Production of Aluminum-Silicon Alloy and Ferrosilicon and Commercial Purity Aluminum by the Direct Reduction Process

CONS-5089-6

UNCLASSIFIED UC-95f

MASTER

ALCOA

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Work Preformed Under
Contract EC-77-C-01-5089

Second Interim Technical Report, Phase B
for the Period 1978 December 01 -
1979 February 28

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DISCLAIMER

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MARCH 1979

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PREPARED FOR THE
DEPARTMENT OF ENERGY
OFFICE OF THE ASSISTANT SECRETARY FOR
CONSERVATION AND SOLAR APPLICATIONS
DIVISION OF INDUSTRIAL ENERGY CONSERVATION
UNDER CONTRACT EC-77-C-01-5089

NOTICE

FORWARD

This is the second interim technical report, Phase "B", submitted in accordance with the requirements of Contract No. EC-77-C-01-5089, a three-year cost-sharing agreement between the Department of Energy and Alcoa. The report describes work performed in the second quarter of the second year of the program.

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ABSTRACT

Phase "B" of a three year cost-sharing contract between the Department of Energy and Alcoa was started on 1978-09-01. Phase "B" is 16 months long and will include four quarterly reports plus an annual report. At the end of the sixth month the program for Phase "B" is estimated as 24.4% complete with 36.1% of the funding expended.

Experimental runs were made to determine the effect of a cooler product reservoir on metal alloy yield and recovery. The reservoir temperature had no significant effect. Difficulties were experienced with operation of an oxygen injected bench scale reactor. Many tests were terminated by burden bridging or flooding of the oxygen tuyeres with metal and slag. A series of runs were made in which refluxing vapors were condensed in a liquid slag. The addition of CaO decreased the tendency for formation of thick, strong burden bridges but did not completely eliminate bridging. Reduction of flame temperatures did not affect the volatilization rate in the bench reactor. Operation of VSR-1 pilot reactor with oxygen injection was achieved after resolving reactor shell leakage problems, primarily by replacing the permeable ceramic shell with impermeable fused silica. Various combustion parameters were investigated, including coke size, burden height and oxygen flow rate. Steady state operation of the oxygen-coke system was attained with smooth burden movement and a 2000°C bed temperature in the raceway vicinity. To further reduce heat losses from the raceway area, VSR-1 was redesigned to facilitate locating an induction coil below the oxygen inlets.

Further evaluation of effects of impurities on alloy purification in the bench scale unit indicated a 50% decrease in product yield for starting charges containing Fe greater than 5%. The multipurpose high temperature induction furnace was installed including all electrical and mechanical work. Designs of the pilot crystallizer heating system and the alloy product holding ladle were completed. Site installation for the entire alloy purification complex was completed.

Operations were continued in the bench scale units to obtain design information for the pilot commercial grade Al purification unit. Procurement of construction materials was continued. A unit start-up procedure was established.

Primary activities in the third three month period will include: revision of the oxygen injected bench scale reactor to a side entry tuyere; determination of the effects of CO sweep rates and carbon stoichiometry on burden bridging and product yield; operation of the pilot reactor VSR-1 with ore-coke

mixtures to investigate mechanical and thermal techniques to promote burden movement; evaluation of low bed operation with a hot grate; initiation of a larger size VSR design; testing of a mechanical auger at process temperatures in the crucible reactor; design of a coke injection system through the oxygen tuyeres; lining and start-up of the multipurpose furnace; fabrication of the product holding ladle; procurement of long lead time materials; initiation of site construction for the commercial grade Al purifier.

DISCUSSION

The primary objective of the three year program is to demonstrate technical feasibility of a pilot sized direct reduction process for producing aluminum and aluminum-silicon alloy. The process includes three major tasks, reduction to produce impure alloy, alloy purification and purification to commercial grade aluminum. Goals for the second phase are to build and operate a vertical shaft pilot reactor, to modify the reactor design for optimum performance, to complete detailed design, construction and installation of the purification pilot units, and to start up the purification units.

In the second period of Phase "B" Carnegie-Mellon University rebuilt their small induction heated furnace to improve the quality of the preliminary experiments for determining approximate conditions of invariant reactions relating to the Al-Si-O-C phase equilibria. Assembly of the large, graphite resistance heated furnace was continued. Preliminary modelling studies of the Fe-Si and Al-Si processes were also conducted at CMU by a visiting scientist, Professor T.A. Engh, and Professor G.K. Sigworth. It is planned to continue this effort during the fourth three month period of Phase "B" by retaining Professor Engh as a consultant to Alcoa. Professor Julian Szekely consulted for 4 days on kinetics modelling and burden movement. Koppers Company consulted on VSR-1 construction and operation including calculations of adiabatic flame temperature at the tuyere zone and top-gas piping pressure drop. Koppers also provided preliminary specifications and estimated costs to produce tonnage quantities of furnace coke for large scale carbothermic reduction.

Meetings were held with Professor K. Motzfeldt and J.R. Johanson regarding the possibility of consulting assistance to the project. It was tentatively planned to retain Professor Motzfeldt to conduct a determination of the Al-Si-C phase diagram. Mr. Johanson submitted a proposal to work on burden movement problems. The proposal is under consideration.

Department of Energy approval was requested for several time and material subcontracts involving mechanical and electrical installations.

The status of the Direct Reduction project was reviewed with Dr. M.B. McNeil, Department of Energy.

Progress for the three main tasks is reported by sub-task as identified in the modified project outlines submitted on 1978 August 16. It is estimated that for Phase "B" the reduction task is 25% complete, the alloy purification task 30% complete, and the purification to commercial grade aluminum task is 18% complete at the end of the second period.

PROGRESS

A. REDUCTION - PHASE "B"

Task No. 1: Calculate Heat and Mass Balance

Further computations were made on steady state operation of the pilot reactor, VSR-1. A new computer code is being designed to enhance solution of heat balances for ore feed to the reactor.

Task completion is 30%.

Task No. 2: Continue Reaction Kinetic Studies

The kinetics computer model program was revised. The system now integrates the SiO_2/C test case past complete conversion of SiO_2 to SiC. Selection of reaction rates after the conversion are currently being studied. Experimental kinetics runs were made to determine the effect of a cooler product reservoir on metal alloy yield and recovery. Results indicated that a somewhat cooler reservoir did not substantially change the product yield or recovery.

Task completion is 30%.

Task No. 3: Continue Reaction Mechanism Studies

A number of oxides were tested by both computer calculations and experimentally to determine whether refluxing vapors can be condensed in a liquid slag. The experiments generally supported the calculations. It appears that many different reactions can occur as the volatile species back react or condense on the burden. Bench scale reactor runs were set up to simulate continuous operation in order to evaluate volatile condensation and reflux.

Task completion is 40%.

Task No. 4: Supply Burden Materials

Several different batches of extrusions were prepared for the bench and pilot reactors. The batches contained varying concentrations of ores and carbon. The compressive strength of the extrusions fired at elevated temperatures was several times that of material dried to remove absorbed water.

Task completion is 35%.

Task No. 5: Evaluate Burden Preparation Methods

A briquetter capable of producing 50 lb/hr of agglomerated burden was received and installed.

Task completion is 15%.

Task No. 6: Evaluate Burden Materials

Materials tested in this period were similar to those tested in the previous quarter. Burdens contained CaO and Fe_2O_3 additions to the normally used ore-carbon mixes. Low Fe_2O_3 extrusions were not particularly strong after firing. Higher Fe_2O_3 concentrations produced much stronger agglomerates upon being fired. Extrusions containing CaO were also very strong.

Task completion is 30%.

Task No. 7: Develop Analyses and Properties of Critical Compounds

No work was done in this quarter.

Task completion is 0%.

Task No. 8: Complete Construction of Pilot Reactor, VSR-1

Task completion is 100%.

Task No. 9: Effects of VSR Operating Parameters

Operation of the VSR-1 pilot reactor with O_2 combustion was achieved after resolving problems with CO leakage. This was accomplished by replacing the permeable ceramic shell with impermeable fused silica, revising the tuyere connections to the shell, improving the outer Plexiglas enclosure and minimizing back pressure in the off gas system. These changes allowed extended runs to be made with no elevation of CO levels in the surrounding atmosphere. Various combustion parameters were investigated, including coke size, burden height and O_2 flow rate. Steady state operation of the combustion system was attained with smooth burden movement and a 2000°C bed temperature. Measurement of burden level was also improved by installation of conductive probes to complement the mechanical level probe. Construction of two-dimensional and three-dimensional cold models was initiated to study pertinent solids and gas flow phenomena relating to raceway formation and mechanical stirring of the burden in the bridging zone.

Task completion is 15%.

Task No. 10: Effects of Operating Parameters in Bench Reactors

A number of runs were made in the oxygen injected bench scale reactor to evaluate burden formulations and operating parameters such as burden distribution, burden size, oxygen flow rates and flame temperatures. Experimental goals included attainment of burden movement, reduction of volatile species, flow of metal product through burden and slag to the collection reservoir and reduction of product residence time in an oxidizing environment. Although some runs were successful, many tests were terminated by burden bridging or flooding of the oxygen tuyere with metal and slag. Oxygen rates used were about half that of the pilot reactor. Volatiles were apparently reduced by revising the induction coil, reducing the amount of burden exposed to the very high temperatures at a given time. Reduction of flame temperatures did not affect volatilization in the bench reactor. Tuyere plugging was avoided by charging finer sized burden and running at higher oxygen flow rates. However, since the availability of fine sized formulations were limited, the primary approach to eliminating tuyere plugging will involve a revision from bottom entry to side entry. Limiting the burden exposure time in an oxidizing environment prior to attaining reaction temperature resulted in high metallic yields and burden movement. The addition of CaO to the burden decreased the tendency for formation of thick, strong bridges but did not completely eliminate bridging.

Task completion is 35%.

Task No. 11: Evaluate Materials of Construction

Zirconia components were tested in the vicinity of oxygen entry for the bench scale reactor. The material held up in areas where it was partially cooled, and failed where it was directly exposed to combusted coke and liquid metals.

Task completion is 20%.

Task No. 12: Evaluate Alternative Heating Processes

Development of a system to inject powdered carbon with oxygen through the tuyeres was started. This alternative has the advantages of controlling flame temperature and reducing contact between molten alloy product and carbon to avoid carbide formation.

Task completion is 5%.

Task No. 13: Modify VSR Design

It is desirable to supply more heat to the reactor area below the oxygen tuyeres at the upper portion of the collection reservoir. Advantages are the reduction of heat losses to improve combustion zone temperature and promote alloy flowability, and an improvement in efficiency of the induction heating system due to a more favorable coil height-to-diameter ratio. To accomplish this, VSR-1 was redesigned to raise the level of oxygen entry and relocate the bottom stage coil below the tuyeres. In addition, the distance between the top of the burden and the reactor lid will be shortened to facilitate use of bed measuring probes and mechanical devices such as a rotating auger to prevent burden bridging. The auger will be pretested at temperature in the crucible reactor. A new fused silica shell has been ordered for the modified unit.

Task completion is 10%.

Task No. 14: VSR Design Modification Construction

This task has not been started.

Task completion is 0%.

Task No. 15: Develop Ore Beneficiation Methods

This task is not scheduled to start yet.

Task completion is 0%.

Task No. 16, Supportive Phase Identification and Task No. 17, Supportive Analytical cover all work done to identify phases and to determine elemental analyses of reactor product samples. These tasks are 35% complete.

B. ALLOY PURIFICATION - PHASE "B"

Tasks 1 and 2 have no progress to report.

Task No. 3: Establish Effect of Impurities

Bench scale runs were continued on charges containing various concentrations of Al, Si, Fe and Ti. Results of previous runs were verified. Iron or Ti in small amounts did not significantly reduce product yields. In the bench unit yields greater than 50% were obtained. However, the yield figures decreased by half for starting charges containing Fe greater than 5%.

Task completion is 35%.

Task No. 4: Fabricate and Install Multipurpose Furnace

The multipurpose induction furnace and auxiliary equipment was received. The furnace was installed complete except for the refractory lining. The lining form has been fabricated.

Task completion is 95%.

Task No. 5: Install Furnace Instruments and Controls

Electrical and mechanical installations were completed. A preliminary electrical shakedown was performed.

Task completion is 100%.

Task No. 6: Fabricate, Install Crystallization Vessel and Heating System

Design of the induction coil was completed and approved. The coil and auxiliary equipment were ordered.

Task completion is 15%.

Tasks 7, 8, 10, 11 and 12 are not scheduled to start yet.

Task No. 9: Fabricate and Install Product Removal System

Design of the holding ladle was completed. All components have been ordered and steel fabrication started.

Task completion is 25%.

Task No. 13: Supportive Analysis

Chemical analyses were performed for bench scale runs made under Task No. 3.

Task completion is 35%.

Task No. 14: Supportive Mechanical Engineering

Site preparation was completed including the following: installation of the cooling tower; structural steel members; foundation concrete.

Task completion is 90%.

Task No. 15: Supportive Phase Analysis

Phase analyses were performed for bench scale runs made under Task No. 3.

Task completion is 35%.

Task No. 16: Supportive Facilities Engineering

Facilities Engineering coordinated the work effort of subcontracors for the electrical, mechanical and concrete jobs completed on site preparation under Task No. 14.

Task completion is 70%.

C. PURIFICATION TO COMMERCIAL GRADE ALUMINUM - PHASE "B"

Task No. 1: Support Pilot Plant Work

Operations were continued in the bench scale units to obtain design information for the pilot unit with emphasis on materials flow and unit capacity.

Task completion is 40%.

Task No. 2: Continue Evaluation of Types

Tests were run to evaluate the effect of oxide impurities on unit operation. No long term, large bench scale tests were completed in this quarter due to a furnace failure. Repair of the furnace has been started. Price quotations were obtained for several critical materials selected for the pilot unit.

Task completion is 40%.

Task No. 3: Fabricate, Install Unit

Procurement of construction materials was continued. A construction procedure was established.

Task completion is 10%.

Task No. 4: Fabricate, Install Residue Removal System

Calculations were made to determine the effects of residue concentration on the design and operation of the residue removal system.

Task completion is 10%.

Task No. 5: Fabricate, Install Product Removal System

Further details were completed for installation and operation of a vacuum tapping product removal system.

Task completion is 10%.

Task No. 6: Fabricate, Install Alloy Charging System

No progress for this task in the second quarter.

Task completion is 5%.

<u>Task No. 7</u>: Install Instruments and Controls

The multipoint temperature recorder was received and installed.

Task completion is 10%.

<u>Task No. 8</u>: Fabricate, Install Handling System

Design of the system was initiated.

Task completion is 5%.

<u>Task No. 9</u>: Start and Shake Down System

A unit start-up procedure was established. Production of alloy for unit start-up was scheduled for the high temperature melting furnace early in the third quarter.

Task completion is 5%.

<u>Tasks 10 and 11</u>: Supportive Analytical and Supportive Phase Identification are 40% complete.

PHASE "B" THIRD PERIOD PROGRAM

Administrative

Approval will be requested for two additional consultants.

Technical

Reduction: Heat and mass balance calculations and reaction kinetics modelling will be continued. The oxygen injected bench scale reactor will be revised to provide side entry injection. The unit will be operated to evaluate several burden formulations. Runs will be made to determine the relative effects of CO sweep rates and carbon stoichoimetry on burden bridging and product yield. Pilot reactor VSR-1 will be operated with ore-coke charges. VSR-1 will also be run to investigate mechanical and thermal techniques to promote burden movement; to evaluate cokes of different reactivities; and to evaluate low bed shaft operation with a hot grate. Design of a larger size VSR will be initiated. Cold models of tuyere flow will be observed to establish the relative effect of system variables on raceway formation. A motor driven auger will be tested at temperature in the crucible reactor. Design of a coke injection system through the oxygen tuyeres will be continued.

Alloy Purification: The multipurpose furnace will be refractory lined and started up. The product holding ladle will be fabricated. Procurement of equipment will be completed for the crystallizer construction and fabrication of crystallizer auxiliaries will continue. The bench scale crystallizer will be operated to further evaluate the effects of Fe and Ti at high concentrations.

Purification to Commercial Grade Aluminum: Further work will be done on the detailed design of the pilot unit. Procurement of long lead time materials will be continued. Site preparation construction will be initiated. The large bench scale furnace will be repaired and restarted to evaluate new materials in long term runs.

COST SUMMARY

Expenditures for the second three months of Phase "B" totaled $491,697. Distribution was $242,619 for reduction, $221,606 for alloy purification, and $27,472 for purification to commercial grade aluminum. Total cumulative spending through the first 18 months of the contract was $1,788,767. Actual spending for Phase "B" is compared to estimated spending in Figure 1.

ASSIGNED PERSONNEL

The actual man-hours expended by engineers and technicians for the second three months of Phase "B" are shown in Table 1 and compared to estimated man-hours. Through the first six months cumulative engineer man-hours were 9.7% above estimate. Cumulative technician man-hours were 4.4% above estimate. Total cumulative man-hours were 6.4% above estimate.

TASK/MILESTONE SCHEDULE

Attachment 2 shows the task-time relationship for the 3 major tasks from initiation of the current contract in DOE fiscal year 1977, 4th quarter, through completion of the current contract in DOE fiscal year 1981, first quarter. Tasks for proposed extended phases through demonstration of the process are also indicated. Completed milestones are noted by filled in circles.

TABLE 1

Summary of Man-Hours Expended

	Months 1-3 Man-hr		Months 4-6 Man-hr		Months 7-9 Man-hr		Months 10-12 Man-hr		Months 13-16 Man-hr		Cumulative Man-hr		Cumulative Per Cent Deviation
	Actual	Est.	Actual	Est.	Actual	Est.	Actual	Est.	Actual	Est.	Actual	Est.	
Engineers	3190	2737	3355	3230							6545	5967	+9.7
Technicians	4714	4046	5026	5287							9740	9333	+4.4
TOTAL	7904	6783	8381	8517							16285	15300	+6.4

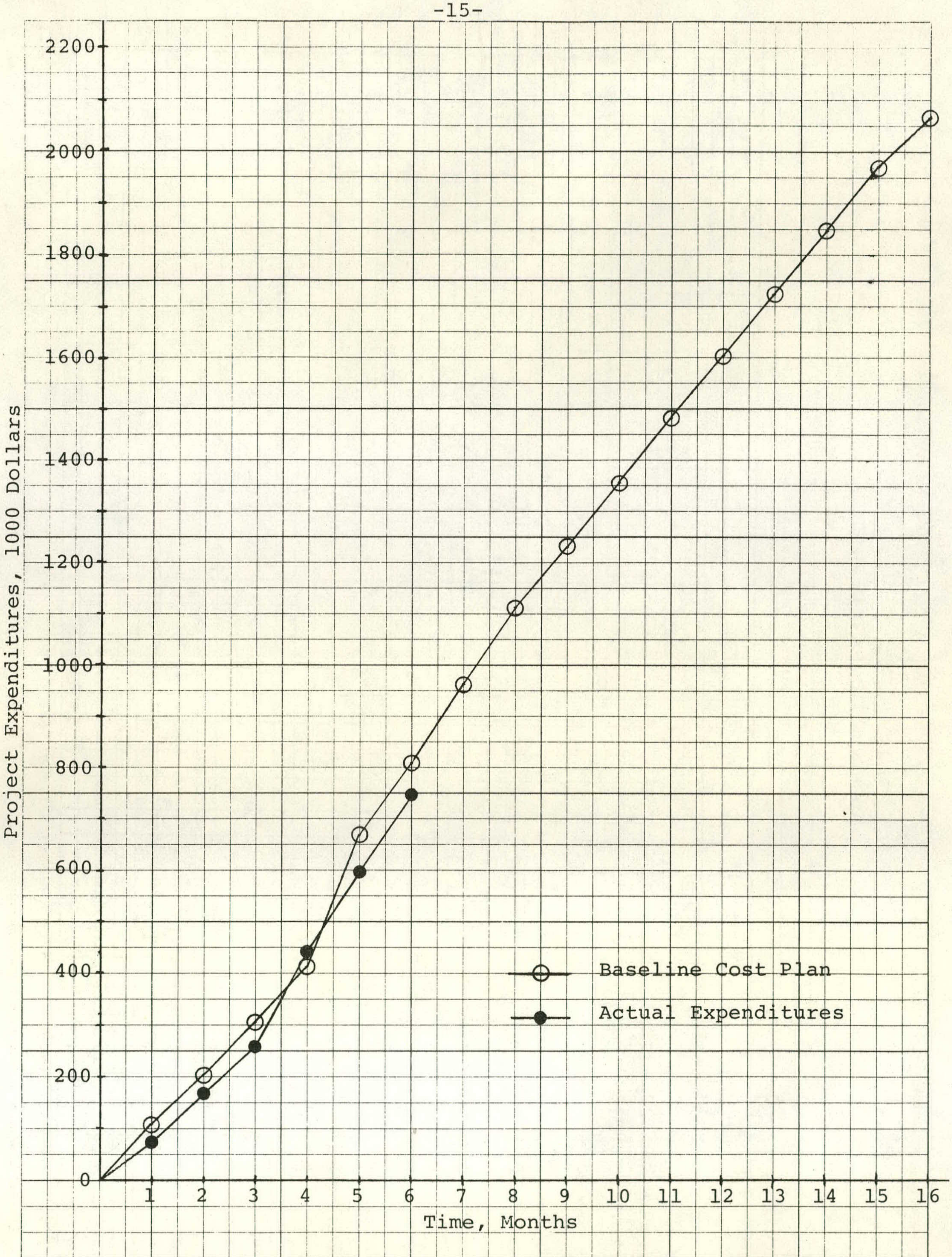

FIGURE 1 - Phase "B" Cost Chart: Total Expenditures vs. Time

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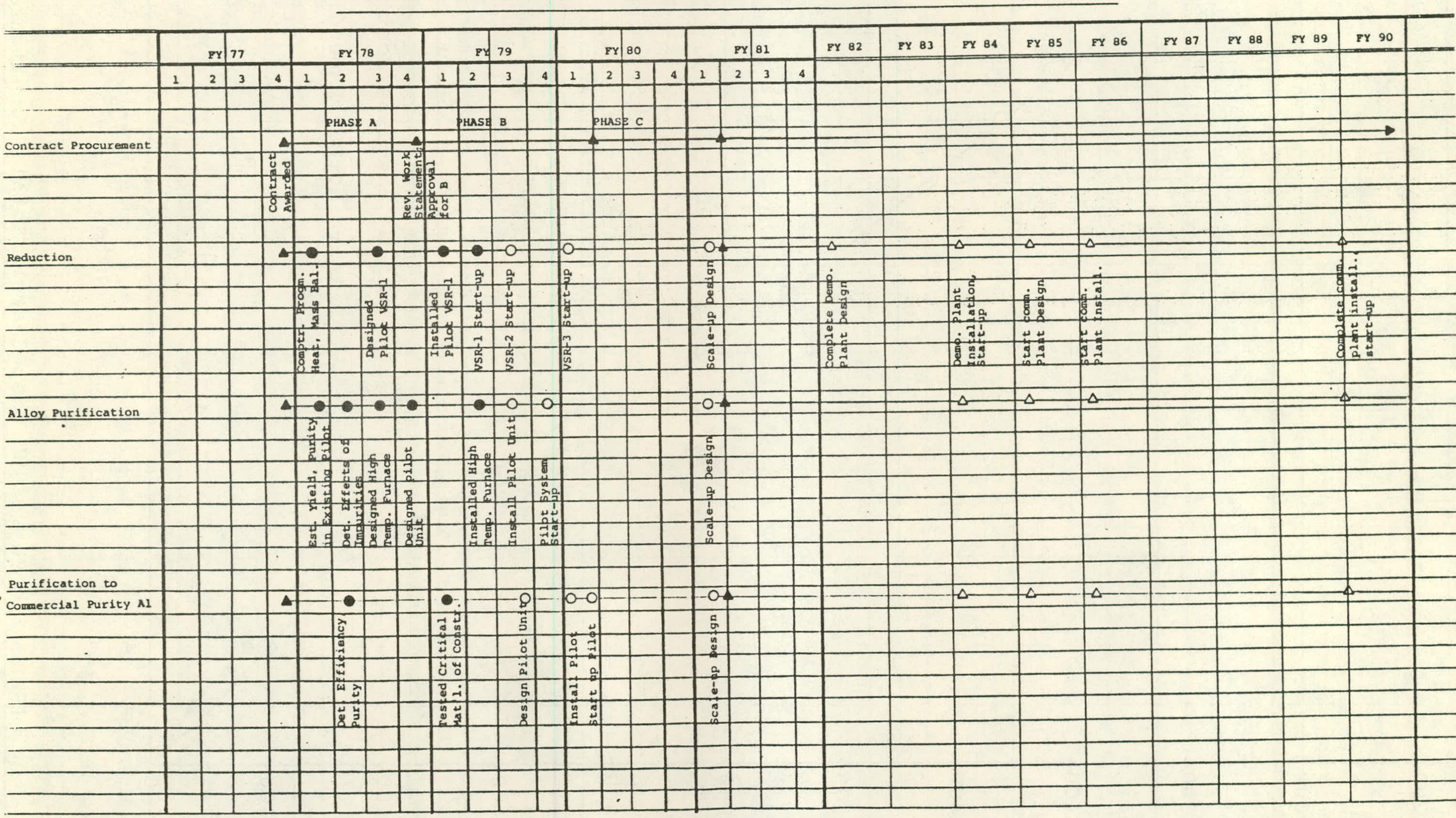
Attachment 2

Direct Reduction Project

Task/Milestone Schedule



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