplet. These stresses are functions of the diameter of the droplet and the rate of solification. In addition, the partially solidified droplet may fracture upon impact at the bottom of the tower if the shell is not thick enough to withstand the shock. Attempts to predict the criterion for failure of the shell are complicated by the lack of information on the mechanical properties of silicon at high temperatures. This problem must also be resolved experimentally.

1.6.3 Fluid-Bed Development

This development program includes all analytical, experimental, and design effort associated with developing a fluid-bed reactor as an alternative or backup system to the free-space reactor.

1.6.3.1 Fixed-Bed Deposition

A special apparatus was designed, fabricated, and used to perform fixed-bed deposition experiments. This program was successfully completed and the test data generated will be used in designing the fluid-bed silane pyrolysis PDU. Tests with a fixed-bed, instead of a fluid bed, resulted in obtaining the critical silane concentration curve and the deposition-rate data quickly and inexpensively. The specific objectives of the fixed-bed experiments were:

a. To determine heterogeneous decomposition rate of silane.

b. To define bed temperatures and silane feed concentrations which favor heterogeneous deposition and minimize homogeneous decomposition.

c. To qualitatively describe deposition morphology.

The specific objectives of the fixed-bed experiments were all studied. The deposition rate measurements agreed favorably with literature. Homogeneous decomposition was minimized by determining the dependency of decomposition on silane concentration and temperature. The conclusion of the growth morphology was that a strong solid bond could be achieved.

Rate measurements showed the decomposition of silane to be a first-order irreversible reaction. The rate measurements also showed no composition or velocity dependency in the reaction within the range of data collected. The results of the kinetic rate study compared well with literature, especially with the work of Eversteijn[(1)] as follows:

[(1)] Eversteijn, F. C. and Put, D. M., J. Electrochem. Soc.: Solid State Science and Technology, 120, 103 (1973)

Temperature (^{o}C)	Reaction Rate Constant (Min^{-1}) Eversteijn's Curve Fit	This Work
576	0.012	0.010
627	0.040	0.026
676	0.096	0.112

A further comparison shows the activiation energy for heterogeneous decomposition to be 37 kcal/mole from Eversteijn's rate study and 39 kcal/mole from this kinetic rate study.

The dependency of silane homogeneous or heterogeneous decomposition on the concentration of silane and the temperature is described as the critical silane concentration curve (Figure 3). The distinction between homogeneous and heterogeneous decomposition, which is presented as a band instead of a definitive line because of the selection of the temperatures, seems to be an extention of Murthy's [(1)] data. At 20% silane concentration, the temperature band is 650 to 670^{o}C. At 10% silane, the temperature band is 700 to 750^{o}C, and at 1% silane, the temperature band is 820 to 895^{o}C. These results were determined with a packed bed of high-purity unetched silicon particles. If the silicon particles are washed with an acid and then etched, the results change. The heterogeneous decomposition region was lowered by approximately 50^{o}C (Figure 4). The critical silane concentration curve for etched high-purity silicon lies somewhere between the data of Eversteijn[(2)] and Murthy.

Heterogeneous decomposition using unetched silicon particles in the bed resulted in nodular and fiber silicon growth with the dominant type being fibrous (Figure 5). Use of acid-washed and etched silicon particles in the bed strongly reduced fiber growth (Figure 6). The silicon deposits on

[(1)] Murthy, T.V.M.S., et al, J. Crystal Growth 33, 1 (1976).

[(2)] Eversteijn, F.C., Philips Res. Repts. 26, 134 (1971).

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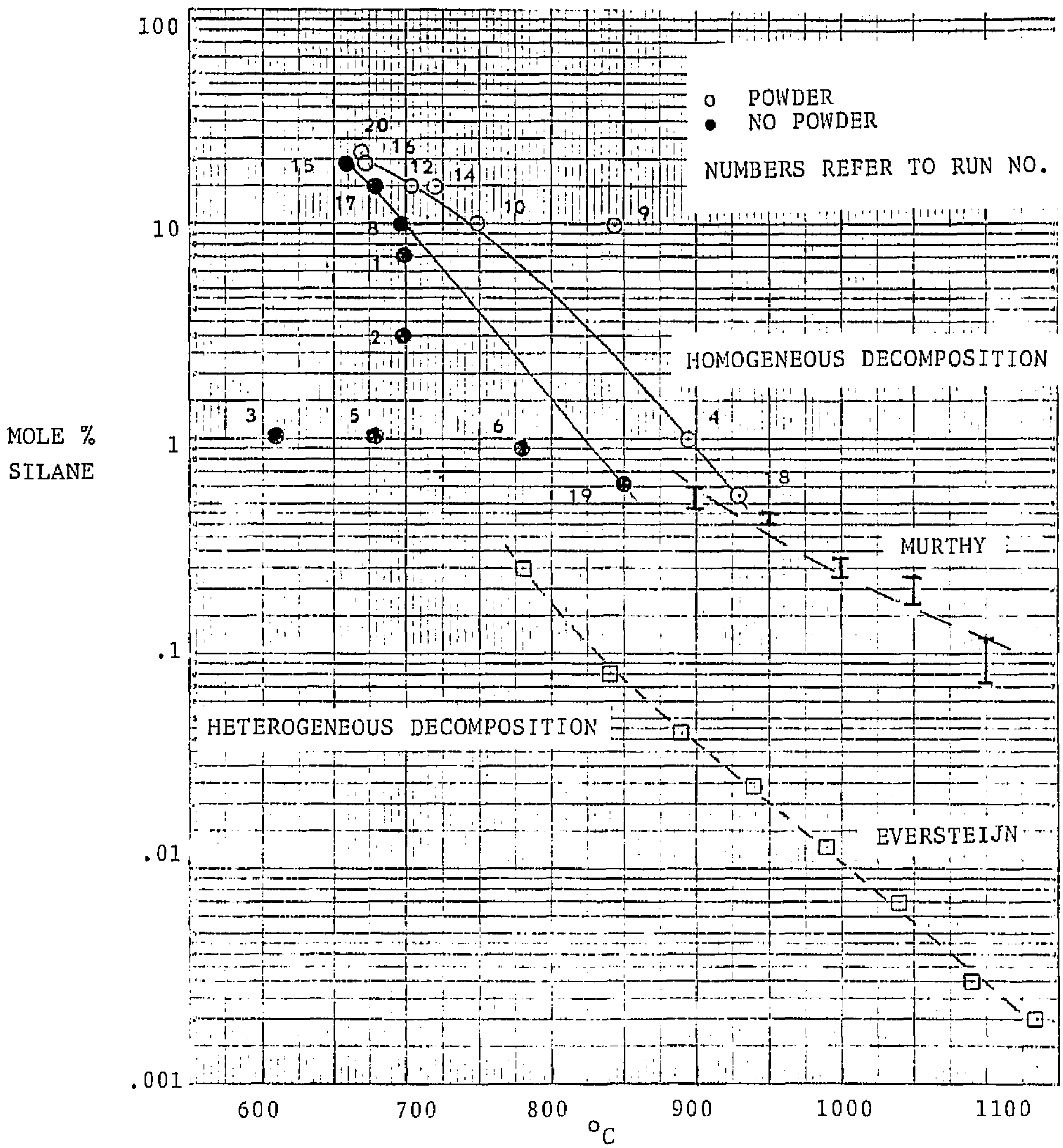


FIGURE 3. TEMPERATURE DEPENDENCE OF CRITICAL SILANE CONCENTRATION FOR PACKED BED OF HIGH-PURITY UNETCHED SILICON.

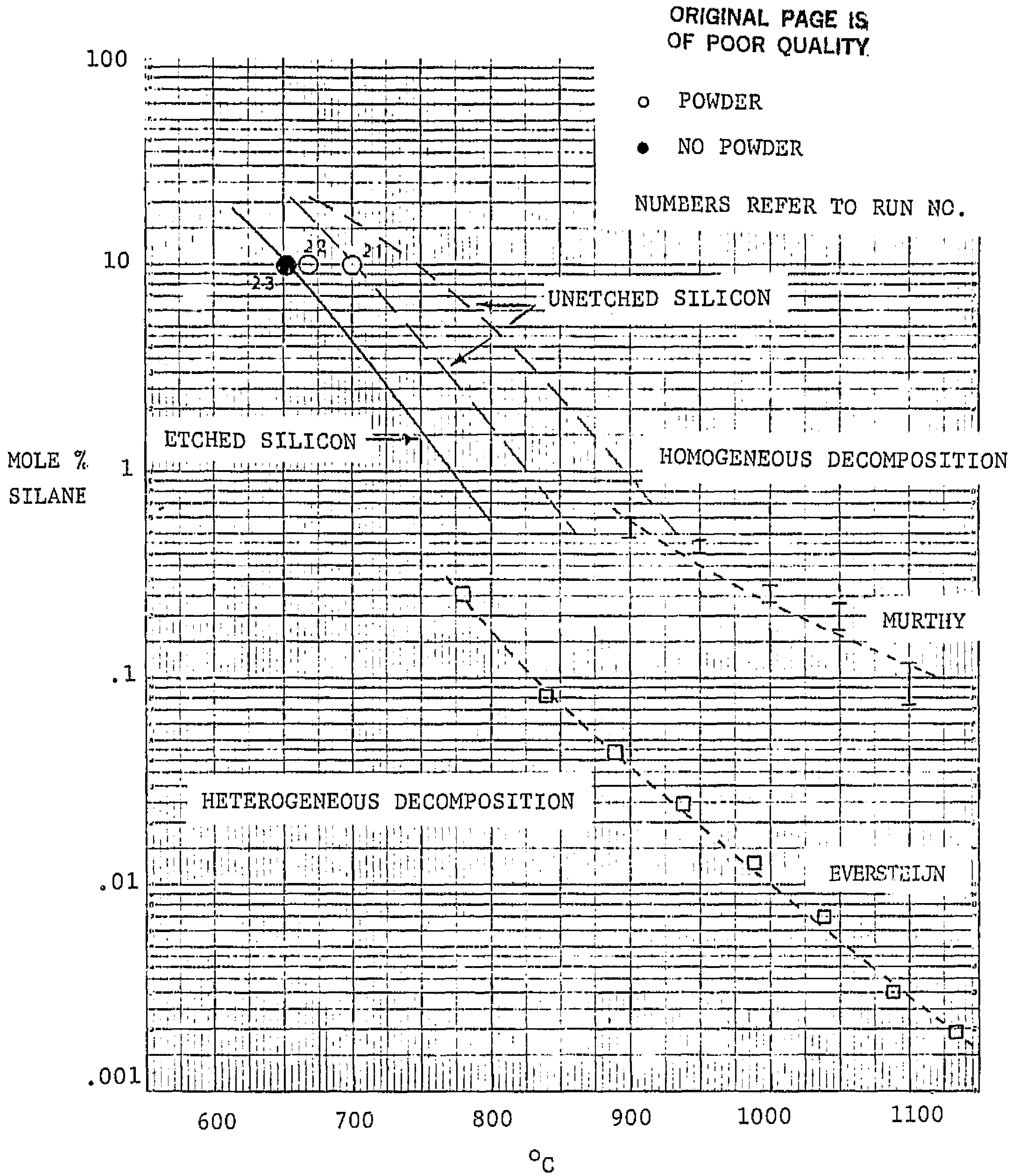
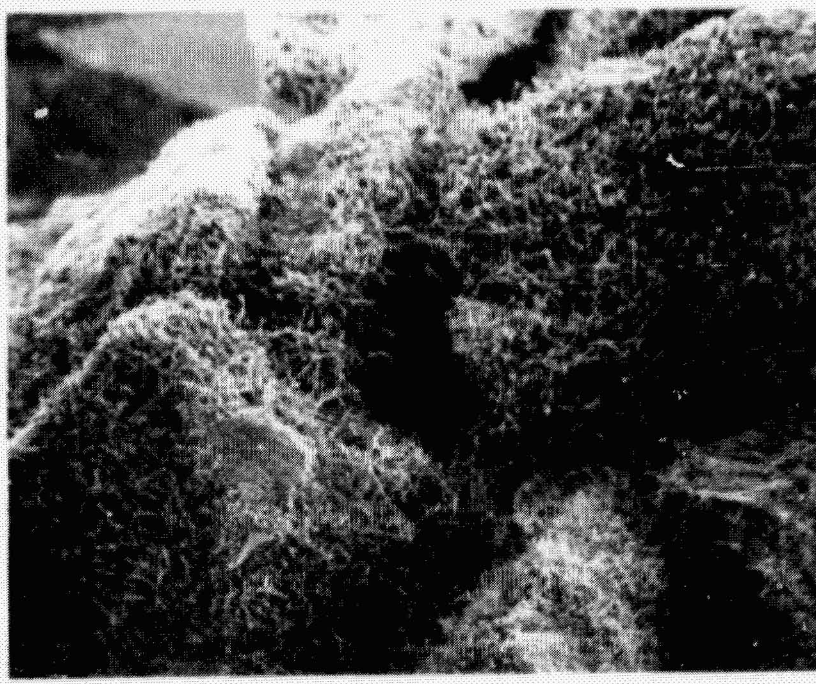


FIGURE 4. TEMPERATURE DEPENDENCE OF CRITICAL SILANE CONCENTRATION FOR PACKED BED OF ETCHED HIGH-PURITY SILICON



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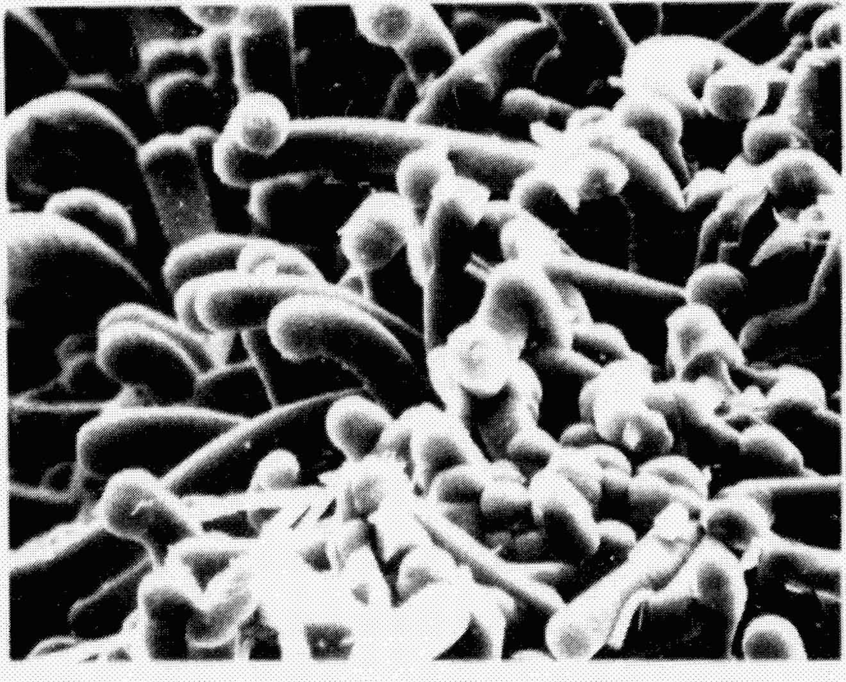


700X

FIGURE 5. SCANNING ELECTRON PHOTOMICROGRAPH OF HIGH-PURITY UNETCHED SILICON PARTICLE SURFACE AFTER HETEROGENEOUS SILANE DECOMPOSITION, RUN 8, 9.8% SILANE, 696°C.

500X
Run 23

500X
Run 23

FIGURE 6. SCANNING ELECTRON PHOTOMICROGRAPH OF CROSS-SECTIONED ETCHED HIGH-PURITY SILICON PARTICLE SURFACE AFTER HETEROGENEOUS SILANE DECOMPOSITION RUN 23, 9.8% SILANE AND 652°C.

the etched particles were dense, coherent, and well attached to the particle surface. Analysis of the acid-washed and etched silicon particle surface indicated a large reduction of metal contamination and removal of surface residue. These factors must be contributing to the favorable reduction of surface fiber growth.

The fixed-bed experiments supplied valuable information for the design of the first-generation fluid-bed silane pyrolysis development unit. Important information was gathered in the areas of silane decomposition rates, heterogeneous versus homogeneous silane decomposition, and the morphology of heterogeneous decomposition.

Initially, the fixed bed was difficult to operate properly. However, after stable temperature equilibrium was established throughout the bed and other operational techniques improved, reliable data was obtained. Acid-washing and etching of the high-purity silicon particles definitely affected the results of the critical silane concentration curve (heterogeneous versus homogeneous decomposition) and probably the rate data. It is uncertain if acid-washing without etching is sufficient to accomplish the same results.

It is recommended that all further fluid-bed work be performed with high-purity silicon that is acid-washed and etched <u>after</u> the silicon is ground and screened. This procedure should also be standardized and documented so that further experimental results gathered can be compared on a common basis.

The results of the fixed-bed experiments indicate that a fluid-bed silane pyrolysis reactor can operate at 10% silane and 650°C. This is an excellent operating point because:

a. The reaction rates are high at this temperature and complete silane conversion can be realized in a small fluid bed.

b. Hydrogen requirements are reasonable at these temperatures.

c. The high silane concentration makes fluid-bed operation economical.

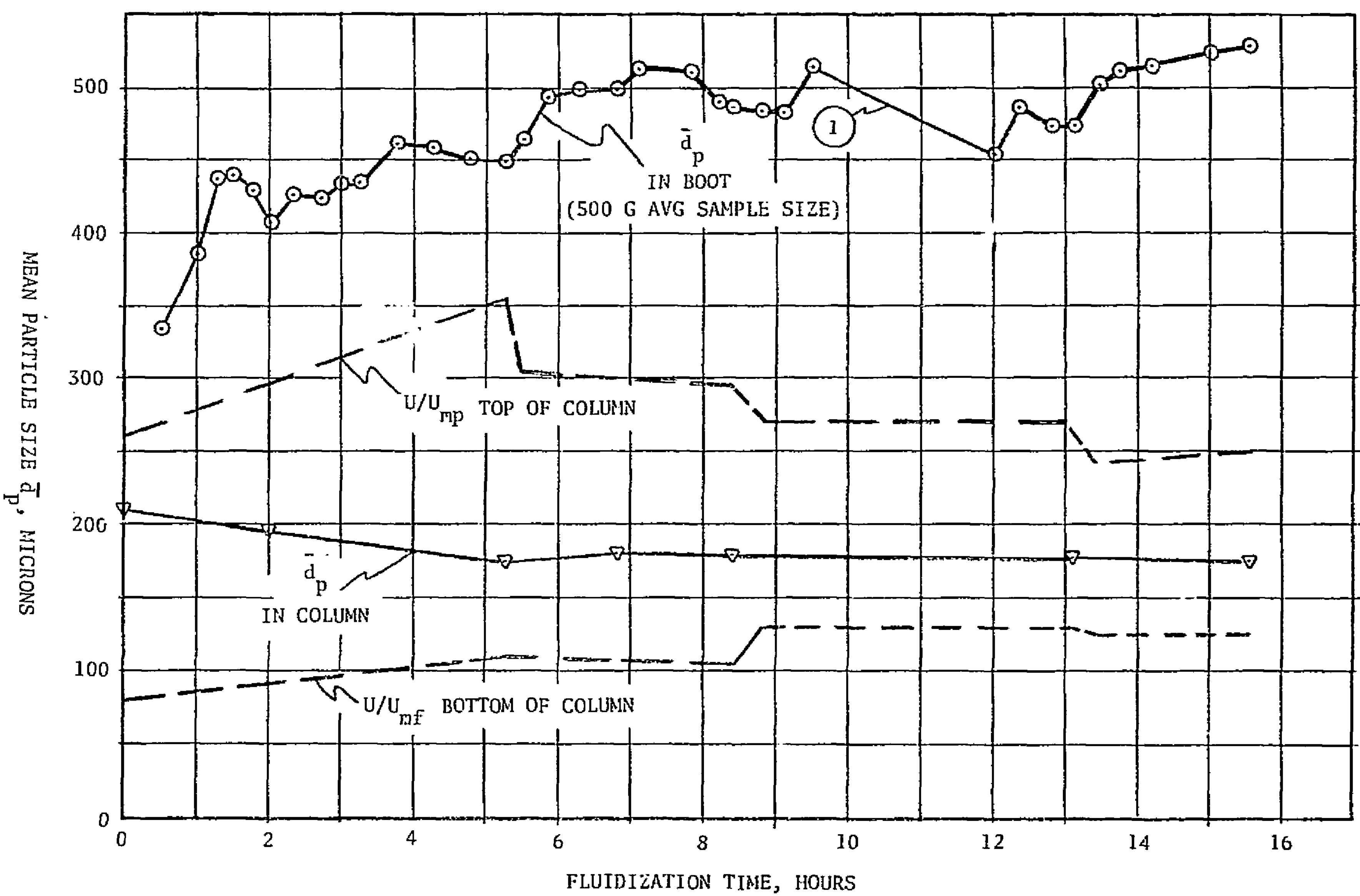
1.6.3.2 Fluidization Experiments With Inert Gas

This test program, started in 1979, consists of particle separation tests and capacitive heating tests. The former are being conducted in a special 6-inch pyrex reactor, and the latter in a 2-inch stainless steel reactor.

Particle Separation Tests:

The 1½-inch diameter plastic boot and the vacuum hopper were installed on the 6-inch diameter pyrex column. Test No. 13 was run using a bed of wide particle-size distribution at 1/6 atmosphere of helium. For this test, after every other boot sample was extracted, silicon was added to the column using the vacuum hopper. This maintained the bed at a constant height over the test period. By observing the fluidization in the boot, operating characteristics of the system have been determined. The results presented in Figure 7 show that continuous separation, at high boot/column diameter ratios varying from a minimum of 2 to a maximum of 3, has been demonstrated. For this test, velocity at the top of the boot varied from 0.6 to 0.8 ft/sec and the U/Umf at the bottom of the column was between 1.6 and 2.6.

With test No. 15, continuous particle separation has been demonstrated for a silicon bed depleted 33% in volume and 31% in particle size over a test period of 17.5 hours by extracting 450-500μ mean particle size silicon in 0.9-lb increments. A U/U_{mf} of 2 was maintained at the bottom of the 6-inch column, while the U/U_{mf} at the top of the boot was gradually decreased from 10 to 7 over the test period. It was necessary to decrease the boot velocity as the bed mean size became smaller in order to prevent a mixing type of slugging flow after withdrawal of material. An alternate opinion which was not tried would be to reduce the quantity of silicon removed. On the average, it took about one hour for the boot tube to refill with coarse silicon. This is about 2.5 times longer than what was required when the bed was maintained at constant size (Test No 13).



NOTE: TEST NO. 13 SILICON OF WIDE PARTICLE SIZE DISTRIBUTION 0.17 - 0.20 ATM HELIUM, 0.6 - 0.8 FT/SEC BOOT VELOCITY

(1)- TEMPORARILY LOST SEPARATION WHEN TEST WAS INTERRUPTED IMMEDIATELY AFTER EMPTYING BOOT OF COARSE PARTICLES.

FIGURE 7 PARTICLE SEPARATION WITH 1-1/2 INCH BOOT, TEST NO. 13

The completed tests identified the operating parameters for particle separation using the 1½-inch diameter boot assembly. These tests were conducted with helium and nitrogen at reduced pressures to simulate, respectively, hydrogen at 1 atm and 680°C, and hydrogen at 1.44 atm and 21°C. The separation criterion was whether the boot tube refilled with coarse particle size silicon ($\bar{d}_p$ = 450 - 500μ) following the extraction of about 500 grams of silicon from the bottom of the boot. This quantity is about 70% of the boot volume, and is the maximum amount that can be withdrawn at one time without creating a mixing type of slugging flow in the boot for the velocities which will normally be used. The column bed was maintained at constant volume and particle size ($\bar{d}_p$ = 185μ) by replacing silicon with previously withdrawn samples. The results of the work are summarized in Figures 8 and 9. It is expected that if the amount of silicon withdrawn is reduced to a smaller percentage of the boot tube volume, the curves defining the region of no separation will shift upward.

Capacitive Heating Tests:

The last of the series of high temperature tests, Test No. 25, was completed using a new high-purity silicon bed of 222μ mean particle size. Capacitive heating was operated for 46 hours at an average input power of 200 watts. Operating temperature ranged from 575°C to 760°C (666°C mean). For the first 20 hours, the bed impedance averaged 5.3 Ω, about what it should be for the field geometry. For the last 26 hours, the impedance averaged 10.6 Ω. The reason for the change is not known. The sintering rate was 6.3% per day. The mean impedance for the test run was 8.3 Ω. Based on earlier work, a 12% sintering rate would be expected. Bed/Wall ΔT averaged 10-15°C. A summary report will be presented in the next quarter.

The next series of tests examined electrical characteristics of multiple electrodes in a large (6-inch diameter) fluid-bed column, which is the size of the PDU being designed. An extensive number of electrical characteristic measurements have been made with the electrode assembly installed in the 6-inch diameter fluid-bed column. Tests were conducted with helium and nitrogen at pressures of 0.1, 0.5, and 1.0 atm and at U/Umfs

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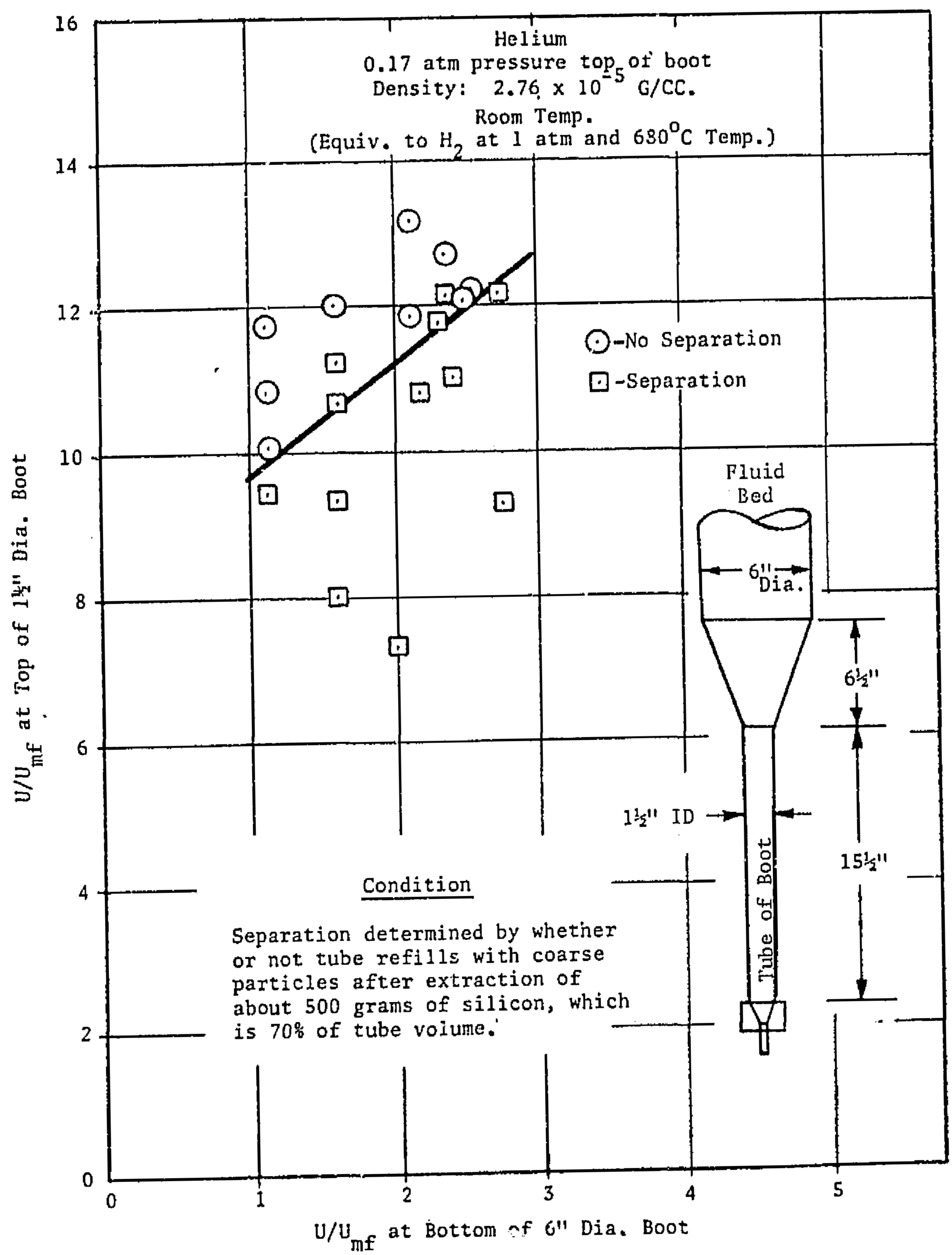


FIGURE 8. SUMMARY OF PARTICLE SEPARATION TESTS IN HELIUM

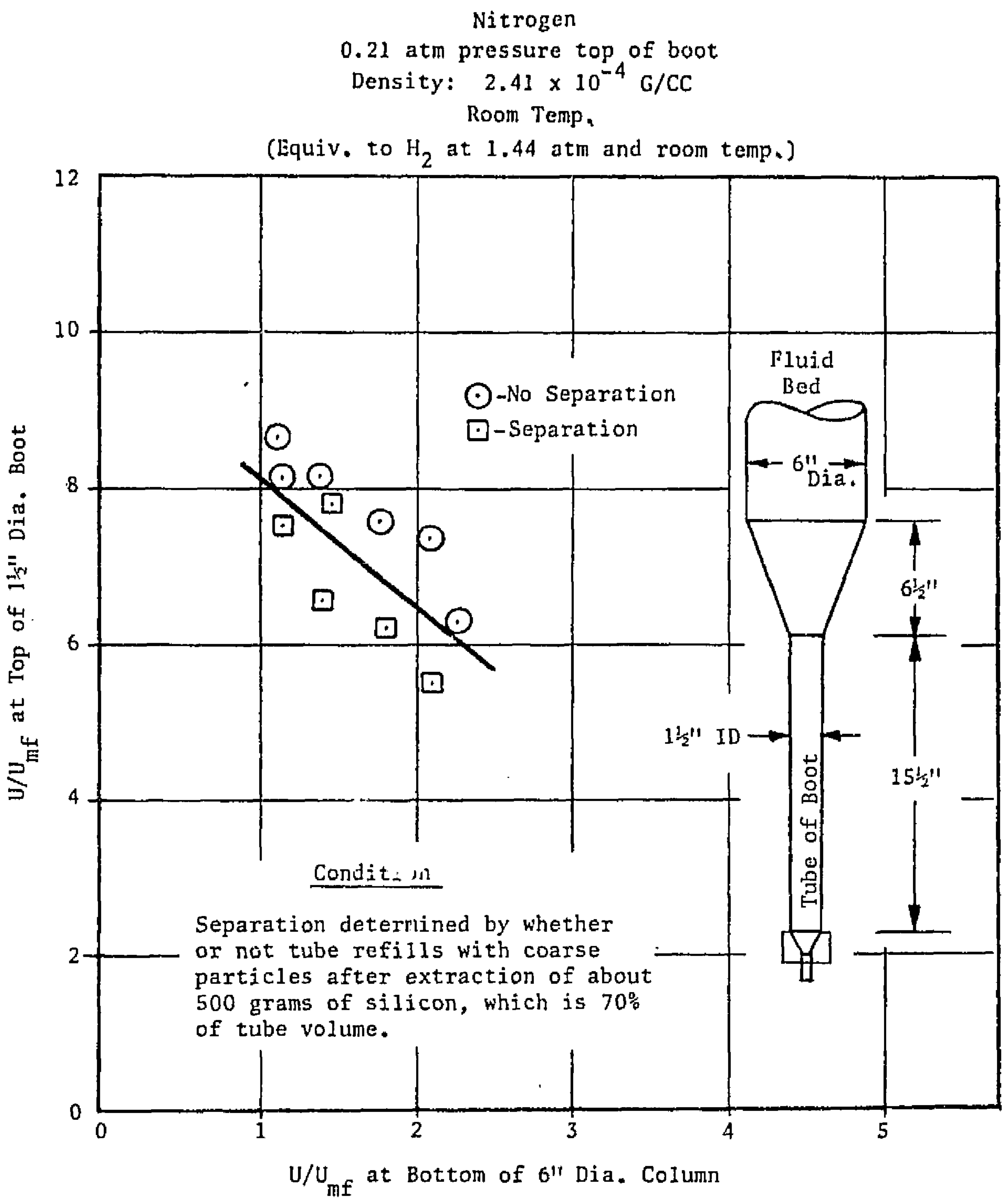


FIGURE 9. PARTICLE SEPARATION TESTS IN NITROGEN

from 1 to 4.

Electrode spacings of 0.63, 0.82, 1.00, 1.19, 1.38, and 1.55 inches were examined at bed penetrations varying from 1 to 8 inches. Some general patterns were observed. The silicon bed resistivity measurements made in nitrogen increased with U/Umf and decreased with pressure. Resistivity appears to be constant for U/Umf greater than 4. The helium measurements did not exhibit any dependency on pressure or velocity.

The maximum electrode spacing in nitrogen was 1.0 inch. Beyond this, it was not possible to obtain fluid dielectric breakdown with the power system used. Spacing of 1.55 inches in helium was possible. Since the dielectric constant for hydrogen is between nitrogen and helium, operation of the 1-inch diameter electrodes in a 6-inch diameter fluid-bed reactor appears feasible. Some effort will be required to correlate these data and the 2-inch diameter capacitive heating reactor data so that a matching transformer network having the required flexibility can be specified.

1.6.3.3 Evaluation Fixed-Bed/Fluidization

This effort consists of the analysis of the data from the fixed-bed and fluidization experiments for applicability for designing the fluid-bed PDU.

An evaluation report is being prepared to present the results of the critical silane concentration curve determination and silane decomposition reaction rate constant study. The final critical silane concentration curve lies between the curves defined in the literature by Eversteijn and Murthy. The calculated activiation energy for silane decomposition from our data is 39 kcal/mole which compares well with Eversteijn's value of 37 kcal/mole. The agreement for reaction rate constants is also favorable, as shown in the following:

k, rate constant (min^{-1})

^{o}C	Eversteijn	This Work
576	0.012	0.010
627	0.040	0.026
676	0.096	0.112

1.6.3.4 Fluid-Bed Pyrolysis (PDU)

This work item consists of all effort associated with the design, fabrication, and testing of an experimental unit to establish design data for an EPSDU-scale system.

Design of the fluid-bed pyrolysis PDU was started. A preliminary flow sheet and P&I diagram were issued and reviewed. The reactor consists of a 6-inch pipe about 6-feet long which is attached to a 5-foot long expanded head 18-inches in diameter. Silicon partical separation and withdrawal is made in a 1½-inch diameter boot directly under the 6-inch pipe.

After evaluating a number of possible installation locations, the fluid-bed PDU will be installed in the same explosion-proof test cell that now houses the free-space reactor PDU. A number of safety features will have to be enforced as a result of putting two PDU's in a single test-cell. These requirements were identified and will be implemented. By installing the fluid-bed PDU in the existing test cell, both PDU's can use the silane feed and exhaust vent systems currently being used for the free-space reactor. This is cost effective and should reduce the installation schedule.

1.6.3.5 Fluid-Bed Process Design For EPSDU

This work item, which includes preparation of a process design package, cost estimate, and economic analysis for a test unit that could be tested as an add-on unit to EPSDU, will use the PDU work as a basis.

1.6.3.6 Fluid-Bed Model Update

The fluid-bed model developed under Phase I of this contract is being updated to reflect current data and requirements.

The computer model for sizing the silane pyrolysis fluid bed for diameter and bed height at a specified percentage silane concentration uses the rate data of Eversteijn. The model was updated by incorporating the rate data resulting from this work. The result of the update was a calculated bed size about 12% larger, in the temperature range of 575° to 675°C.

1.6.4 Quality Control

This task includes all activities associated with the development of quality control equipment, techniques, and procedures for use during ESPDU operations.

1.6.4.1 Silane Analysis - Slim-Rod Deposition

This effort involves the design, fabrication, and operation of a laboratory-scale reactor to develop a technique for analyzing silane. This technique is based on depositing a substantial amount of silicon on a heated slim rod and characterizing the silicon deposit by electrical measurements.

The flow controllers, ordered last August and received late this quarter, have been installed. The silane purge gas lines are complete and have been leak tested. Excellent progress was also made on the electrical power supply panel. The high-current DC power unit and the high-voltage AC power controllers were operated and performed as specified. The control panel, complete except for the mass flow control unit for the epitaxy reactor system, was moved to the laboratory area for final connection. Final checkout should be completed before the mid-April saftey review. Preliminary operating instructions, written in preparation for the review, will be finalized after the unit is operated and the analytical methods are developed. A supply of silicon seed rods is on hand and an ingot of nominally 60 Ω cm P-type silicon was ordered so that additional seeds can be made.

1.6.4.2 Silane Analysis - Direct Measurement

This work item covers the investigation of analytical techniques for total carbon analysis and chromatographic methods for boron and phosphorus.

The determination of trace levels of phosphine in silane can be achieved with a detection limit of about 6 parts-per-billion (ppb) utilizing a gas chromatograph with a photoionization detector. This method should be utilized to augment other analytical techniques for silane quality control in the planned 100 MT/Yr silane-silicon EPSDU. The detection of low levels of diborane by chromatographic separation from silane was not successful. Alternate methods of analysis for the important electronically active species should be explored.

Phosphine Analysis:

The Union Carbide Corporation process utilizes the high volatility of silane advantageously as a means for removing harmful impurities through conventional distillation. One of the closest boiling impurities potentially present in silane (boiling point -112°C) is phosphine (boiling point -87.4°C). Phosphorus is an extremely potent donor-type impurity in silicon and is not easily removed by normal unidirectional freezing methods during crystal growth (segregation coefficient = 0.35). Rapid, high-sensitivity analysis of the silane intermediate provides important advantages for control of silicon quality and trouble-shooting for trace contaminants.

If analytical methods were available for accurate determination of phosphine, the most volatile phosphorus compound, control of product silicon quality could be effected by the on-line addition of controlled amounts of phosphine. Measuring the level of phosphine in silane in the low part-per-billion range is important for assessing the performance of the final silane distillation column, correlating silane quality with final silicon quality, or providing a means for on-line doping to achieve consistant quality silicon.

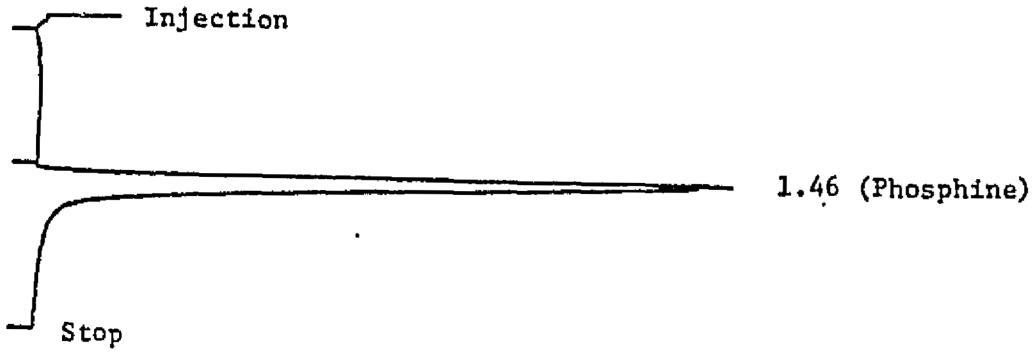
The determination of trace quantities of electrically active impurities in silane and chlorosilanes has been historically performed by deduction from the resistivity value of silicon films produced by decomposition of the silane in question.[1] Other methods, such as substoichiometric separation - isotope dilution mass spectroscopy, low temperature infrared spectroscopy, and wet chemistry methods have been proposed, but all require the use of expensive equipment, time consuming standardization, or preconcentration steps. The purpose of this present work was to develop an analytical technique which was rapid, quantitative, and capable of ready application to on-line quality control of silane purity.

A chromatographic technique has been developed which can determine the level of phosphine in silane at a detection limit of about 3 parts-per-billion. The heart of the technique is the use of a photoionization detector which is particularly sensitive to phosphine. A GOW-MAC 550 chromatograph was equipped with an HNu, 10.2 electron volt photoionization detector operating at 120°C. The separation column was 6 feet of 1/8-inch O.D. stainless steel tubing packed with Poropak P, 60-80 mesh. At an oven temperature of 50°C and 24 cc/min helium carrier flow, a 0.5 cc sample of 13 parts phosphine in helium was eluted in 1.46 minutes (Figure 10). The total area of 829679 corresponds to a response of 8.42×10^{13} area counts/gram of phosphine. Elution time and volume were reproducible.

A gas blend of 1 part-per-million phosphine in silane was prepared. The chromatogram of this sample, Figure 10, clearly shows the silane elution at 0.92 minute followed by the phosphine peak at 1.42 minutes. In this case, the integration calculation utilized a "tangent skim" method since the large silane volume had not been completely eluted. Using the same sensitivity

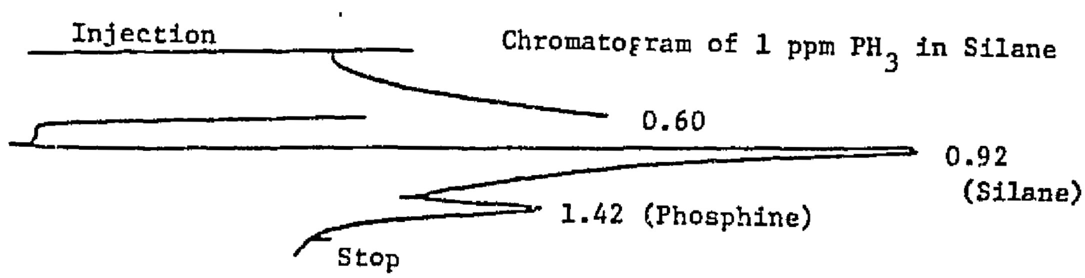
[1] Yusa, A. Rev. Sci. Instrum., Vol. 47, No. 2, February 1976, P. 224

Chromatogram of 13 ppm PH_3 in He



TIME	TYPE	AREA	ID #	AMT
1.46	1	834316	1	13.13 PPM
			TOTAL	13.13 PPM

Chromatogram of 1 ppm PH_3 in Silane



TIME	TYPE	AREA	ID #	AMT
.60		489151		
.92	IM	1827126		
1.42	T	72567	1	1.142 PPM
			TOTAL	1.142 PPM

Equip:	Gow Mac - 550
Carrier:	He at 24 cc/min.
Column:	6.0-ft. x 1/8-inch OD, S.S. Poropak P, 60-80 mesh
Oven Temp:	50^{o}
Sample:	0.5 cc
Detector:	HNu 10.2 ev Photoionization type

FIGURE 10. TRACE PHOSPHINE DETECTION BY CHROMATOGRAPHIC METHOD

factor determined for the phosphine-only case, the calculated quantity of phosphine was determined to be 1.142 parts-per-million. Thus, it was confirmed that in the presence of silane, phosphine in trace levels can be separately eluted, that the sensitivity of the photoionization detector to phosphine is unaffected by the presence of silane, and that the "tangent skim" method of peak area determination is satisfactory.

For part-per-billion levels of phosphine, the sample size could be increased to 1.0 cc. Samples larger than 1-2 cc would not be as effective, because they would tend to overload the column and wash out the sensitivity. Assuming a 1.0 cc sample size and an ultimate sensitivity of 200 area counts for the "tangent skim", the limits of detection of phosphine would be 3 parts-per-billion.

Boron Analysis:

Chromatographic determination of low levels of diborane in silane was not successful. To establish the type of chromatographic column which would quantitatively elute diborane and to establish detector sensitivity, gas mixtures containing 1% diborane in helium and 1% diborane in silane were prepared. Since diborane is extremely reactive with moisture, care was taken to thoroughly dry the helium carrier gas, column packing, sampling valve, and associated plumbing. The first problem encountered and solved was that a simple, hot-wire type thermal conductivity detector exhibited a non-reproducible response to diborane. Detector sensitivity varied with the sample injection frequency. A switch to a thermistor-type detector, which operates at a much lower temperature, appeared to solve that problem so that a reproducible detector response was measured for a given quantity of diborane injected. With a sensitivity of 500 area counts/10^{-7} gm of diborane on the GOW-MAC 500/HP 3388 unit used, this provided an ultimate detection limit of 50 ppm.

Several column packings were evaluated first to achieve a delayed (with respect to the carrier gas flow), yet quantitative elution of diborane, and then later to achieve a differential elution volume between diborane and silane. Since diborane is thermally unstable, a low temperature control system was provided to evaluate chromatographic separation at temperatures as low as -40°C.

TABLE II

COLUMN PACKING EVALUATION FOR DIBORANE

	Mixture	
Packing	Diborane/Helium	Diborane/Silane
Carbopack	No elution	-
Silicon Oil	No elution	-
Dioctylphthalate	OK	No separation
Poropak QS	No elution	-
n-hexadecane	OK	No separation

All tests were performed over a temperature range of -40° to +40°C.

Since these column packings cover a broad spectrum of polarity and activity, the chance of finding a suitable system for achieving good separation does not appear promising. Reactive techniques similar to those applied to the determination of hydrocarbons in silane do not work well with diborane since diborane, rather than the silane, is the more reactive with most common reagents. Further, even if a chromatographic separation could be achieved, the modest detector sensitivity would give a lower limit of detection of only about 50 ppm. Other detectors, such as flame photometric or photoionization, are not very sensitive to boron as compared to the excellent results which can be obtained with phosphorus compounds.

Further analysis reveals that diborane's rapid hydrolysis in water, as compared with silane, offers a possible wet chemical technique whereby a relatively large volume of silane could be bubbled through acidified water.

The acidified water could then be analyzed for boron using standard methods of complexing with curcumin or carminic acid and measuring its UV absorbance.

With the poor detector response to diborane and the failure to achieve a significant chromatographic separation between diborane and silane, work in this area has been suspended.

1.6.4.3 Silane Analysis - Epitaxy

This effort includes the design, fabrication, and operation of an epitaxial reactor to develop methods for measuring silane purity use during EPSDU operation. This technique is based on measuring the resistivity of an epitaxial layer of deposited silicon to determine silane purity.

The control system for the epitaxy reactor is being incorporated into the slim-rod reactor electrical panel. All components have been received except for the mass flow controllers which are now scheduled for early April delivery. All of the reactor parts have been prepared for interconnecting.

A supply of 10 Ω cm P type wafers, 30 mm diameter is on hand for initial operation. A supply of N and P type wafers has been ordered for June delivery. Since it is not known which impurities will dominate EPSDU silane, the epitaxy test will utilize one wafer of each type. Thus, either a P-N or an N-P junction will be grown and the resistivity of the epitaxy film measured conveniently with a conventional four-point probe.

1.7 MANAGEMENT AND DELIVERABLES

The baseline package of the Performance Measurement System (PMS) was prepared. The Monthly Financial and Management Report, starting with the month of January 1980, is being prepared in accordance with the PMS. The report contains a summary of technical, cost, and schedule status and projected cost and schedule with emphasis on the following six months. The information is presented through correlation of schedule and financial performance at the Work Item level with the Work Breakdown Structure (WBS), Baseline Cost Estimates, and Program Plan. The Program Plan provides, among other things, the detailed schedule and monthly or bi-monthly milestones to be reached.

Based on the analysis technique derived from the Performance Management System (PMS), the report:

- indicates the progress of work
- relates cost and schedule performance
- identifies cost and schedule problems and their sources
- defines the impact of problems and corrective action
- projects changes in the estimated cost through completion

The following major milestones were reached this quarter:

WBS No.	Milestone	Description of Major Milestone
1.1.3.2	(A)	Electrical one-line diagram issued.
	(D)	Electrical Power System equipment specifications complete, RFR's issued.
1.1.5.1	(A)	EPSDU Facility Cost estimate issued.
1.2	(A)	Quarterly Procurement Status Report (PSR) issued.
1.6.1.1.3	(B)	First silane decomposed in Free-Space Reactory (FSR) - powder sample available for shipment.
1.6.1.1.4	(C)	Run No. 1 in FSR complete - internal report issued.
1.6.1.2.2	(F)	Internal report issued on development of new FSR model.
1.6.2.1.2	(B)	Sub-contract signed for development of melter.
1.6.3.1.3	(A)	Internal report issued on Fixed-Bed Deposition experiments.

WBS No.	Milestone	Description of Major Milestones
1.6.3.3	(C)	Internal report issued evaluating silicon deposition experiments in a fixed-bed reactor.
1.6.4.2.2	(M)	Draft report completed on Chromatographic Methods for Boron & Phosphorus Determination.
1.6.4.3.1	(B)	Epitaxy reactor final process flow sheet and equipment purchase orders issued.
1.6.4.3.2	(E)	Epitaxy reactor in place and ready for system connections.
1.7.3	(A) thru (C)	Monthly Financial and Management Reports for December 1979 and January, February 1980 issued.
1.7.3	(A) thru (C)	Monthly Technical Progress Reports for December 1979 and January, February 1980 issued.
1.7.3	(A)	Second Bi-monthly JPL/UCC review held.
1.7.3	(A)	Quarterly Technical Progress Report for the 4th quarter 1979 issued.
1.7.3	(A)	Program Plan and PMS Baseline issued.
	(B)	Hydrogenation Reactor Design memo issued.
	(I)	Chromatographic Methods for Boron & Phosphorus Determination memorandum issued.

SECTION III. CONCLUSIONS

The significant highlights and conclusions are presented according to WBS numbers.

1.1 DESIGN/PROCUREMENT

1.1.1 Process Design

- The hydrogenation design was completed with a change to the M-G silicon conveying system from a dense-phase pneumatic system to a flexible screw conveying system which is simpler and more economical.

- For the purpose of defining the pyrolysis reactor/melter building, preliminary sizing and configuring of the free-space reactor, meltor, and the quality control laboratory were made.

- All four distillation columns were designed and their procurement specifications have been written.

- The equipment dry-out procedure for plant startup was completed as well as the function specification for the dry-out nitrogen heater. The waste stream neutralization scheme was also completed with the decision to use caustic soda to neutralize all waste streams in one common tank.

- The waste-burner test was completed. The test report and the EPSDU burner functional specifications, originally scheduled for completion in February, will be issued in April.

1.1.2 Facility Layout

- The layout drawings were completed and are ready for review. These drawings incorporate the revised waste treatment system, environmental considerations, and personnel safety.

1.1.3 Equipment Design, Specifications, Procurement

- The electrical one-line diagram was completed and issued. Work is in progress on the schematic wiring diagram.
- Process control instrument specifications have been issued to obtain vendor quotations.
- Requests for requisition (RFR's) for process equipment were issued for twenty-six major items.
- Equipment specifications for the electrical power system were completed, and all RFR's were issued.

1.1.4 Installation Design, Specification

- Design and drawing work has been initiated on schedule for EPSDU site preparation.

1.1.5 Cost Estimating

- The detailed cost estimate for EPSDU was completed based on definition developed during June-December 1979 pre-engineering activities. Costs for Report Items 1.1 and 1.2 are compatible with the PMS baseline costs; however, the construction sub-contract cost (RI 1.3) is higher than baseline cost.

1.2 EQUIPMENT FABRICATION/DELIVERY

- Validated purchase orders were issued for the power supply, three pumps, the therminol heater, and the cooling tower. Some delays in issuing purchase orders were experienced, but this will be corrected in the coming quarter.

1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS

- Inputs necessary for heat and mass balance work have been prepared, and the work was started on schedule.

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Development

- Successful start-up of the free-space reactor PDU was accomplished in January.
- Improvements were made to the powder scraper design and the silane feed valving.
- After the above modifications, the PDU was operated continuously 24 hours at an average silane feed rate of 4.3 lb/hr.
- More experimentation is required to protect the quartz liner from breaking during long-duration operation.

1.6.2 Melting/Consolidation

- Kayex Corporation won the melter sub-contract, and work was initiated in March.

1.6.3 Fluid-Bed Development

- Deposition rate experiments in a fixed-bed at 625°C have indicated the rate to be first order.
- Continuous separation of course particles from a fluid bed was

demonstrated in a 6-inch diameter bed having a 1½-inch diameter boot.

- A preliminary P&I diagram for the fluid-bed pyrolysis PDU was completed for review.

1.6.4 Quality Control

- A chromatographic technique has been developed for determining trace levels of phosphine in silane. Using a photoionization detector, phosphine was reliably detected in silane at the one part-per-million level with sensitivity such that the estimated detection limit appears to be three parts-per-billion.

- The slim-rod reactor is being assembled and completion is expected within one month.

- Completion of epitaxy reactor is expected within 4 to 6 weeks.

1.7 MANAGEMENT & DELIVERABLES

- The approved Baseline PMS package was issued.

- The Monthly technical report and the financial and management report were formatted in accordance with the Baseline PMS starting with the January 1980 submittal.

SECTION IV. PROJECTED QUARTERLY ACTIVITIES

1.1 EPSDU-DESIGN/PROCUREMENT

1.1.1 Process Design

- Final EPSDU process design package will be completed.
- Equipment layout will be reviewed and approved during the next reporting period.
- The functional specifications for the waste treatment section will be completed.

1.1.2 Facility Design

- The facility layout drawings will be issued.
- The facility safety review will be completed
- The scale model work will start.

1.1.3 Equipment Design

- A preliminary schematic wiring diagram will be completed.
- The RFR's for field instrumentation will be issued.
- The RFR's for the waste treatment equipment will be issued.
- RFR's for the auxiliary and support equipment will be issued.
- The equipment list for the data collection system will be made.

1.1.4 Installation Design Specification, Sub-contract

- The site package will be issued for bids.
- Concentration on the civil/structural design, mechanical/piping design, and electrical design will be accelerated.

1.1.5 Cost Estimating

- Cost improvement because of process and facility design changes will be studied.

1.2 EQUIPMENT FABRICATION/DELIVERY

- Validated purchase orders will be issued for over 85% of the equipment.

1.3 INSTALLATION & CHECK-OUT

- Installation sub-contracting requirements will be defined.

1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS

- A functional process design package will be assembled.
- Engineering design work will be initiated.

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Development

- A second 24-hour run will be made.
- A report covering the experimental work will be written.

1.6.2 Melting/Consolidation

- Prototype design work will start.

1.6.3 Fluid-Bed Development

- Fabrication drawings for the PDU will be completed.
- Purchase orders will be issued for major PDU components.

1.6.4 Quality Control

- The slim-rod reactor will be installed, checked out, and started-up.
- The epitaxy reactor will be installed, checked out and started-up.
- Development of test methods for slim-rod deposition analysis and epitaxial deposition will be completed.

APPENDIX A

SAMPLE PROCUREMENT DOCUMENTATION

CONTENTS

EXHIBIT A SAMPLE FUNCTIONAL SPECIFICATION (Hot Oil Pump)

Issue Date 6/1/7[illegible]
Rev A

LOW COST SILICON SOLAR ARRAY PROJECT

PUMPS

ORIGINAL PAGE IS OF POOR QUALITY

EQUIPMENT NO. 466-02

EQUIPMENT NAME Hot Oil Pump

PROJECT NAME 100 MTY EPSDU

NO. REQD One

TYPE AVS Centrifugal

FLUID HANDLED Therminol 60

Flowrate: Theo. - Design 150 GPM

Viscosity of Liquid 0.868 CP @ 300^{o}F

Density of Liquid 56.9 Lb/Ft^{2} @ 300^{o}F

Temp. of Liquid 311^{o}F Operating, 500^{o}F Design

DESIGN PRESS. 60 PSIG

NPSH Available 30 Ft (minimum)

Suction Press. 25 PSIA

Discharge Press. 65 PSIA

Differential Pressure: Theo. 40 PSI = 100 Ft Design 130 Ft TDH

Dissolved Gases None

Suspended Solids None

Type Seal Single Internal, Unbalanced

Material of Construction Ductile Iron or Manufacturer's Standard

Corrosion/Erosion Caused by

REMARKS:

EXHIBIT B SAMPLE ENGINEERING SPECIFICATION
(Hot Oil Pump) Page 1 of 2

ORIGINAL PAGE IS
OF POOR QUALITY

GS-21 ROTATING PUMP DATA SHEET

PROJECT 7002 | ES. NO. 6893 Rev. 1

LOCATION E. Chicago, IN | REQ. NO. 50012

ATMOS. PRESS. 14.6 psia

MANUFACTURER			
CONDITION NUMBER	1	2	3
A-SERVICE AND PERFORMANCE			
Number of Pumps Required	1		
Fluid Handled	Therminol 60		
Capacity each pump in GPM	150		
Specific Gravity	.905		
Pumping Temp. °F (max/norm/min)	311		
Viscosity (min/norm/max)	0.868		
Vapor Pressure (max/norm/min) mm Hg	30		
Total Differential Head in ft.	130		
NPSH (available/req'd) ft.	30/3		
Efficiency %	52%		
BHP	8.6		
Speed in RPM	1780		
Performance Curve No.	CDS 1680-5		
B-PUMP DETAILS			
Model or Type	3196 MT		
Casing (vert.split) Horiz. split)	Vertically		
Maximum Operating Speed	1800		
Maximum Case Working Pressure	275 psig		
Maximum Case Working Temperature	500° w/Cooling		
Line Bearing Type	Ball		
Thrust Bearing Type	Ball		
Bearing Lubrication	Oil		
Suction Flange Size	3		
Rating & Type Facing	300# RF		
Discharge Flange Size	1½		
Rating & Type Facing	300# RF		
Packing or Mechanical Seal	Mech. Seal		
Description	Chesterton 880 D1W1		
Impeller Type	Open		
Coupling Type & Mfg.	Woods		
C-DRIVER TYPE (MOTOR/TURBINE/OTHER)	Motor		
Driver Characteristics	460/3/60		

UNION CARBIDE CORPORATION Linde Division Engineering Department						
	Rev.	1				
	Date	2/19/80				
	Initial	KAK				

GS-21 ROTATING PUMP DATA SHEET

PROJECT 7002

LOCATION E. Chicago, IN

ATMOS. PRESS. 14.6 psia

ES. NO. 6893 Rev. 1

REQ. NO. 50012

ORIGINAL PAGE IS
OF POOR QUALITY

D-MATERIALS OF CONSTRUCTION	
Casing	Ductile Iron
Case Wear Rings	None
Impeller Wear Rings	None
Shaft	SAE 4140
Shaft Sleeves	316 SS
Impeller	Ductile Iron
Bearing Housing	Cast Iron
Base Plate	Cast Iron
Packing or Mechanical Seal	
Strainer	None
E-GENERAL	
Weight Pump Assembly	
Driver	
Other	
Total	
Lubrication Type/Capacity	Trico Oilers
F-SPACE REQUIREMENTS	
Length	
Width	
Height	
Outline Drawing Number	
G-REMARKS AND/OR ADDITIONAL INFORMATION	

UNION CARBIDE CORPORATION Linde Division Engineering Department							
	Rev.	1					
	Date	2/19/80					
	Initial	KAK					

UNION CARBIDE CORPORATION
Linde Division
Tonawanda, New York

REQUISITION OR QUOTATION REQUEST

TO: Purchasing Dept.
ATTN: F. H. Anderson
Buyer

DATE February 19, 1980
PROJECT 7002 E. Chicago, IN
REQUISITION 825-50012
ESTIMATED COST $2,897.00
WORK ORDER & ACCOUNT # C885-000-7002-242

Attached are 4 copies of the following:

SPEC NO./DWG. NO.	DATE	REVISED DATE	NO. OF PAGES
ES-6893 Rev. 1		2/19/80	

TO BE USED FOR: Pumping Therminol - Item 466-02

IF QUOTATION REQUEST: QUOTE

IF REQUISITION REQUEST: SELLER Goulds
Name Address City & State

DESCRIPTION: One (1) Goulds pump, Model 3196MT, size 1½ x 3-13

(A) Quotations required not later than ________.

(B) Drawings for approval required in accordance with Form PUR-101, not later than Noted on GS-1 Data Sheet.

(C) Operating and Instruction Manuals are required

- [x] In accordance to PUR 101.
- [ ] ____ copies superseding PUR requirements.
- [x] Instruction Material Specification GS-2 applies.
- [ ] Other restrictions

(D) Equipment required on site not later than January 31, 1981, but not before January 1, 1981.

(E) Engineering questions and quotations shall be directed to K. A. Kuberka.

(F) Additional Remarks: (If purchased against a Requirement Order or Price Agreement, so state)

ORIGINATOR K. A. Kuberka 2/19/80
K. A. Kuberka

AUTHORIZATION SIGNATURE CAS 2/19/80 [signature] 2/20/80
(Engineering) F. J. Doyle

AUTHORIZATION SIGNATURE [signature] 2/21/80
(Project Management)

..., N. Y.

Form PUR-201 Issued 4/15/63, Revised 2/4/77

LINDE DIVISION

DATE 2/19/80

SPECIFICATION FOR Hot Oil Pump

SPECIFICATION NO. ES-6893 Rev. 1

PLANT LOCATION E. Chicago, IN

REQ NO 50012

BY K. A. Kuberka APP [signature]

PROJECT 7002

SPECIFICATION NO. ES-6893 Rev. 1 THE FOLLOWING MADE UP OF AND CONSISTING OF		NO. OF PAGES	REVISION NO. & DATE
Cover Sheet		1	
Revision Sheet		1	
Remarks Sheet		1	
GS-21 Rotating Pump Data Sheets		2	
GS-76 Standard NEMA Frame Size Motor Specification		2	
GS-1 General Requirements Data Sheet		1	
		8	
The following attached general specifications also apply and become a part of this specification.			
GS-1	General Requirements Specification	2	3 12/24/68
GS-2	General Specifications for Instruction Materials	1	3 1/15/68
GS-21	Rotating Pump Specification	1	
PUR-101	Instructions for Submission of Drawings for Approval and Other Data	1	2 5/9/77
TOTAL PAGES		13	

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REMARKS

NON DESTRUCTIVE TESTING REQUIRED: [ ] YES [XX] NO

UNION CARBIDE CORP LINDE DIVISION ENGINEERING DEPT.		
REV	1	
DATE	2/19/80	
BY	KAK	
APP	[initials]	

A-5

Revision Sheet

Revision	Comments and Location	Revised By	Date	Approved	Date
1	Changed RFQ to RFR.	KAK	2/19/80	[illegible]	2/26/80

PROJECT NO. 7002

LOCATION E. Chicago, IN

ES NO. 6893 Rev. 1

REQ. NO. 825-50012

REMARKS SHEET

1. One (1) Goulds pump, Model 3196MT, size 1½ x 3-13 is required for pumping therminol 60 heat transfer fluid at E. Chicago, IN.
2. The pump shall be equipped with water-cooled stuffing boxes.
3. Maximum motor load shall not exceed 98% of the motor nameplate rating.

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GS-21 ROTATING PUMP DATA SHEET

PROJECT 7002

LOCATION E. Chicago, IN

ATMOS. PRESS. 14.6 psia

ES. NO. 6893 Rev. 1

REQ. NO. 50012

MANUFACTURER			
CONDITION NUMBER	1	2	3
A-SERVICE AND PERFORMANCE			
Number of Pumps Required	1		
Fluid Handled	Therminol 60		
Capacity each pump in GPM	150		
Specific Gravity	.905		
Pumping Temp. °F (max/norm/min)	311		
Viscosity (min/norm/max)	0.868		
Vapor Pressure (max/norm/min) mm Hg	30		
Total Differential Head in ft.	130		
NPSH (available/req'd) ft.	30/3		
Efficiency %	52%		
BHP	8.6		
Speed in RPM	1780		
Performance Curve No.	CDS 1680-5		
B-PUMP DETAILS			
Model or Type	3196 MT		
Casing (vert.split) Horiz. split)	Vertically		
Maximum Operating Speed	1800		
Maximum Case Working Pressure	275 psig		
Maximum Case Working Temperature	500° w/Cooling		
Line Bearing Type	Ball		
Thrust Bearing Type	Ball		
Bearing Lubrication	Oil		
Suction Flange Size	3		
Rating & Type Facing	300# RF		
Discharge Flange Size	1½		
Rating & Type Facing	300# RF		
Packing or Mechanical Seal	Mech. Seal		
Description	Chesterton 880 DIW1		
Impeller Type	Open		
Coupling Type & Mfg.	Woods		
C-DRIVER TYPE (MOTOR/TURBINE/OTHER)	Motor		
Driver Characteristics	460/3/60		

UNION CARBIDE CORPORATION Linde Division Engineering Department	Rev.	1					
	Date	2/19/80					
	Initial	KAK					

GS-21 ROTATING PUMP DATA SHEET

PROJECT 7002

LOCATION E. Chicago, IN

ATMOS. PRESS. 14.6 psia

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ES. NO. 6893 Rev. 1

REQ. NO. 50012

D-MATERIALS OF CONSTRUCTION	
Casing	Ductile Iron
Case Wear Rings	None
Impeller Wear Rings	None
Shaft	SAE 4140
Shaft Sleeves	316 SS
Impeller	Ductile Iron
Bearing Housing	Cast Iron
Base Plate	Cast Iron
Packing or Mechanical Seal	
Strainer	None
E-GENERAL	
Weight Pump Assembly	
Driver	
Other	
Total	
Lubrication Type/Capacity	Trico Oilers
F-SPACE REQUIREMENTS	
Length	
Width	
Height	
Outline Drawing Number	
G-REMARKS AND/OR ADDITIONAL INFORMATION	

UNION CARBIDE CORPORATION Linde Division Engineering Department	Rev.	1					
	Date	2/19/80					
	Initial	KAK					

GS-76 STANDARD NEMA FRAME SIZE MOTOR SPECIFICATION

1.0 SCOPE

1.1 Purpose - This specification is to be used to specify motors in the NEMA frame size range for equipment supplied by original equipment manufacturers, hereinafter referred to as O.E.M., on pumps and packages. The O.E.M. shall supply "Mill and Chemical Industry" type motors for all applications and in accordance with the requirements listed below.

1.2 Operating Condition

1.2.1- Motor load at design operating point of the driven equipment shall not exceed 85% of the motor nameplate rating. Care must be exercised in sizing lube-oil pump motors operating outdoors in northern climates and cooling tower fans in cold weather. (Horsepower will increase as temperature decreases). Seller shall supply operating curves for the driven equipment indicating the horsepower requirement for the most severe operating load.

In addition, driven equipment that operates more than 30% of the time shall be supplied with "energy efficient", Mill and Chemical Industry type motors by the O.E.M. and in accordance with the requirements listed below. Typical applications include water pumps, crankcase vacuum pumps, lube oil console vacuum pumps, cooling tower fans, D.C.A. pumps, chiller auxiliaries, regeneration blower auxiliaries, recirculation gel trap pumps, etc.

2.0 MOTOR REQUIREMENT

2.1 Electrical

2.1.1- Voltage 460 Phase 3 Hertz 60

2.1.2- Insulation and Temperature Rise -

B (80°C) or F (105°C) rise by resistance over 40°C ambient.

2.1.3- Service Factor - 1.0

2.1.4- Locked Rotor KVA Code - Code G unless otherwise specified Code ____________

2.2 Construction

2.2.1- Enclosure - Cast Iron (Frame - Terminal Box - Fan Guard) Totally Enclosed Fan Cooled (TEFC)

2.2.2- Service - Mill and Chemical Industry Duty

2.2.3- Bearings - Anti Friction - Grease Lubed

Linde Division - GS-76

3.0 NAMEPLATE DATA BY SELLER

The following motor manufacturer nameplate data shall be supplied by Seller to Buyer within two (2) months of receipt of order.

3.1 Motor Manufacturer________________________

3.2 Horsepower__________ Service Factor__________ F.L. Speed__________

3.3 Voltage______________ Phase______________ Hertz__________

3.4 F.L. Amperes__________ KVA Inrush Code______________

3.5 Insulation Class__________ Temp. Rise over 40°C Ambient __________ by Resistance.

3.6 NEMA Efficiency Code__________________

3.7 NEMA Torque Designation______________

3.8 Frame Size________________________

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4.0 APPROVED VENDOR LIST

The Seller shall supply motors as manufactured by the vendors listed below and of the class listed for them. No exceptions will be allowed.

Manufacturer	Class of Motor: Std Mill & Chemical Industry Type	Class of Motor: Energy Efficient Mill & Chem Ind Type
Allis Chalmers	Severe Service Process Type RGZ-CH	--
Baldor	Chemical Process Motor Type CP (NEMA Frame 254T and Larger - All cast iron)	--
Delco Division of General Motors Corp.	Chemical Industry Type CIM	--
General Electric Co.	Custom Severe - Duty Type K	Type K Severe Duty Energy Saver to 150 HP
Gould	Severe Duty Type SC	E-Plus Severe Duty Type SCE @ 1800 RPM to 25 HP
Louis Allis	Pacemaker and Armor Line Severe Duty Motors	Spartan Pacemaker Severe Duty
Reliance	Duty Master XT (Extra Tough)	High Performance Duty Master XT
Toshiba	Type ACI with all Cast Iron T. Box, Fan Guard & Fan	--
U.S.Electrical Motors	Corro-Duty	Energy Efficient Corro-Duty
Westinghouse Electric Corporation	Type MAC	MAC II High Efficiency

LINDE DIVISION - GS-1

GENERAL REQUIREMENTS DATA SHEET

PROJECT: 7002 LOCATION: E. Chicago, IN ES NO: 6893 Rev.1 REQ NO: 50012

INFORMATION REQUIRED	SELLER DRAWING NAME/NUMBER*	WEEKS ARO **
Drawing Index List		
Equipment Data Sheets		
Motor Data Sheets		2 Months
Heat Exchanger Data Sheets		
Recommended Spare Parts List (Priced)*		
Auxiliary Power Requirements		
Std. Installation & Erect. Procedures		12
General Arrangement (All Connections)		4
Gen. Arrgt. (Gas & Water Connections)		
Gear Box Details		
Foundation Details		4
Coupling Details		4
Control Panel Details		
Tabulated Operating Clearances		
Loading Diagram - Foundation		4
Allowable Loading - Major Connections		4
Performance Curves - Expected*		
- Actual (Test)		
Process Gas Diagram		
Seal Drawing		8
Cooling Water Diagram		4
Lube Oil Diagram		
Wiring Diagram		
Control Schematics(Pneu., Elect., etc.)		
Instrumentation List		
Lube Oil Arrangement		
Detail Construction Drawings		
Cross Section		
Bill of Materials in Contact with O_2		
Samples for Test in Contact with O_2		
Cleaning Specifications		
Shipping Weights and Dimensions		
Complete Parts List		
Preliminary Inst. Manual (GS-2;2-1)		
Instruction Manual (GS-2;2-2)		12
Test Reports		
Code Certificate - Data Report		

*Required with Quotation.

**Required date for Seller drawings to be in-house at Linde Division, Tonawanda, weeks after receipt of order, based upon a purchase order date of February 22, 1980.

NOTES: 1.) See [illegible] PUR-101, 103, 104, 106, 107, 111 or 112 for mailing address and quantities.

2.) Seller shall complete the "Seller Drawing Name/Number" column stating where the required information shall be found, either on a drawing or separate submittal. Associated with this is a "Certified" drawing or transmittal date in weeks after receipt of purchase order. Any additional drawings or information should be listed in the space provided. This information is required with the quotation.

3.) Where one drawing serves several functions, identify the drawing name or number with each function, (e.g. one drawing could show General Arrangement as well as Foundation Details).

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UNION CARBIDE CORPORATION
Linde Division
Tonawanda, New York

GENERAL REQUIREMENTS SPECIFICATION

Standard Specification GR-1
Revised 12/24/68

NOTE: For Special Requirements on Equipment Purchased for ' Service, See Section 4.0

1.0 SCOPE

This specification outlines the general requirements of Union Carbide, Linde Division, for the purchase of major equipment.

2.0 PROPOSALS (General Requirements)

Seller shall state in his quotation all exceptions to the Buyer's specifications, data sheets, notes, and other attachments to the inquiry. Buyer assumes that Seller will furnish all items strictly in accordance with the requirements of the inquiry for the price stated, unless exceptions are specifically listed.

3.0 DRAWINGS AND DATA REQUIREMENTS

3.1 General

The Seller's drawings and other data shall be submitted for Buyer's approval on or before the date specified on the attached, "General Requirements Data Sheet." If the Seller cannot meet the specified dates, he shall immediately notify the Buyer of his proposed submittal dates. The purpose of the GR-1 Data Sheet is to identify, expedite and assure the completeness of Seller's drawings and data. The number of copies of the required data are set forth in one of the attached Buyer's forms, "Instructions for Submissions of Drawings and Other Data" (Form PUR-101, PUR-103, PUR-104, PUR-106, PUR-107, PUR-111). After Buyer's drawing approval the Seller shall not change layout or dimensions of equipment without Buyer's written agreement.

Buyer's equipment purchase order number, project number and equipment number shall appear on all drawings, data sheets, performance curves, calculations, parts lists, instruction books, packing list or other material submitted by the Seller

3.2 Drawing Information

The drawings and data submitted by the Seller shall be of sufficient detail to enable the Buyer and/or his contractors to prepare complete construction drawings for the installation and integration of the Seller's equipment into the Buyer's plant. The information supplied shall give equipment outlines, bolting, weights, connections and other normal details and include sufficient detail for important considerations such as operator safety, operating space and maintenance clearances.

As a minimum, the Seller shall provide the drawings and data specified on the "GR-1 General Requirements Data Sheet." The first submittal of information from the Seller shall include the "Drawing Index List" which lists all of the individual drawings and pieces of data that the Seller supplies to the Buyer for the specific project. If this list includes drawings or data not specifically listed and scheduled on the "General Requirements Data Sheet," the Seller shall state his intended submittal date so that the Buyer can determine if such dates are compatible with the project schedule.

The drawings and data listed on the "GR-1 General Requirements Data Sheet" are determined for the specific equipment on a specific order. In general, the following details are required where applicable:

Size - Overall and major component length, width and height referred to a common reference point such as a shaft centerline, base, foundation bolt or datum line.

Clearance - Designate operating areas (control panels, control valves, etc.), operating space, platform requirements, maintenance space (pulling dimensions, etc.), crane hook height and travel, location of hot surfaces or moving parts requiring shielding for operator protection.

Loading - Static weights both unloaded and loaded (empty/full, dry/wet, etc.) and their distribution at mounting points, dynamic loads with their direction and points of application; center of gravity, equipment and component weights for minor and major maintenance; load criteria for wind, seismic, snow and other loads.

Foundation - Recommended foundation layout with detail dimensioning of openings, pits, sumps and piers, size, type, location, projection and grip of foundation bolts; grout thickness and recommended type of grout, location of sole plates, floor channels or sub-bases. Note: The Buyer shall supply all foundation bolts except where otherwise agreed.

Piping - Type, size, material, location and identification of all points requiring connection by the Buyer for equipment operation and type, size and location of all drains and vents. All requirements for field welding shall be shown on appropriate drawings. Maximum allowable moments and forces and their direction shall be stated for each major piping connection. A Seller's statement of zero allowable moments or forces on a connection is unacceptable.

Electrical - Size and location of all points requiring connection by the Buyer for equipment operation including grounding lugs. Voltage, wattage, cycles, number of phases or other electrical characteristics shall be designated. All terminal points shall be identified with numbers corresponding with those on the wiring diagrams furnished. Motor drawings shall state amperage requirements.

Instrumentation - Type, size, location and function of all tie in points. All instruments furnished shall be provided with identification numbers; their location shall be shown on the equipment and the Seller shall provide an instrument list tabulating each piece furnished.

Lubrication - All lubricants required shall be listed as to type, properties, make-up rates and quantity. A list of three or more acceptable products of each type shall be furnished. Where the Buyer must supply the oil piping, such as between a machine and a lube oil console, the Seller shall recommend minimum line sizes, slope of lines and location of console.

Outdoor Installation - Need for Buyer installed weather hoods, insulation, tracing or other special protection shall be stated by the Seller.

Shipping - Seller shall state the method of transportation and the degree of disassembly in shipping. He shall state the number and size of the disassembled parts so that the Buyer may make proper arrangements for handling and assembly of components. All requirements for field welding of the assembly shall be stated. Locations of lifting lugs and need for spreader bars or other special rigging shall be shown on the Seller's drawings.

LINDE DIVISION-GS-1

4.0 SPECIAL REQUIREMENTS FOR EQUIPMENT PURCHASED FOR OXYGEN SERVICE

Special procedures which must be followed in connection with equipment to be supplied for oxygen service are covered in Linde Standard Specification GS-35.

5.0 SPARE PARTS

5.1 Recommended Spare Parts Lists

The Seller shall submit a list of recommended spare parts for startup and one year's operation. This list shall contain:

1. Recommended quantity of each part or assembly.
2. Weight (net and shipping).
3. Price and delivery time.

This information is required by the date specified on the "General Requirements Data Sheet."

5.2 Complete Parts Lists

The Seller shall submit complete spare parts lists for items requiring replacement after long use. Complex assemblies shall be shown on parts drawings giving nomenclature, parts numbers and location. The lists and drawings shall bear the following information:

1. Equipment identification (for which part is intended).
2. Part number (actual Manufacturing No.).
3. Part description, including material.
4. Buyer's equipment purchase order number.
5. Price and delivery time.

These lists may include sub-assemblies, rather than individual parts. The complete parts list is required by the date specified on the "General Requirements Data Sheet."

6.0 INSPECTION

The material shall be subject, in whole or in part, to inspection by the Buyer, or his agent, at any time during manufacture and before shipment. Final acceptance will be made only after receipt at job site. Inspection release will only be given through the Buyer.

The Seller shall notify the Buyer when the order is ready for any specified inspection. Notification to the following address by letter or telegram shall be made at least five (5) days in advance

UNION CARBIDE CORPORATION
Linde Division
P. O. Box 44 - Tonawanda, New York 14150
Attention: Purchasing Agent (Telephone - 716-877-1600)

Notification of inspection shall state the following:

1. When and where material will be ready for inspection.
2. What portion of the order is ready.
3. Buyer's purchase order number.

7.0 EXPEDITING

The equipment, drawings, data, and requested procedures are subject, in whole or in part, to expediting by the Buyer or his agent at any time during manufacture and before shipment.

UNION CARBIDE CORPORATION
Linde Division
Tonawanda, New York

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Standard Specification CS-2
Revised 1/15/68

CS-2: GENERAL SPECIFICATIONS FOR INSTRUCTION MATERIALS

1.0 PURPOSE

This specification is to establish the requirements of the Buyer regarding Instruction Materials to be furnished by the Seller.

2.0 SCHEDULE

1. Within 30 days after order for equipment is placed, The Seller shall transmit, for comments and approval, two copies of preliminary or rough draft of Instruction Materials for subject equipment, or typical instruction materials for similar type of equipment, unless an exception is made in the order.

Seller is not expected to furnish final clearances and dimensions with his initial transmittal, but shall present the scope and format of the proposed final documents.

2. After review by the Buyer, the Seller shall revise, as necessary, the preliminary issues and resubmit for final approval two copies of Instruction Materials, at least 60 days prior to final shipment of the subject equipment.

3. After final approval, the Seller shall transmit the specified number of copies of all final approved Instruction Materials, on or before 30 days prior to final shipment of equipment. All transmittals of Instruction Materials shall be sent to:

UNION CARBIDE CORPORATION
Linde Division
Marketing Communication Department
P. O. Box 44
Tonawanda, New York 14150
Attention: Mr. Lynn A. Yeomans

Copies of all correspondence and transmittal letters shall be forwarded to:

UNION CARBIDE CORPORATION
Linde Division - Purchasing Department
P. O. Box 44
Tonawanda, New York 14150
Attention: Expediting Manager

3.0 CONTENTS

3.1 The Instruction Materials required shall consist of a complete engineering description of construction, installation, operation, and maintenance of all equipment supplied by the Seller. Detail break-down as follows:

1. Complete information for installation consisting of foundation, grouting, alignment, location of all customer connections and supports, diagrammatics of all main and auxiliary systems, and information as to the settings (values) of instruments.

2. Design operating conditions consisting of temperatures, pressures, speeds, (normal and critical), diameters, and clearances, including design points and their limits of variations. All projected performance data shall be revised to reflect any shop test information. (This may be furnished later as an Appendix.)

3. Physical and functional description of all equipment including all auxiliary equipment, and weights of all major components.

4. Complete manufacturer's recommended procedures (step-by-step) for pre-start, startup, normal operation, and shutdown. Also recommended procedures for other than normal operation, such as reduced capacity, and also emergency shutdown.

5. Complete maintenance instructions for all components including disassembly procedures, clearance checks, reassembly, setting dimensions and adjuutments, including all necessary detail drawings with sections. At least one cross-section should be included. A complete "trouble-shooting" procedure listing possible troubles with checks to be made and corrective action to be taken. All special tools required shall be listed clearly.

6. Recommended Ordering Procedure for spare parts including a complete parts list and necessary component detail drawings.

3.2 All drawings shall be reduced to either 8½ x 11 or 11" x 17" size and bound in the manual. In cases where drawings are of such size that this reduction would cause them to become unreadable, they may be submitted full size, folded and inserted into individual envelopes. The envelopes shall in turn be bound into the manual and clearly marked with title and number of enclosed drawings.

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UNION CARBIDE CORPORATION
Linde Division

ENGINEERING DEPARTMENT
Tonawanda, New York

CS-21: ROTATING PUMP SPECIFICATION

1.0 SCOPE

1.1 Purpose: This specification establishes the minimum requirements for rotating pumps purchased by the Linde Division. All rotating pumps shall conform to this specification and the accompanying data sheet(s) unless the Seller secures written approval from the Buyer for any exceptions.

1.2 Guarantee: All equipment furnished shall be guaranteed by the Seller to meet the specified flow and horsepower at the design point as indicated in Paragraph 2.1 of this specification. The Seller shall be responsible for the guarantee of all equipment, accessories and parts sub-contracted by the Seller that are furnished under this specification.

2.0 PERFORMANCE

2.1 Guarantee Point: The pump shall be guaranteed to deliver the specified discharge pressure (-0%) at the design flow with the specified suction conditions and within the design brake horsepower (+5%). The horsepower, capacity, suction conditions and discharge pressure at design conditions are specified on the attached pump data sheet. The discharge pressure specified is at the discharge flange.

2.2 Performance Curve: The Seller shall provide certified curves showing total head, brake horsepower, efficiency, and NPSH or submergence requirements versus flow. The Seller shall also indicate on the curves the total head and BHP for the maximum and minimum diameter impellers applicable to the pump assembly offered.

2.3 Performance Test: If the Seller does not conduct a performance test of the pump in his shop, the Buyer may make a performance test of the pump at his installation. The test, instrumented and supervised by the Buyer, shall be conducted to determine if the pump meets its guaranteed performance (Para. 2.1). The method of test shall be mutually agreed to by both the Buyer and Seller. The Seller shall have the right to review the test installation and witness the test.

3.0 CONSTRUCTION

3.1 General Specification: The pump assembly shall include the pump, strainer, baseplate, necessary couplings, gears, guards, shafting, driver, special tools and wrenches, etc. as specified on the attached data sheet. All materials shall be as specified on the data sheet.

3.2 Casing: The pump casing shall be of cast iron, or as specified on the attached data sheet.

3.3 Impellers: The impellers shall be designed for minimum hydraulic thrust. For this s ification, impeller refers to the rotating element.

3.4 Shafting: Line shafting, when required, shall be adequately supported by an intermediate bearing.

3.5 Column Pipe: The column pipe, when required, shall be designed to minimize the friction loss between the inlet and the discharge connections.

3.6 Strainer: When required, a basket type strainer shall be furnished for sump installation and of the cone type for well installations unless otherwise specified on the attached data sheet. The material shall be bronze unless otherwise specified on the attached data sheet.

3.7 Thrust: The pump shall be designed to minimize total thrust. For vertical pumps, the driver or power transmission device shall be rated for a total thrust equal to or greater than the highest total thrust load possible when operating at any condition from shut-off to maximum pump capacity.

3.8 Pressure Bolting: All pressure bolting shall conform to ASTM Specifications A-193 Grade B-7 for studs and A-194 Grade 2 for nuts.

3.9 Flanges and Connections: All ozzles 1-1/2" or larger shall be flanged. Flanges shall conform to ASA Specification B-16.

3.10 Piping Assembly: Seller's piping assembly shall be fabricated with labor compatible to Buyer's requirements.

4.0 TESTS

4.1 Shop Test: As a minimum, Seller shall perform a hydraulic test of the pump in his shop. A witness test, when required, will be specified on the attached data sheet.

4.2 Hydrostatic Test: All pressure casings shall be hydrostatically tested at 1.5 times the design pressure.

5.0 CLEANING AND PREPARATION FOR SHIPMENT

All components shall be shipped suitably protected and free of all dirt, scale, grease, moisture or any other foreign matter. Light coatings of petroleum preservatives will be permissible for corrosion protection unless otherwise specified on the attached data sheet.

UNION CARBIDE CORPORATION
Linde Division
Tonawanda, New York

MAJOR EQUIPMENT SUPPLIER

INSTRUCTIONS FOR SUBMISSION OF DRAWINGS FOR APPROVAL AND OTHER DATA

In accordance with this Purchase Order, the Seller is required to submit drawings and other data in accordance with the schedule below:

A. Four (4) Copies:	All correspondence including transmittal letters and bulletins.
B. One (1) Reproducible (sepia) and Five (5) Prints	Certified drawings for Buyer's approval.
C. One (1) Reproducible (sepia) and Five (5) Prints	Final certified approved drawings for Buyer's files.
D. Four (4) Copies:	Data reports on all pressure vessels falling under ASME unfired pressure vessel jurisdiction including National Board of Boiler and Pressure Vessel Registration Number.
E. Four (4) Copies:	Recommended Spare Parts Lists with Prices.

All of the above to be forwarded to:

UNION CARBIDE CORPORATION
Linde Division
P.O. Box 44
Tonawanda, New York 14150
Attention: Vendor Print Control

One Copy of Transmittal Letter Only to be forwarded to:

UNION CARBIDE CORPORATION
Linde Division
P.O. Box 44
Tonawanda, New York 14150
Attention: Purchasing Department

F. Eight (8) Sets	Final approved instruction material in accordance with Linde Division Standard GS-2.

All instruction material shall be forwarded to:

UNION CARBIDE CORPORATION
Linde Division
Marketing Communications Department
P.O. Box 44
Tonawanda, New York 14150

NOTE I: "Reproducible (sepia) must be of good quality to enable Buyer to make legible prints. If sepia quality is not acceptable, sepia and all drawings will be returned for a new submission. Original submission will not be considered as a contractual submission.

NOTE II: All transmittal letters, bulletins and drawings must show the purchase order number and project destination.

NOTE III: All drawings must show shipping weight and equipment operating weight.

FORM PUR-101 (Issued 4/15/64, REVISED 5/9/77)

Solar Grade Silicon

Painting Instructions

The instructions below shall be used to establish painting procedures for all equipment used in Project Number 7002.

	Vessel Operating Temperature	
	Less Than 350°F	350°F and Greater
Type	Catalyzed Epoxy-Phenolic	Silicone
Manufacturer	Wisconsin Protecting Co.	PPG
Name	Plasite 7122-H	Hi-Heat UC-45732
Surface Preparation	SSPC-SP-6	SSPC-SP-1 Solvent Clean
Number of Coats	2	2
Wet Film Thickness per Coat, Max.	12 Mils	3-5 Mils
Dry Film Thickness per Coat, Min.-Max.	6-7 Mils	1½-2 Mils
Application Method	Spray or Airless Spray	Brush or Spray
Mixing and Thinning	SSPC-PA-1 and Manufacturer's Instructions	SSPC-PA-1 and Manufacturer's Instructions
Application	SSPC-PA-1 and Manufacturer's Instructions	SSPC-PA-1 and Manufacturer's Instructions

Note: In general, most vessels operating at temperatures of 350°F or greater will be of stainless steel construction. However, any carbon steel vessels in this class require SSPC-SP-6 surface preparation rather than SSPC-SP-1, solvent cleaning.

KA Kuberka 2-19-80 | P-7002 E. Chicago, IN | Pumping Therminol - Item 02 | WG Bancroft, 2-21-80

#466-02

UNION CARBIDE CORPORATION

LINDE DIVISION
P. O. BOX 44, TONAWANDA, N. Y. 14150
HEREAFTER CALLED "BUYER"
MAIL INVOICE IN TRIPLICATE & BILL OF LADING TO ABOVE ADDRESS, ATTN: ACCOUNTS PAYABLE UNLESS OTHERWISE NOTED.

DATE WANTED ON SITE	VENDOR SHIPPING PROMISE
1-1-81	

PURCHASE ORDER 825 50012 U

PAGE 1 OF 1

THIS NUMBER MUST APPEAR ON ALL INVOICES, PACKAGES, CASES, BILLS OF LADING, ETC.

VENDOR CODE 237

Goulds Pumps
3435 Harlem Road
Cheektowaga, New York 14225

HEREINAFTER CALLED "SELLER"

SHIP TO
[ ] TRUCK & EXPRESS - 175 EAST PARK DRIVE, TONAWANDA, N.Y.
[X] P-7002
Shipping Instructions to follow

EXHIBIT D

THIS ORDER NOT BINDING UNLESS UCC VALIDATION APPEARS

BUYER HEREBY ORDERS SUBJECT TO THE TERMS AND CONDITIONS HEREIN CONTAINED, INCLUDING THOSE ON THE REVERSE SIDE HEREOF.

TERMS OF PAYMENT: NET 30	RATE % AND TERMS	TRANSPORTATION TERMS	ACCOUNT OF BUYER	ACCOUNT OF SELLER	OTHER TERMS/OR SPECIAL INSTRUCTIONS	F.O.B. SHIPPING POINT	DEST.	SPECIFY	SHIP VIA P.P.	TRUCK	RAIL FGT.	OTHER
X			1	X 2	3	X		Seneca Falls		X		

ITEM NO.	COMM. CODE	ACCOUNT NUMBER	ORDER QUANTITY	U/M	DESCRIPTION	UNIT PRICE	AMOUNT
1	2650F	C885-000-7002-242	1	ea	Goulds pump, Model 3196 MT size 1 1/2 x 3 -13 as modified by Buyer's Specification (hereinafter called "Specification") ES 6893, Rev. 1 dated 2-19-80 (hereinafter called "Equipment") attached hereto and made a part hereof.	$2897.00	$2897.00
					Drawings and Other Data: Drawings for approval are required in accordance with Buyer's Form PUR 101 attached hereto and made a part hereof, not later than noted on GS-1 data sheet. Operating and instruction manuals are required in accordance with Form PUR 101. Instruction Material Specification GS-2 applies.		
					Warranty: The warranty of this Equipment shall be in accordance with Attachment "A", dated 3-5-79, entitle- "Warranty", which is attached hereto and made a part hereof. The Quality clause on the reverse side hereof does not apply.		

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SAMPLE PURCHASE ORDER FORM (Hot Oil Pump)

NOTE: "Where transportation is for account of Buyer on shipments subject to Item 33200 of Uniform Freight Classification No. 6 (rail) and/or Item 33465 of National Motor Classification No. 15 or Item 60000 of National Motor Classification No. A-6, or successive issues thereof, the released value of the shipment shall be entered on the bill of lading at not exceeding 50 cents per pound in the manner prescribed in such items."

[ ] ACKNOWLEDGE ONLY IF SHIPPING DATE OR OTHER TERMS OR CONDITIONS OF THIS ORDER ARE NOT ACCEPTABLE.

[ ] ACKNOWLEDGE BY DATE________

[ ] This order is subject to a State Tax for________ and/or local Sales/Use Tax at a rate of______%.

[X] This order is exempt from State and/or local Sales/Use Tax. See exemption certificate.

[ ] THIS ORDER SUPERSEDES ORAL ORDER TO________ ON________ BY________

EXPEDITOR NOTE: REQUIRED ON SITE NO LATER THAN________

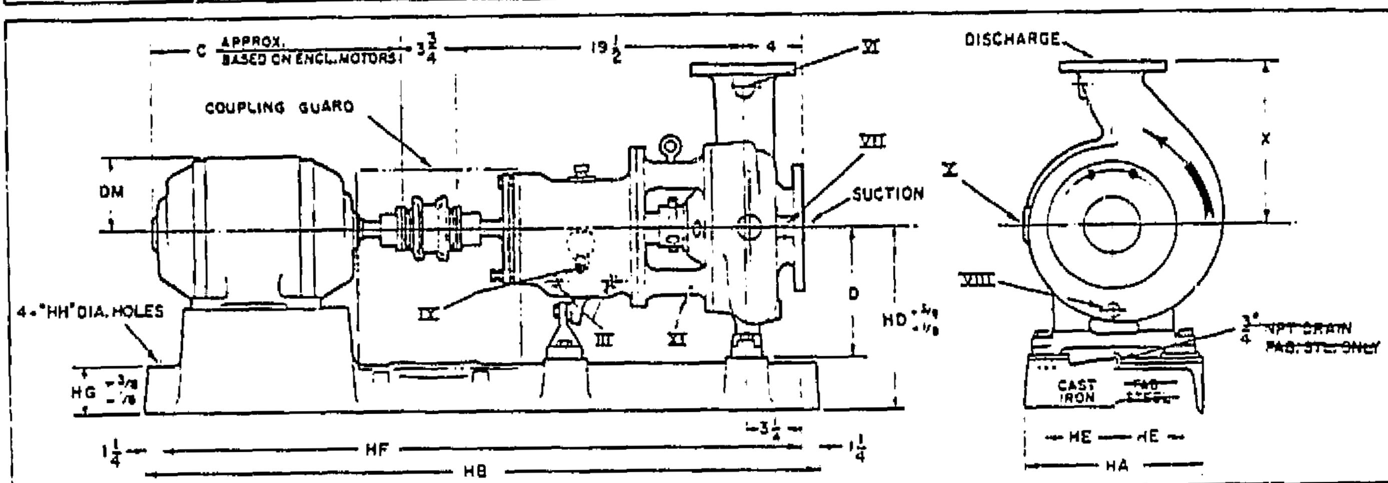
ADDRESS QUESTIONS TO PURCHASING DEPT.: RJ Serrianne

STATUS OF THIS DRAWING
- [ ] Approved. Proceed with fabrication per this drawing
- [x] Approved as noted. Proceed with fabrication per this dwg. Revise & resubmit by
- [ ] Not Approved. Revise and resubmit by
- [ ] Approval Not Required
- [ ] Certify Dwgs. Show Unde P.O. #, Proj. #, Date of Cert.

Approval of drawings, wholly or in part, does not ...

EXHIBIT E SAMPLE CERTIFIED VENDOR DRAWING

GOULDS PUMPS INC. ENGINEERED PRODUCTS DIVISION | MODEL 3196 "MT" | STANDARD BEDPLATES — RIGHT HAND ROTATION



DIMENSIONS DETERMINED BY MOTOR				
MOTOR FRAME SIZE	C	DM	WT INCL. CPLG.	BEDPLATE NO.
143T	12 1/2	3 1/2	40	1
145T	13 1/2	3 1/2	45	1
182T	14 1/2	5	65	1
184T	15 1/2	5	75	1
213T	18	5	150	1
215T	19 1/2	6	160	1
X 254T	22 1/2	6 1/2	255	2
256T	24	6 1/2	280	2
284T	25 1/2	7 1/2	375	2
284TS	24 1/2	7 1/2	345	2
286T	27	7 1/2	430	2
286TS	26	7 1/2	380	2
324T	28 1/2	9	575	3
324TS	27	9	485	3
326T	30	9	640	3
326TS	28 1/2	9	570	3
364T	33	9 1/2	810	3
364TS	31	9 1/2	755	3
365T	34	9 1/2	950	3
365TS	32	9 1/2	865	3
404TS	34	11	1080	4
405TS	35 1/2	11	1110	4
444TS	38 1/2	12 1/2	1510	4

DIMENSIONS DETERMINED BY PUMP						
ANSI NO.	DISCH SIZE	SUCT SIZE	CAS'G CLASS	D	X	WT
A10	2	3	6	8 1/4	8 1/4	180
△A50	2	3	8	8 1/4	9 1/2	200
A70	3	4	8	8 1/4	11	220
△A70	3	*4	8	8 1/4	11	220
A05		2	10	8 1/4	8 1/2	205
A50	1 1/2	3	10	8 1/4	8 1/2	220
A60	2	3	10	8 1/4	9 1/2	230
A70	3	4	10	8 1/4	11	265
	4	■6	10	10	13 1/2	305
X A20	1 1/2	3	13	10	10 1/2	245
A30	2	3	13	10	11 1/2	275
A40	3	*4	13	10	12 1/2	330
	4	■6	13	10	13 1/2	405

* SUCT. FLG HAS 8 - 3/4 - 10 UNC TAP FOR 300 LB CASING ONLY

■ SUCT FLG HAS 8 - 3/4 - 10 UNC TAP FOR 150 LB. CASING, 12 - 3/4 - 10 UNC TAP FOR 300 LB CASING.

△ HOLES ARE NOT TAPPED FOR GREASE LUBE

● 3X4-8 & 3X4-13 1/4" PIPE TAP 4X6-10 & 4X6-13, NO TAP

▲ DIMENSIONS DETERMINED BY BEDPLATE												
NO.	USED WITH MOTOR FRAME	HA	HB	HD D=8 1/4	HD D=10	HE	HF	HG	HH	WEIGHT IRON	WEIGHT STL.
1	143-215	12	45	12	13 3/4	4 1/2	42 1/2	3 3/4	3/4	105	130
X 2	254-286	15	52	[redacted]	14 1/2	6	49 1/2	4 1/8	3/4	155	[redacted]
3	324-326	18	58	13	14 3/4	7 1/2	55 1/2	4 3/4	1	205	275
3	364-365	18	58	14	14 3/4	7 1/2	55 1/2	4 3/4	1	205	275
4	404-405	18	60	15	15	7 1/2	57 1/2	4 3/4		240	285
4	440 SER	18	60	16	16	7 1/2	57 1/2	4 3/4	1	240	285

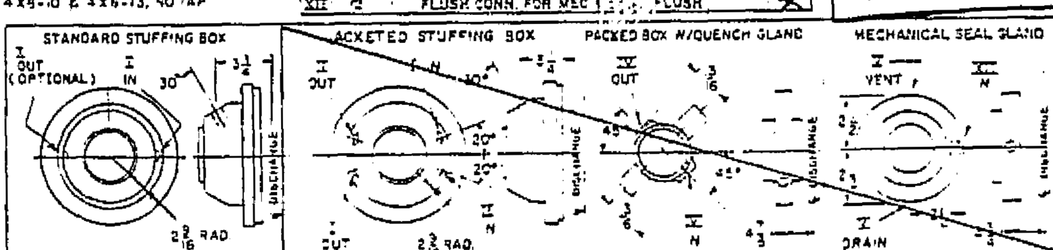
INSTALL FOUNDATION BOLTS IN PIPE SLEEVES. ALLOW 3/4" TO 1 1/2" FOR GROUTING. SEE INSTRUCTION BOOK FOR DETAILS.

▲ FABRICATED STEEL HAS DRIP PAN UNDER PUMP WITH 3/4" DRAIN. STANDARD CAST IRON HAS NO DRAIN PROVISIONS.

TAPPED OPENINGS					
NO.	SIZE	QTY.	PURPOSE	FURNISHED YES	FURNISHED NO
I	3/8	1	LANTERN RING OR MECH. SEAL FLUSH	X	
II	3/8	2	STUFFING BOX JACKET	X	
III	1/2	4	BEARING FRAME COOLING JACKET	X	
IV	1/4	2	QUENCH CONN. FOR PACKED BOX		X
V	1/2	2	VENT & DRAIN FOR MECH. SEAL		X
VI	3/8	1	DISCHARGE GAGE CONNECTION		X
VII	● 3/8	1	SUCTION GAGE CONNECTION		X
VIII		1	CASING DRAIN 3/8 1/2	X	X
IX	3/8	1	OIL DRAIN - OIL LUBE	X	
X	1/2	1	BY-PASS CONNECTION	X	X
XI	1	1	FRAME ADAPTER DRAIN	X	
XII	1/2	1	FLUSH CONN. FOR MECH. SEAL FLUSH		X

CERTIFIED DRAWING
Approved for Construction Purposes
GOULDS PUMPS, Inc.
SENECA FALLS, N.Y.
MAR 31 1980
SIGNED [signature]

FLANGE DIMENSIONS					
NOM. I.D.	O.D.	B.C.	THK.	NO OF HOLES	SIZE OF HOLES
150 LB FLANGE - FLAT FACE					
1	4 1/4	3 1/8	9/16	4	5/8
1 1/2	5	3 7/8	11/16	4	5/8
2	6	4 3/4	3/4	4	3/4
3	7 1/2	6	15/16	4	3/4
4	9	7 1/2	15/16	8	3/4
6	11	9 1/2	1	8	7/8
300 LB FLANGE - FLAT FACE					
1	4 7/8	3 1/2	11/16	4	3/4
X 1 1/2	6 1/8	4 1/2	13/16	4	7/8
2	6 1/2	5	7/8	8	3/4
X 3	8 1/4	6 5/8	1 1/8	8	7/8
4	10	7 7/8	1 1/4	8	7/8
6	12 1/2	10 5/8	1 7/16	12	7/8



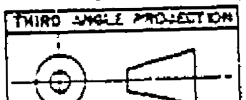
NOTES:

DRAWING IS NOT TO SCALE.

ALL DIMENSIONS ARE IN INCHES.

ALL WEIGHTS ARE APPROXIMATE AND IN POUNDS. ...

THIRD ANGLE PROJECTION



OPTIONS FURNISHED WHEN CHECKED		
BEDPLATE	X	CAST IRON
		FABRICATED STEEL
COUPLING GUARD FURNISHED BY	X	GOULDS
		OTHERS
STUFFING BOX COVER		STANDARD
	X	JACKETED
STUFFING BOX SEALING		PACKING WITH QUENCH GLAND
		MECHANICAL SEAL WITH FLUSH GLAND
		MECHANICAL SEAL WITH VENT & DRAIN GLAND
	X	MECHANICAL SEAL WITH PLAIN GLAND

GOULDS PUMPS INC. SENECA FALLS, N.Y. U.S.A.

GOULDS SERIAL NO. 708C196

CUSTOMER UNION CARBIDE CORP.

CUSTOMER P.O. NO. 825-500.17 U

ITEM NO. 466-02 (TAG)

SERVICE THERMINOL 60 CIRCULATION

ENG FORM NO. ... DATE 1-4-80

DWG. NO. 105-23 1/3

CE... OF POOR ...

STATUS OF THIS DRAWING

- ☐ Approved: Proceed with fabrication per this drawing
- ☒ Approved as Noted: Proceed with fabrication per this dwg. Revise & resubmit by 6/6/80
- ☐ Not Approved: Revise and resubmit by____
- ☐ Approval Not Required
- ☐ Certify Dwgs: Show Linde P.O. #, Proj. #, Date of Cert.

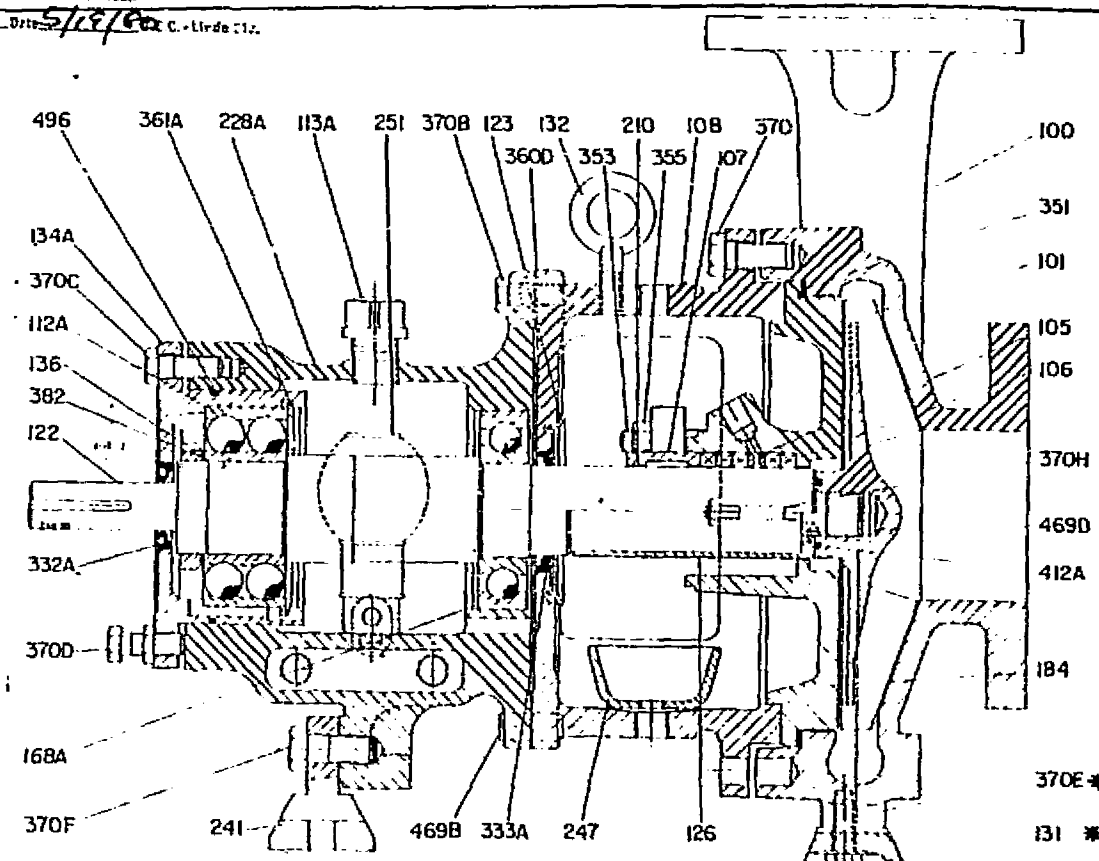
Approval of drawings, wholly or in part, does not relieve vend r's responsibility in complying with applicable specifications and contract.

By Engr. CAS Date 5/19/80 C. Linde Div.

A-21

BILL OF MATERIAL

ITEM NO	QTY PER PUMP	PART NAME	MATERIAL
100	1	CASING	DI
101	1	IMPELLER	DI
~~105~~	~~2~~	~~LANTERN RING HALF~~	~~TEFLON~~
~~106~~	~~1~~	~~STUFFING BOX PKG~~	~~ASBESTOS~~
~~107~~	~~1~~	~~GLAND QUENCH~~	
108	1	FRAME ADAPTER	CI
112A	1	BALL BRG OTBD	STEEL
113A	1	BREATHER	STEEL
122	1	SHAFT	STEEL
123	1	DEFLECTOR	GL & NYL
126	1	SHAFT SLEEVE	316SS
131	1	PUMP FOOT	CI
132	1	EYEBOLT	STEEL
134A	1	BRG HOUSING	CI
136	1	BRG LOCK NUT	STEEL
168A	1	BALL BRG INBD	STEEL
184	1	STUFF BOX COVER	DI
~~210~~	~~1~~	~~PACKING GLAND~~	~~ASBESTOS~~
228A	1	FRAME	CI
241	1	FRAME FOOT	CI
~~247~~	~~1~~	~~DRIP BASIN~~	
251	1	SIGHT OILER	GL & STL
332A	1	OIL SEAL OTBD	BUNA RUB
333A	1	OIL SEAL INBD	BUNA RUB
351	1	GASKET CASE	AFR ASB
353	2	STUD GLAND	316SS
355	2	NUT GLAND STUD	304SS
360D	1	GASKET FR ADPTR	VELLUMOID
361A	1	RETG RING BRG HSG	STEEL
370	8	H CAP SCREW CASE	STEEL
370B	4	H CAP SCREW ADPTR FR	STEEL
370C	3	H TAP BOLT BRG HSG	STEEL
370D	3	H TAP BOLT IMPER ADJ	STEEL
370E	2	H CAP SCREW FOOT	STEEL
370F	1	DOWEL BOLT FR FOOT	STEEL
370H	2	STUD COVER ADPTR	304SS
382	1	BRG LOCK WASHER	STEEL
412A	1	O RING IMPELLER	TEFLON
469D	2	PRCN ALIGN FR ADPTR	ALLOY STL
469B	1	ROLL PIN SLEEVE	410SS
496	1	O RING BRG HSG	BUNA N



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NOTE: IF UNIT IS FURNISHED WITH MECHANICAL SEAL, DELETE ITEMS 105, 106, 107, & 210 AND REFER TO SEAL DRAWING NO. MSCH-1012

☒ ATTACHED

~~TO FOLLOW~~

✱NOTE: ON SIZES 2×3-13, 3×4-13 & 4×6-13, PART #131 IS CAST INTEGRAL WITH CASING.

GOULDS PUMPS, INC. ENGINEERED PRODUCTS DIVISION SENECA FALLS, NEW YORK 13148	SECTIONAL ASSEMBLY	GOULDS SERIAL NO 708C196	DRAWN BY VAJ	CKD BY VAJ	DATE 6-19-78	SCALE NONE
	OIL LUBE	CUSTOMER UNION CARBIDE CORP.	MODEL 3196-MT	DRAWING SA1960LM		REV 0
	MODEL 3196 MT	CUSTOMER P.O. NO 825-50012 U				
		EQUIP. OR ITEM NO 566-02 (TAG)				
		SERVICE THERMINOL CIRCULATION				

[redacted]

☐ Approved: Proceed with fabrication per this drawing
☑ Approved as Noted: Proceed with fabrication per this dwg. Revise & resubmit by 6/16/80
☐ Not Approved: Revise and resubmit by ________
☐ Approval Not Required
☐ Certify Dwgs: Show Linde P.O. #; Proj. #; Date of Cert.
Approval of drawings, wholly or in part, does not relieve vendor of his responsibility in complying with applicable specifications and contract.
By Engr. CAS Date 5/19/80 U.C.C.-Linde Div.

A-22

BILL OF MATERIALS

ITEM NO.	ROTARY UNIT	MATERIAL
1	MOUNTING COLLAR	316 STAINLESS
2	DRIVE SCREW (3)	316 STAINLESS
3	STATIC "O" RING	VITON
4	DYNAMIC "O" RING	VITON
5	SPRING (10)	HAST "C"
6	SEAL RING HOLDER	316 STAINLESS
7	SEAL RING	CARBON
	CODE - DIWI	
ITEM NO	**STATIONARY UNIT**	**MATERIAL**
8	STATIONARY SEAT	TUNG. CARBIDE
9	GASKET (2)	ASBESTOS
11	GLAND	316SS BY GOULDS

GOULDS PUMPS, INC.
SENECA FALLS, NEW YORK USA

CHESTERTON 880
1 3/4 D SHAFT SEAL
FOR MODEL 3196 MT

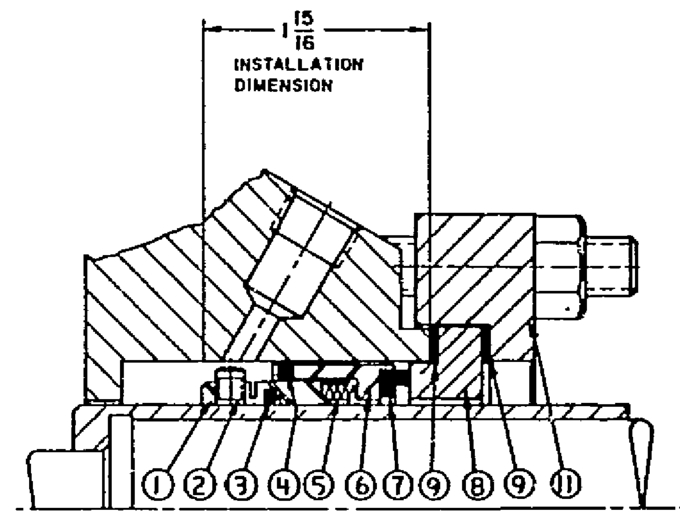

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EXHIBIT E

C-2

CHESTERTON REF # 45812

OPERATING CONDITIONS:
LIQUID THERMINOL 60
SP. GR. .905
TEMP. 311 °/F SPEED 1780 R.P.M.

GOULDS SERIAL # 708C196
CUSTOMER # UNION CARBIDE CORP.
CUSTOMER P.O. # 825-50017 U
EQUIP. OR ITEM # 466-02 (TAG)
SERVICE THERMINOL CIRCULATION

GOULDS PART NO.
ROTARY 92720-0175
SEAT 92720-2000

REV.

1	WAS 770 TYPE 22878 VAV

DRAWING MSCH-1012 REV. 1

... THIS DRAWING
☐ Approved: Proceed with fabrication per this drawing
☑ Approved as Noted: Proceed with fabrication per this dwg. Revise & resubmit by 6/6/80
☐ Not Approved: Revise and resubmit by
☐ Approval Not Required
☐ Certify Dwgs: Show Circle P.O. #, Prct. #, Date of Cert.
Approval of drawings, wholly or in part, does not ...

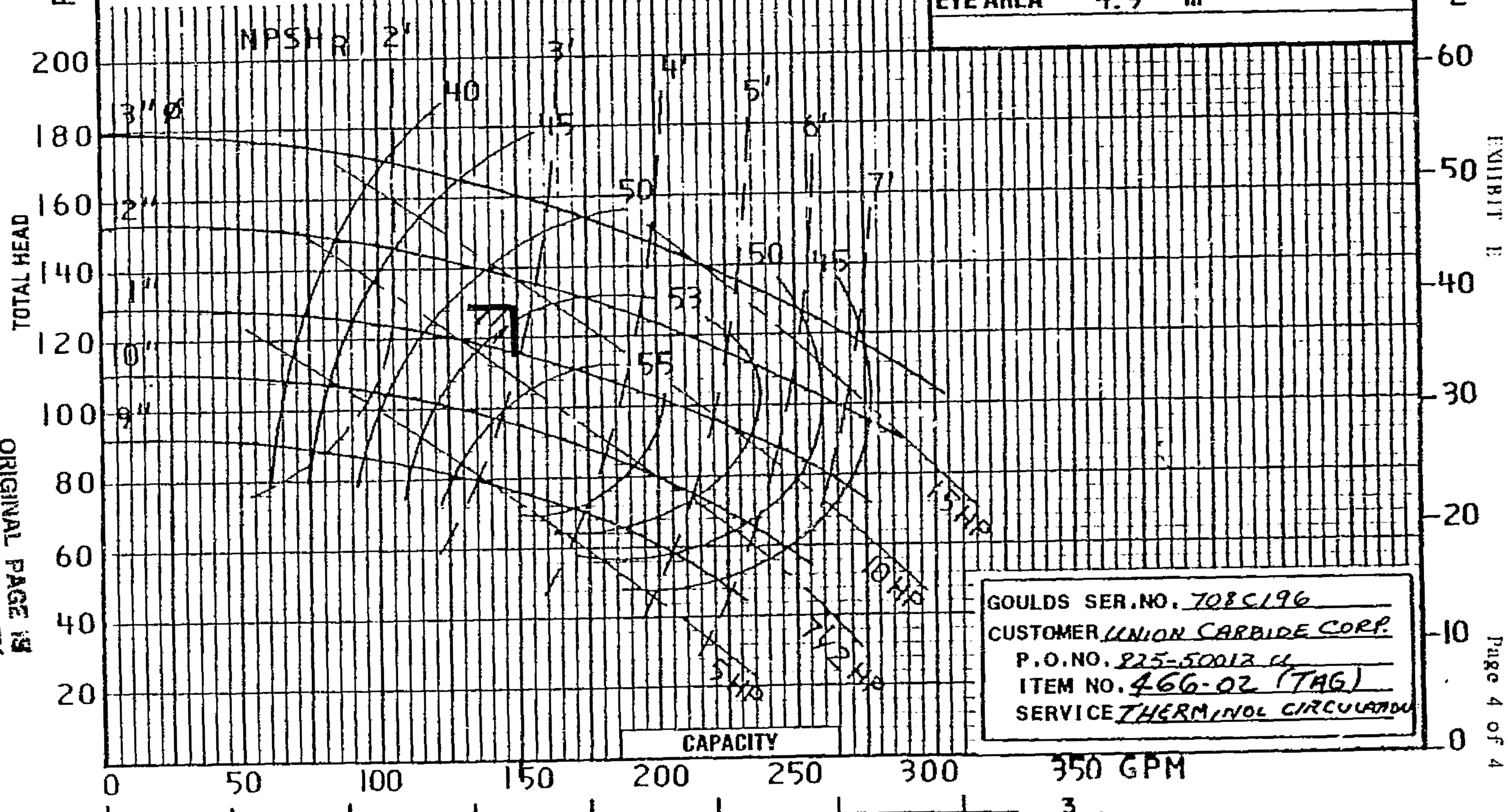
DATE 2-13-63 REV. DATE 12-2-77 ISSUE #5 B.E.F.

GOULDS PUMPS, INC.
SENECA FALLS NEW YORK 13148

CENTRIFUGAL PUMP CHARACTERISTICS

RPM ~~1750~~ 3500 CDS	1680-5
MODEL 3196 MT	11 3/4" IMP.
SIZE 1 1/2 x 3-13	
IMP. DWG. 100-538	STEEL 100-537
PATTERN 53971	53970
EYE AREA 4.9 in²	

CERTIFIED PERFORMANCE DATA
GOULDS PUMPS, INC.
SENECA FALLS, N.Y.
Van ... Johnson



GOULDS SER. NO. 708C196
CUSTOMER UNION CARBIDE CORP.
P.O. NO. 825-50012 a
ITEM NO. 466-02 (TAG)
SERVICE THERMINOL CIRCULATION

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A-23

EXHIBIT E

APPENDIX B

EQUIPMENT PROCUREMENT STATUS

MARCH 31, 1980

APPENDIX B

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
453-04, 05 Waste Gas Induction Blower	50001	✓	✓	✓				
453-02 Agglomeration Blower	50002	✓	✓	✓				
	50003							
	50004							
426-02 Quench Contactor Pump	50005	✓	✓	✓				
436-02 TCS Distillate Pump	50006	✓	✓	✓				
426-04 Cond. Wash Pump	50007	✓	✓	✓	✓			
	50008							
	50009							
423-02, 03 Recycle H_2 Compressor	50010	✓	✓	✓				
4430-2 Pyrolysis H_2 Compressor	50011	✓	✓	✓				
466-02 Hot Oil Pump	50012	✓	✓	✓	✓	✓		
466-04, 05 Cooling Water Pump	50013	✓	✓	✓	✓	✓		
424-02 Quench Condenser	50014	✓	✓	✓				
424-04 434-06, 10 Reboilers	50015	✓	✓	✓				

APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
434-02 Stripper Condenser	50016	✓						
421-12, 16 441-06 Tanks	50017	✓	✓	✓				
464-02, 04 Ventilation Heat Excheners	50018	✓						
434-08, 14, 18 Column Condensers	50019	✓						
	50020							
	50021							
434-12, 16, 24 444-02 Coolers	50022	✓						
	50023							
	50024							
	50025							
	50026							
	50027							
434-26 Refrig. Heating Coil	50028	✓						
	50029							
	50030							

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APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
	50031							
	50032							
411-02, 441-04 461-08 Bins	50033	✓	✓	✓	✓			
	50034							
421-02,04,06,08,10,14,18 451-04,06 Tanks	50035	✓	✓	✓	✓			
	50036							
	50037							
	50038							
	50039							
431-04, 06, 08, 10 435-02, 04 Tanks & Reactors	50040	✓	✓	✓	✓			
	50041							
	50042							
	50043							
	50044							
	50045							

APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
	50046							
	50047							
	50048							
	50049							
	50050							
451-10 Acid Tank	50051	✓	✓	✓				
	50052							
	50053							
	50054							
461-02 Hot Oil Expansion Tank	50055	✓						
425-02 Hydrogenation Reactor	50056	✓	✓	✓	✓			
445-02 Quartz Liner	50057	✓	✓	✓				
	50058							
445-02 Pyrolysis Reactor and Hopper	50059	✓	✓	✓				
417-02 457-04, 06, 08 Filters	50060	✓	✓	✓				

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APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
427-02 Crude TCS Filter	50061	✓	✓	✓				
437-02 Silane Ultra Filter	50062	✓	✓	✓				
	50063							
	50064							
	50065							
	50066							
448-08, 10 Loading Scales	50067	✓	✓	✓				
448-10 Waste Powder Flaker	50068	✓	✓	✓				
448-04 Boule Cart	50069	✓						
448-14 Boule Scale	50070	✓						
	50071							
458-02 Silica Collection Cyclone	50072	✓						
458-04 Silica Drum Packer	50073	✓	✓	✓				
	50074							

APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
459-02, 04 Venturi and Scrubber	50075	✓						
429-04 Superheater	50076	✓	✓	✓				
	50077							
	50078							
	50079							
	50080							
	50081							
	50082							
459-08, 10, 12, 14 Waste Burners	50083	✓	✓	✓				
	50084							
454-04 Silica Agglomerator	50085	✓	✓	✓				
469-02 Cooling Tower	50086	✓	✓	✓				
469-06 Cooling Tower Treatment	50087	✓	✓	✓				
469-12 Refrigeration System	50088	✓	✓	✓				

APPENDIX B

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
469-16 Therminol Heater	50089	✓	✓	✓	✓			
452-02 Muriatic Tailing Column	50090	✓						
456-08 Tailing Column Pump	50091	✓	✓		✓			
469-14 Instrument Air Package	50092	✓	✓	✓				
642-02 MCC	50093	✓	✓	✓	✓			
641-02 Transformer	50094	✓	✓	✓	✓			
	50095							
365-02 Quality Control Trailer	50096	✓	✓	✓				
	50097							
411-08 TL Argon Tank	50098	✓						
461-04, 06 Fuel Oil Storage Tank	50099	✓						
	50100							
466-08, 09, 10, 11 Sump Pumps	50101	✓						
643 Emergency Generator	50102	✓	✓	✓				

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OF POOR Q[illegible]

APPENDIX B

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
463-06, 08 Ventilation Blower	50103	✓						
	50104							
426-06, 07 STC Pumps	50105	✓	✓	✓				
	50106							
For 432-08 Internals for Silane Column	50107	✓	✓	✓				
Chlorosilane Analysis	50108	✓	✓	✓				
UV Spectrophotometer	50109	✓	✓	✓				
Elemental Analysis	50110	✓	✓	✓				
Silicon Melting Furnace	50111	✓	✓	✓				
432-02 Stripper Column	50112	✓	✓		✓			
432-04 TCS Column	50113	✓	✓		✓			
432-06 DCS Column	50114	✓	✓		✓			
459-16 Agitator	50115	✓	✓	✓				
429-02 Quench & Solids Removal Contractor	50116	✓	✓	✓				

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EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
	50117							
	50118							
466-06, 07 Fuel Oil Pumps	50119	✓	✓	✓	✓			
436-04 DCS Distillate Pump	50120	✓						
436-05 Chlorosilane Pump	50121	✓						
432-08 Silane Column	50122	✓						
	50123							
	50124							
	50125							
	50126							
	50127							
	50128							
	50129							
	50130							

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APPENDIX B

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
	50131							
	50132							
	50133							