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CONS-5089-12

PRODUCTION OF ALUMINUM-SILICON ALLOY AND FERROSILICON AND COMMERCIAL PURITY ALUMINUM BY THE DIRECT REDUCTION PROCESS

Second Interim Technical Report, Phase C, April 1—June 30, 1980

By
M. J. Bruno

October 1980

Work Performed Under Contract No. AC01-77CS40079

Aluminum Company of America
Alcoa Laboratories
Alcoa Center, Pennsylvania

U. S. DEPARTMENT OF ENERGY

Division of Industrial Energy Conservation

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SECOND INTERIM TECHNICAL REPORT, PHASE "C"
FOR THE PERIOD 1980 APRIL 01 - 1980 JUNE 30

MARSHALL J. BRUNO

OCTOBER 1980

ALUMINUM COMPANY OF AMERICA
ALCOA LABORATORIES
ALCOA CENTER, PA 15069

PREPARED FOR THE
DEPARTMENT OF ENERGY
OFFICE OF THE ASSISTANT SECRETARY FOR
CONSERVATION AND SOLAR APPLICATIONS
DIVISION OF INDUSTRIAL ENERGY CONSERVATION
UNDER CONTRACT DEAC01-77CS40079

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This is the second interim technical report, Phase "C", submitted in accordance with the requirements of Contract No. DEAC01-77CS40079, a three-year cost-sharing agreement between the Department of Energy and Alcoa. The report describes work performed in the second quarter of the third year of the program.

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Abstract

Phase C of a three year cost-sharing contract between the Department of Energy and Alcoa was started on 1980-01-01. At the end of the second quarter, the program for Phase C is estimated as 56.6% complete, with 51.6% of the funding expended.

Beneficiation of bauxite by high intensity wet magnetic separation to remove Fe and Ti was not successful. Pilot reactor VSR-3 was modified and operated to evaluate the blast-arc reduction process concept. Modifications included a tapered upper shaft section, vertical stroke ram, and CO preheater system. The taper/ram revisions were successful in maintaining bed movement, resulting in several continuous runs in which large quantities of burden were fed and metal product was recovered. Flows of 100-200 SCFH of preheated CO were injected at the first stage SiC reaction zone. The 200 SCFH CO rate represented 67% of the combustion products predicted for supplying first stage heat requirements. Higher rates resulted in unacceptably high top gas temperatures. The VSR-3 hearth was further modified to facilitate tapping molten alloy during operation. Attempts to tap were only partially successful due to high metal viscosity from excess carbides. A preliminary process flow sheet was developed for a commercial blast-arc concept. The SOLGASMIX computer program was revised to include a series of data bases for various solutions. The bench scale reactor studies included attempts to run at high CO sweep rates, evaluation of limiting SiO_2/Al_2O_3 ratio, optimum reduction carbon stoichiometry and carbon solubility in the alloy product. Metal was not produced at CO sweep rates of 22.5 l/m (50% higher than the normal CO rate) due to severe bridging from volatiles. The practical upper limit for SiO_2/Al_2O_3 is 1.2; higher ratios generate extreme volatilization. Optimum carbon stoichiometry appears to be 85 to 90% of theoretical. Solubility tests showed a low solubility of carbon in the alloy; rather, carbon is dissolved as SiC. At 0.43 to 0.58 SiO_2/Al_2O_3 ratio, Al_4SiC_4 is stable with the alloy produced (0.38-0.51 Si/Al ratio). Phase identification of bench reactor metal product showed no significant variations of phases in samples at the various SiO_2/Al_2O_3 ratio. However, the microstructure and slag concentrations did vary. More slag was found at the 0.79 to 1.0 ratios, probably due to increased slag fluidity.

Pilot VSR samples were also analyzed. The major phases contained Si and $FeSi_2Al_4$ intermetallics in a matrix of eutectic Al-Si.

The pilot crystallizer installation was completed and a shake-down run was made to test the hardware.

Installation of the pilot membrane cell continued. Product removal, electrolyte melting, cell shell cooling, electrical buswork and AC power systems were all completed.

Primary activities in the third quarter of Phase C will include: a special technical report on the feasibility of the combustion heated reduction process; modifications to the pilot reactor to improve the alloy tapping operation; conversion of the large bench scale crucible reactor to a submerged arc unit; calculations to predict the effects of increased pressure on a combustion heated blast furnace concept; determination of improved heat and mass balances for blast-arc and submerged arc concepts; advancement of the SOLGASMIX-REACTOR computer program to handle recycle for the Al-Si-C-O-Fe system; operation of the pilot crystallizer on simulated alloys containing Al, Si, Fe, and Ti; completion of pilot membrane cell installation.

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 third phase are to determine the feasibility of supplying high temperature reaction heat by oxygen combustion of coke in the vertical shaft reactor, to evaluate alternative reduction processes utilizing electric heat in the metal-producing zone, to complete construction and installation of the purification pilot units, and to demonstrate alloy refining and purification in the pilot units.

In the second quarter of Phase C, the following work was accomplished by subcontractors and consultants. Carnegie-Mellon University completed the addition of a recording balance and a rapid sample changing system to their high temperature furnace apparatus. Experiments to be conducted for the remainder of the program were described in a thesis overview titled, "The Carbothermic Production of Aluminum and Phase Relationships in the Al-C-O and Al-C-O-Si Systems." Professor Julian Szekely consulted for three days, and Dr. J. C. Agarwal consulted for 4.5 days on alternative reduction concepts.

Alcoa submitted information to substantiate a projected cost overrun, including the status of all sub-tasks under the three main tasks, the logic for revising Phase C tasks, and the cost savings to the contract by reducing the effort on the purification tasks, plus delaying a scale-up of the pilot reactor until the desired reduction concept has been selected. In addition, a Contract Pricing Proposal (optional form 60) and revised Cost Plan (form DOE 533P) were submitted to reflect the cost overrun.

An updated, revised milestone chart for the pilot reactor program was submitted to DOE by Alcoa at the request of the DOE Technical Manager.

Alcoa hosted a visiting delegation from the Inspector General's Office of DOE, which included a review of the Direct Reduction Process contract. Information requested on invention disclosures, background patents, technical reports, technical papers, and subcontract documents was provided as requested.

A list of subcontractors for time and material work was submitted, updating this category through Phase C, second quarter.

Amended agreements with Carnegie-Mellon University, Koppers Company, and Professor Julian Szekely were approved by DOE.

The contract number was changed from EC-77-C-01-5089 to DEAC01-77CS40079, effective 1980 April 10.

Alcoa obtained DOE approval to use certain equipment being fabricated under the contract for independent research and development on a non-interference basis.

Technical progress was reviewed for the DOE Project Manager and Professor J.F. Elliott one time during the quarter.

Progress for the three main tasks is reported by sub-task as identified in the modified project outlines submitted on 1980 March 26. It is estimated that for Phase C the Reduction task is 59% complete, the Alloy Purification task is 54% complete, and the Purification to Commercial Grade Aluminum task is 45% complete at the end of the second quarter.

A. REDUCTION - PHASE C

Task No. 1: Supply Burden Materials

In the second quarter, all burdens were agglomerated as balls, made in the Eirich mixer or the small-Sprout-Waldron disk pelletizer. Materials prepared are described in Table 1.

Task completion is 50%.

Task No. 2: Burden Beneficiation

Experiments using Rebolado bauxite demonstrated that no significant reduction in either iron or titanium can be obtained for this material by high intensity wet magnetic separation. For the condtions tested, iron showed an average decrease of 1.3 wt% Fe_2O_3 (from 12.0 to 10.7 wt% Fe_2O_3) while titanium showed essentially no change. The well dispersed nature of the magnetically susceptible minerals indicated in the tests suggests simple physical separation method will not produce the desired lowering of the iron or titanium levels.

Task completion is 100%.

Task No. 3: Effects of Pilot Operating Parameters

Trials were conducted during the second quarter to demonstrate the feasibility of operating a combined "blast-arc" for the production of Al-Si alloy. The pilot reactor was therefore modified to allow for the injection of carbon monoxide into the Stage I reaction zone at a temperature of 1850°C. Furthermore, the upper section of the vertical-shaft reactor (VSR) was tapered and a vertical-stroke ram was added to the design in an attempt to prevent bridging problems which occurred during previous experimental runs. These design changes are illustrated in Figure 1 and are described in this report under Task 4 (Pilot Modifications).

The taper and ram proved successful in preventing bridge formations during the initial pilot run (VSR-36). An average bed movement of over 7 kg of ore/hr was established. This corresponds to the best solids flow obtained during the pilot reactor campaign. Furthermore, this rate of movement was maintained when 100 SCFH of preheated CO was injected at 1850°C at a point in the Stage I reaction zone to simulate the "blast-arc" gas flow pattern. A further increase in the CO injection rate was prevented by a high temperature in the upper stack and an irregular rate of feeding (charging) pellets. A substantial ingot was produced during VSR-36.

The next run (VSR-37) was prematurely shutdown due to high temperatures in the upper shaft and the formation of a weak, condensate bridge above the taper in the vicinity of the reactor stockline.

Several horizontal pegs were added to the ram prior to VSR-38 to combat condensate bridges in this upper area. Apparently the injection of CO gas into Stage I transfers a substantial amount of heat to the upper shaft. Therefore, the power distribution from the induction system was adjusted to maintain a lower stack temperature. The packed bed for preheating CO and the catchbasin were also altered at this time to permit future tapping experiments to be conducted (see Figure 2).

In order to maximize production time on the pilot unit, run VSR-38 was started up with the "birdsnest" from VSR-37 left on the reactor grate as illustrated by Figure 3. This run would be analogous to restarting a commercial reactor after a temporary shutdown or delay. CO injection was not attempted during this run.

The vertical-stroke ram and taper were again successful in maintaining bed movement. Solids flow exceeded 8.7 kg of ore/hr for a four hr period. Reactor material (pellets) still had the tendency to stick during the run and bed movement was irregular. However, over 55 kg (121#) of pellets were consumed during the run.

After shutdown, excavation revealed that metal (alloy) had eroded through the graphite catchbasin and had passed through the packed bed of graphite tubes designed to preheat the CO gas (see Figure 4). These graphite tubes (3/4" O.C. x 1/2" I.D.) acted as a filter and a large amount of metal was retained in the packed bed. However, several kilograms of very fluid alloy passed through the entire 26" length of packed tubes and ran into and through the 1/4" holes in the bottom distributor plate. This metal appeared to be very clean and melted readily with a heliarc torch.

The chemical composition of this filtered alloy is presented in Table 2. The typical carbon content of ingots produced to date in the VSR reactor has been in the 5-8% range. The packed bed therefore was very effective in removing carbon from the alloy to the .16% level. The substantial drop in the Si/Al ratio after filtering indicates that carbon is being removed from the metal in the form of SiC. Therefore, the use of a carbon filter could be employed in the future to extract carbides from the alloy and to enhance the flow characteristics of the unrefined Al-Si alloy.

In May, the pilot reactor continued to be operated in a manner that would simulate a commercial "blast-arc" for direct reduction of Al-Si alloy. During run VSR-39, carbon monoxide (100 SCFH) was injected into the Stage I reaction zone prior to the attainment of metal-producing temperatures. The objective of this experiment was to certify that all of the metal produced in the catchbasin had been made from pellets exposed to CO sweep in Stage I.

The bed moved rapidly during VSR-39 at an average rate of 7.3 kg of ore/hr. As in all of the pilot runs to date, the burden tended to fuse and bridge. However, the motion of the vertical-stroke ram maintained a regular downward flow of solids. This movement of bed material was maintained when the CO injection rate was increased later in the run to 200 SCFH. (Bed reactions at this time were generating an additional 150 SCFH of carbon monoxide.) An injection rate of 200 SCFH corresponds roughly to 67% of the CO generated by a char-oxygen burner meeting the energy requirements of Stage I.

After a scheduled shutdown, an autopsy of the reactor revealed that a record 14.1 kg ingot had been produced during VSR-39. Since 50.3 kg of ore (pellets) had been consumed, the alloy to charge yield was 28%. This corresponds to 82% of the theoretical maximum rate of metal production anticipated from raw ore at steady state.

The excavation of the VSR unit (Figure 5) also showed the erosion of the CO injection tubes at a point 3" above the grate. (Approximately 13" below the designed Stage I injection level.) Based on an unanticipated pressure drop decrease in the CO tubes during the run, carbon monoxide probably was diverted into Stage III (instead of Stage I) during the last two hours of operation. A definite gas pattern was evident in the Stage III semi-metallic "birdsnest" at the points of injection tube erosion damage. This suggests that some amount of localized CO sweep into Stage III may not inhibit bed movement or alloy production during a pilot reactor run.

The top of the ore bed had become extremely hot (sparks) during the last hour of operation. The excavation of the reactor contents indicated metallic slag formation at a high level in the VSR shaft. This suggests that heat and mass flow considerations may limit the rate of CO injection to 200 SCFH with the present design of the pilot reactor.

After demonstrating in part the feasibility of the "blast-arc" concept, the experimental program shifted to the installation of a system for tapping Al-Si alloy from the pilot reactor. Intermittent tapping would permit the pilot unit to be operated for extended periods where steady-state conditions could hopefully be attained. Furthermore, trials to purify the alloy (by filtering and/or ladle additions) could be conducted prior to delivering the alloy to the two downstream refining steps. A schematic of the initial location of the taphole and the detailed taphole design features are presented in Figures 6 and 7. A description of the system is contained in the Task 4 section of this report.

The objective of the first run (VSR-40) after installing the taphole was to remelt and cast into a preheated crucible the Al-Si metal produced during VSR-39. The pilot reactor was therefore brought to normal operating temperature and the VSR-39 ingot was lowered in a graphite container into the high temperature zone. A load-cell attached to this container was used to monitor the process of melting. Unfortunately, only 48% of the metal in the ingot could be melted at temperatures up to 2050°C. Apparently, the more fluid constituents quickly flowed from the ingot leaving behind a solid alloy-carbide matrix in the container. The metal that did drain from the ingot either vaporized as evidenced by condensation rings on the reactor wall or dripped into the 2" reservoir beneath the taphole. Therefore, metal was not withdrawn through the taphole during VSR-40.

The next trial (VSR-41) involved remelting a synthetic Al-Si alloy of known composition (75% Al, 25% Si) and melting temperature (less than 1100°C). This alloy which was heated to the melting temperature on the grate readily drained into the catchbasin as observed through the top site-port. This alloy was successfully confined in the catchbasin by the graphite plug and by the freezing action of the water-cooled pipe inserted in the plug. At a prescribed time, the pipe was removed and the graphite plug allowed to heat. The plug was then withdrawn from the hole and metal was successfully drained into a tercod crucible.

After demonstrating the reliability of the taphole design an attempt (VSR-42) was made to produce alloy directly from ore pellets and to simultaneously drain the metal into the outside crucible prior to solidification. Burden movement during this run was very smooth resulting from improved control of the vertical-stroke ram. The ore pellets were composed of bauxite, clay, and coke breeze and very little condensation or dusting was evident.

After consuming 54 kilograms of ore, the water-cooled pipe was removed from the taphole and a stream of very viscous metal slowly flowed for a short time into the tercod crucible. Metal at times had to be raked from the graphite tapping tube with a graphite or steel rod. A total of 4.5 kg of Al-Si alloy was collected in the crucible. A videotape film was made of this initial procedure.

An autopsy of the reactor showed that 8.1 kg of Al-Si ingot remained in the catchbasin and did not drain at a temperature of 1500°C. This ingot was similar in appearance to the solid residue in the VSR-40 run and probably contains an excess of carbides.

Task completion is 60%.

Task No. 4: Pilot Modifications

Figure 1 illustrates the reactor alterations made in the second quarter prior to run VSR-36. As described in the last quarterly report under Task 4, a packed bed arrangement of graphite tubes was incorporated into the reactor to internally preheat injected carbon monoxide to temperatures above 1800°C. The pilot shaft was tapered to angle of 86° (similar to the stack batter on a commercial blast furnace) to improve the downward movement of solids. The 6-in. dia rotating auger has been replaced with a 1-7/8-in. dia vertical "ram". This ram has an 8-in. vertical stroke designed to break ore bridges in the diverging section of the reactor shaft.

Further modifications were made to the pilot unit prior to VSR-38 (see Figure 2). The points of CO injection into Stage I were moved to the reactor outer wall. Several horizontal pegs (3/4-in.) were added to the ram to combat weak condensate bridges in the narrow section (6-in. dia) of the shaft. The packed bed and catchbasin were elongated to permit future tapping of the reactor through the ceramic region of the shell.

After successfully demonstrating the initial phase of the "blast-arc" program (VSR 38 and 39) with these modifications, a system for tapping alloy from the pilot unit was installed. Figure 6 shows the general location of the new taphole. A graphite tube (2-in. O.D.) was inserted at an angle of 19° through the ceramic shell, insulation, and graphite susceptor. Castable insulation was set between flanges A and B to minimize heat losses and to protect the metal components from alloy splashing. A tercod crucible placed on refractory brickwork was positioned at the outer end of the tapping tube to collect the molten alloy. The entire tapping arrangement was enclosed in an aluminum partition equipped with an exhaust. Nitrogen and argon was injected into the enclosure near the end of the tapping tube to prevent air-burning of graphite and oxidation of the alloy. Levels of carbon monoxide in the aluminum enclosure were monitored to provide for operator safety.

Figure 7 illustrates the detailed design of the (2-in. O.D.) graphite taphole. A hollow graphite plug (1-3/8-in. O.D. - 7/8-in. I.D.) inserted into the graphite tube is used to block the flow of molten metal prior to tapping. A water-cooled pipe inside this plug helps to freeze molten metal to prevent inadvertent flow.

Just prior to tapping, the tuyere is removed and the plug and tube are allowed to heat. An electric arc (stinger) is available to provide more heat to the graphite to melt alloy in the taphole to initiate tapping. After heating, the plug is withdrawn and metal flows into a preheated (moisture-free) SiC crucible.

Task completion is 60%.

Task No. 5: Reactor Scale-Up Design

A preliminary flow sheet was conceived for production of 300,000 TPY aluminum using a hybrid reactor combined with fractional crystallization and electrolytic purification. It was assumed that an Al, 40% Si alloy diluted by 2% Fe and 1% Ti would be produced from a pellet containing clay, Bayer alumina, and coke. Bayer alumina was selected over bauxite because low iron bauxite is not likely to be available in the future. High iron bauxite would raise the iron content of the raw alloy enough to make reasonable fractional crystallization yields questionable. It was assumed that the charge would be preheated and pre-reduced to SiC using heat supplied by a coal-O_2 burner. This would supply about 50% of the thermal requirements. The remaining energy would be supplied electrically but off-gas CO would be used to generate electricity at 35% efficiency to offset most of the electrical requirement. Thus, less than 2 kWh/lb aluminum would need to be purchased.

A number of critical factors have been identified in the process of making the flow sheet.

1. Effects of Fe and Ti on aluminum yields in fractional crystallization, electrolysis and mechanical separation of crystal bed, if any. The size and capital and operating costs of a 300,000 TPY plant depend directly on the material handled which, in turn, depends upon the yield.

2. Market for silicon and ferrosilicon. A high price and large market for metallic by-products has numerous benefits: higher iron bauxites can be used; clay can be used in sufficient quantities to assure adequate pellet strength; higher silica content in the charge permits more combustion heat to be utilized; the additional combustion heat results in more CO generation which translates into more cogenerated electricity. If silicon and ferrosilicon production must be minimized, relatively pure raw materials must be used; aluminum content in the alloy must be maximized; silicon recycle must be maximized; and the charge will probably have to be briquetted at a higher cost than pelletizing. It may not be possible to design a process operating at less than 4 kWh/lb aluminum if metallic by-product production must be minimized.

3. Coal-O_2 burners. The ability to supply combustion heat to a packed bed using burners needs to be demonstrated. Good heat transfer is required in order to prevent localized melting of the charge. Slag from the coal must coalesce and drain without freezing due to SiC formation.

4. The smelting behavior of prereduced (SiC containing) burdens in an electric arc must be studied in order to determine E-I characteristics and productivity/in. of electrode diameter. Carbon content of the alloy must also be determined under arc smelting conditions.

The process of generating a flow sheet will continue to identify areas of uncertainty which can help direct the formulation of an experimental program and to narrow the range of process options based on economic considerations. Costs and supply of raw materials and energy inputs as well as price and demand for CO and metallic by-products must be considered in the course of optimizing the flow sheet. During the next quarter, preliminary flow sheets for burden preparation, the shaft reactor, the off-gas system, product handling, refining and purification will be prepared and reviewed.

Task completion is 50%.

Task No. 6: Calculate Heat and Mass Balance

Further experience was gained with the FLWEQUIL and FLWEQSEP models in this quarter. Several uncertain parameters in the models were isolated to ranges which produced reasonable results.

Task completion is 60%.

Task No. 7: Process Mathematical Modeling

The SOLGASMIX program was modified to correctly treat ideal and nonideal condensed phase solutions, and a series of databases incorporating various solutions was set up. These will form the basis of process modeling in the future.

A visit was made by Professor Gunnar Erikkson of the University of Umea and Mr. Thomas Johansson of KemaNord Industrial Chemicals Division, and a program exchange effected. The program obtained is a version of SOLGASMIX (called SOLGASMIX-REACTOR) designed to automatically make converged equilibrium mass balances on shaft reactors such as the direct reduction reactor. Conversion to the Alcoa computer systems and expansion to provide conversational program dialog and database management were begun.

Task completion is 70%.

Task No. 8: Effects of Process Variables

Thirteen bench-scale reactor runs were conducted during the second quarter. The details of each run can be found in Figures 8-19 and Tables 4 and 5. This quarter's program focused on producing metal at higher levels of CO sweep and at the practical extremes of the ore burden SiO_2/Al_2O_3 ratio. In addition, a program was begun to determine the optimum metal producing temperature and reduction carbon stoichiometry vs SiO_2/Al_2O_3 ratio in the ore burden. For some months, we have been attempting to run carbon solubility tests in Al-Si-C alloys contained in graphite and SiC crucibles at 2000°C without losing the metal to vaporization, extreme wetting of all solid surfaces, and penetration of the crucible wall. Experimental techniques have been satisfactorily developed, and some preliminary results were obtained.

Previous CO sweep runs had been conducted at 1 atm total pressure and 15 litre/min of CO flow which represented only a small fraction of the sweep gas that would result from a fully combustion heated reactor. In the first run when the CO sweep was increased by 50% to 22.5 litre/min, we had difficulty reaching temperature. The induction coil was repositioned for the next run to better preheat the incoming CO sweep gas. In both runs, no metal was collected in the reservoir below the grate, although phase analysis of the reaction zone material showed some metal had been produced. Each run was terminated prematurely due to severe bridging. These two runs can be directly compared with run AF12 which was identical in all variables except CO sweep. The process operation during this earlier run was smooth, and shutdown occurred only after the reservoir was full of metal.

It was suggested that as the CO sweep is increased, it would not attain a sufficient preheat temperature before it passed through the reacting charge. Since the CO is preheated by passing the gas in a duct in the wall of the carbon susceptor before it enters the reactor below the grate, the reducing potential of gas is determined by the temperature at which CO is equilibrated with carbon. If the gas was colder than the metal-producing zone, it is possible that the metal would be reoxidized and the difficulty of making metal could be attributed to reoxidation rather than the volatilization of all metal species as they attempted to come to equilibrium with the increased flow rate of sweep gas. The only condensed oxygen containing species that should be present in the reacting charge above 1575°C are Al_2O_3 and aluminum oxycarbide, and these are generally found in the metal-producing zone along with the alloy being produced and metal carbides regardless of the operating conditions. In AF17, quantitative analysis for oxygen by neutron activation analysis and qualitative analysis for phases by x-ray diffraction showed that no oxides were present and only alloy, SiC and carbon remained. This clearly indicates that for this run there was sufficient temperature and reducing conditions to make metal, and that the metal producing reactions had either gone to completion after bridging had cut off the flow of burden to the metal-producing zone, or that the gas flow was sufficient to sweep all residual oxygen away in the form of metal suboxides of Al_2O and SiO, or a combination of both mechanisms. Whichever was the case, operation of the furnace at 22.5 litre/min of CO sweep was very irregular and shutdown occurred due to severe bridging with metal being produced at only a negligible rate.

Further experimentation and alteration of the experimental procedure was contemplated to better conclude the effects of CO sweep, but updated thermodynamic calculations predicting severe voltailization of metal and suboxides at even low rates of CO sweep verified our experimental results, and attempts to make metal above 15 litre/min of CO sweep in the bench scale reactor were terminated. If we were to continue the blast furnace phase of the program, we would have to decrease volatilization of the metal species either by increasing the furnace pressure or lowering the Al and Si activities in the metal by producing higher Fe alloys. The induction-heated, quartz-shell reactor cannot sufficiently be pressurized to test the effects of pressure, and currently we cannot purify alloys containing high enough iron to sufficiently reduce the Al and Si activity.

Because some unreacted ore had leaked into the metal collection reservoir in AF14, this run of 15 litre/min of CO sweep and 0.993 SiO_2/Al_2O_3 ratio was repeated in AF18 before going to a higher SiO_2/Al_2O_3 ratio in a clay-coke burden containing no bauxite. Operating conditions were identical for these two runs except AF18 lasted 100 min longer consuming more burden and producing more metal even though more total CO gas passed through the reaction zone relative to AF14 (see Table 4).

Clay-coke burdens of a 1.224 SiO_2/Al_2O_3 ratio were tested in runs AF20 through AF24. Identical conditions were utilized in the CO sweep runs AF20 and AF21 wherein 15 litre/min were passed through the furnace. In AF20, the bed moved very irregularly and severe bridging broke the auger causing a premature shutdown but not before some metal was produced. A new stronger auger was used in AF21, and although the time of these runs was identical before a forced shutdown due to bridging, no metal was accumulated in the reservoir. Phase analysis of material in the reaction zone above the grate showed no presence of oxides in these two runs. In AF22 these runs were repeated, but at a slightly higher carbon stoichiometry in the burden, and again, operation was difficult and no metal was accumulated in the reservoir before an early shutdown. Since it was desired to try to duplicate the metal production under CO sweep as it occurred in AF20 and we had not done so, it was decided to observe the operating conditions of making metal with a clay-coke ore without any superimposed CO sweep. Some metal was made before a forced shutdown due to bridging, and the bed movement was irregular throughout the run. It appears that the recycle loading of volatiles in the gas was higher for the clay-coke burden than for lower SiO_2/Al_2O_3 ratio ores, and that we were possibly near the maximum limit of SiO_2/Al_2O_3 ratio (roughly a 52% Si, 48% Al alloy) for making metal under any conditions in the bench scale reactor. Further tests in this range of SiO_2/Al_2O_3 ratio are required to verify this analysis. It may be that conditions of operation as they affect bridging are different and possibly more severe for high grade ferrosilicon and silicon production as compared to the production of Al-Si alloys. We will test ferrosilicon or silicon production in our reactor to complete the comparison.

It should be noted that from the comparisons of similar and identical runs, it is difficult to reproduce some results, and that subtle variations in operating parameters are not easily tested. It is possible to test the relative effects of variables such as CO sweep, carbon stoichiometry in the ore, the distribution of reduction carbon inside and exterior to the pellet, as well as others with respect to SiO_2/Al_2O_3 ratio, but it is not possible to pinpoint any of these variables except within a range of values. For this reason, we did not take the time to become any more precise with the CO sweep vs $SiO_2Al_2O_3$ ratio measurements and decided to concentrate on studying other variables without a superimposed CO sweep. It was also concluded that since the high temperature part of the process was most likely to be a submerged arc furnace, further tests with CO sweep were not necessary.

Previous tests of whether metal could be satisfactorily produced with all of the carbon placed external to the pellet were conducted under a CO sweep of 13 litre/min with a layer charging in the L bench reactor. Since we've had much more success in making metal under the experimental conditions of the auger-feed (AF) reactor, where continuous operation was established for the first time in the bench scale program, it was decided to retest some of the critical variables studied in the L reactor program. AF24 was a non-CO sweep run wherein the carbon-free ore extrudates were mixed with the pet coke chunks and not as a segregated or layer charge as was previously used. The bed moved very slowly, however, operation was maintained almost until sufficient ore was fed to fill up our reservoir with metal. Bridging was the cause of shutdown with all of the material in the metal-producing zone being reacted to completion leaving a large void below the bridge (see Figure 15). The ingot was very heavily carbided, and the grate was almost completely consumed. The carbided ingot expanded to the point of cracking open the reservoir and the susceptor surrounding the reservoir. It was difficult to decide whether the results of this run were tied to the fact that the reduction carbon was located exterior to the pellet or to the SiO_2/Al_2O_3 ratio of 0.469 which was the lowest run in the AF reactor to date. Chemical analysis verified that the carbided ingot contained considerable metal; therefore, more tests would be required to determine if the severe bridging was caused by the low SiO_2/Al_2O_3 ratio or the location of the reduction carbon. In AF25 an 0.68 SiO_2/Al_2O_3 ratio burden with 85% reduction carbon was tested (see Table 4 and Figure 16) quite normally to a planned shutdown, and the ingot was again severely carbided. The coil had been lowered to better preheat the CO sweep gas during previous runs and was now overheating the metal collection reservoir such that severe attack of the graphite reservoir by the metal was converting the metal to carbides. The induction coil was raised for AF26 and this carbiding problem was eliminated. These results indicate that the metal of $0.34<Si/Al<0.96$ weight ratio is not in equilibrium with solid carbon but SiC and possibly Al_4SiC_4 (see discussion of C solubility studies below).

Generally, some short graphite rods are placed on top of the grate to support the burden in the metal-producing zone and prevent ore pellets or molten oxides from falling into the reservoir before reacting to metal. For the most part, this works very well and the metal drains through the bed of graphite rods coating them with metal and carbides. It is generally easy to find most of the rods after each run with a zone of partially reacted Al_2O_3, Al_4O_4C, and SiC mixed with some metal product on top of the bed of rods (see Figure 10 for an example of a typical successful run). Since the ore burdens are generally slightly substoichiometric in reduction carbon and the heat source is the inductively heated graphite wall, a certain amount of attack of the wall does occur. The coating on the wall consists of all possibilities of metal, carbides, and slag, but in general, there is no severe attack of the walls or even the graphite grate. In previous tests on the L reactor using 13 litre/min of CO sweep and 100% stoichiometric reduction carbon, it was found that a bed of dense low surface area graphite or SiC rods above the grate were not severely attacked, but that the metal drained much better through the graphite bed. This was the basis for choosing graphite rods as the bed material in the AF bench scale reactor. It was decided to test high density SiC chunks coming from the remnants of crucibles used in the carbon solubility studies as a bed material for runs AF27 and 28. These tests were non-CO sweep runs with substoichiometric reduction carbon (see Table 4 and Figures 18 and 19). None of the SiC chunks could be found after each run, and it appeared that in AF27 that the charge was reacting beginning from the SiC bed up as much as from the wall in towards the center as is usually the case (see Figure 18). The SiC chunks probably reacted with the substoichiometric charge and not the metal (see discussion of carbon solubility studies below).

The problems of using a burden containing 100% of the required reduction carbon were discussed in last quarter's report. Most of our experience in the AF reactor has been with 80% stoichiometric reduction carbon and an accumulation of slag in the reaction zone is in evidence for a wide range of SiO_2/Al_2O_3 ratios. We need to know the optimum amount of reduction carbon in the burden to leave no slag accumulation or a carbide accumulation as is the case with 100% stoichiometric reduction carbon in the burden. Table 4 shows that we have tried many runs at approximately 85% and one at 91% carbon to date. Only run AF26 has been without some other special problems. This run shows that at 0.785 SiO_2/Al_2O_3 ratio and 84.5% stoichiometric carbon slag is still accumulating. Considerably more work will be done in this area in the future.

Run AF27 differed from AF13 only in an increase from 80.0% to 84.7% in reduction carbon in the ore and no CO sweep was used in AF27. Since the SiC chunks on the grate were totally consumed, it is hard to evaluate what effective percentage of reduction carbon was present in the reaction zone. Undoubtedly, both the lack of CO sweep and higher reduction carbon contributed to the doubling of metal production in AF27 over AF13, but it is hard to conclude which was more predominant from this test. Run AF28 had a premature shutdown, but if it is compared with AF26, it could be concluded that 91.3% reduction carbon makes more metal than 84.5% and no appreciable accumulations of carbide or slag occurs at 91.3% reduction carbon for an 0.785 SiO_2/Al_2O_3 ratio.

Metallographic phase analysis of all the metal produced in the AF reactor to date have been compared to the Si/Al weight ratio of the metal at various parts of the ingot as shown in Table 6. X-ray diffraction analysis has shown that Al_4SiC_4 was present in ingots made from 0.502 and 0.469 SiO_2/Al_2O_3 ratio ore (corresponding to 0.443 and 0.414 Si/Al ratio for the equivalent metal composition) and that $Al_4Si_2C_5$ was also found in metal coming from 0.502 SiO_2/Al_2O_3 ratio ore. Although there was no indication of Al_4SiC_4 by x-ray diffraction in AF7 at an 0.6 $SiO_2Al_2O_3$ ratio, the metallographic analysis showed a presence of this phase. The X-ray diffraction and quantitative analysis were done on essentially the same sample while the metallographic samples were taken from nearby areas in each case. Due to the heterogeneity of our bench scale ingots, these comparisons can only be approximated. Compilation of the data in Table 6 suggests that Al_4SiC_4 is stable with liquid metal of between $0.38<Si/Al<0.51$ coming from an ore of $0.43<SiO_2/Al_2O_3<0.58$ or an alloy typical of 72.5% Al - 27.5% Si to 66% Al - 34% Si. Further testing in carbon solubility studies will be used to verify this point.

Generally, large accumulations of SiC have been found in the AF bench scale as well as pilot plant ingots of Al-Si alloy, and have never been correlated with the metal Si/Al ratio and carbon chemical analysis. This problem of carbide formation causes great difficulty in the flow of the liquid alloy because the metal is easily entrapped in a honeycomb network of solid carbides. It is, therefore, very difficult to tap metal from the furnace, and filtering of the carbides from the metal is necessary before any solidification or crystallizing technique for alloy purification can occur. Limiting the chemical attack of the furnace walls and hearth area by the reduction reactions and liquid metal respectively is also of great practical importance. The most desirable circumstance is to keep carbide formation to a low level such that all of these operating problems are alleviated.

The inability to correlate the data suggests that equilibrium between carbon and metal does not exist. In order to resolve this problem as well as to pinpoint the stabilities of various carbide phases in the Al-Si-C system, it was desired to study the solubility of carbon in alloys of varying Si/Al ratio at different temperatures in different crucible materials. In our experiments to date, we have tested one initially carbon-free alloy of 0.685 Si/Al ratio in graphite, glassy carbon, and SiC crucibles of varying sizes and material density. At temperatures in the order of 2000°C, the alloy has fairly high metal vapor pressures, easily wets many solids and appears to be highly reactive with carbon and any low to medium density crucible material. The metal flows right through medium density SiC crucible walls like it was a sponge. In order to maintain a pressure inside the crucible of one-atm, graphite lids were used with a small 1/8" hole to allow pressure equalization. Significant metal losses due to vaporization and crawling of the liquid metal through this small hole still occur. It was finally decided to accept whatever pressurization might occur inside the crucible as it is not likely to effect the results to a measurable extent. Such experimental conveniences are typical for high temperature carbon solubility studies of volatile metals. To date, we have used only graphite lids, but will soon have high density SiC crucibles with tight fitting lids to continue the studies.

The results to date show that the alloy studied dissolves carbon as SiC from graphite crucibles, and that this reaction of C from the wall and Si in the metal will likely continue until one or the other is exhausted. The buildup of SiC crystals growing from a graphite wall is generally directed proportional to the time at a temperature of 2000°C. The same alloy contained in dense SiC crucibles shows little to no attack of the crucible wall; however, come carbon is found in the solution. It is not certain whether this minor amount of carbon pickup (less than 2 wt%) comes from the SiC crucible or the metal that wets the graphite lid and drips back into the liquid sample in the bottom of the crucible. It is certain at this time that the carbon pickup from dense graphite crucibles is much greater than from dense SiC crucibles and that SiC may be the solid in equilibrium with this alloy.

If this is true, very low solubilities of carbon may exist in the alloys. It would then be advisable to use SiC hearth materials or possibly those of Al_4SiC_4 to limit carbon pickup and alleviate the problems in tapping the metal from the furnace and carbide removal before purification. These preliminary results also show that the consumption of dense SiC chunks on the grate as discussed above was due to the reaction with slag and not metal.

Task completion is 60%.

Task No. 9: Supportive Analytical

Analyses were completed as required. A total of 635 man-hrs were expended.

Task completion is 50%.

Task No. 10: Supportive Phase Identification

Phase analyses were done on metal produced in a series of runs in the 101.6 mm bench reactor, with varying burden compositions in order to determine the effects of changing burden composition on the metal produced. The SiO_2/Al_2O_3 ratio in the burden was varied as follows: AF-13, 0.5; AF-7, 0.6; AF-12, 0.788; AF-14 and AF-18, 1.0; AF-20, 1.22.

No significant variations were found in the metal phases in the samples from AF-12, 13, and 14; all consisted primarily of three-phase eutectic microstructure, Si, and SiC, with minor amounts of $FeSi_2Al_4/FeSiAl_5$, and $TiSi_2(Al, Fe)/TiSi_2(Al)$. However, there were differences in the microstructure of the ingot, and in the amount of slag contained in the ingot. AF-12 and AF-14 contained increasing amounts of slag in the ingot with increasing SiO_2/Al_2O_3 ratio. This change probably reflects increasing fluidity of the slag, so that larger amounts could flow through the grate into the reservoir. In addition, the metal overlying the slag in AF-14 (in which slag comprised 40-50% of the ingot) contained very abundant Fe-Si-Al intermetallics, due to fractionation of iron into the metal from the large volume of slag. There was also a strong tendency for metal from the low-SiO_2 burden compositions to rapidly form a SiC "skin" in the reservoir; this "skin" of SiC on the surface of the droplets restricted coalescence of the droplets, resulting in very high interstitial porosity, including open voids, near the base of the ingot. The tendency to form this "skin" decreased at successively higher SiO_2/Al_2O_3 ratios.

Metal produced in run AF-7 (SiO_2/Al_2O_3 = 0.6, 100% stoichiometric carbon, no CO sweep) was intermediate in phase composition between AF-13 and AF-12 (80% stoichiometric carbon, 15 ℓ/min CO sweep, and SiO_2/Al_2O_3 ratios of 0.5 and 0.788, respectively). Coalescence of metal in AF-17 was limited, as in other runs in which a CO sweep was used, suggesting that reaction of metal in the reservoir with CO is limited, if present at all.

Comparison of two runs with the same SiO_2/Al_2O_3 ratio and different carbon sources (COED char vs metallurgical coke) yields questionable results. The differences may not be due to the change in carbon source.

Comparison of two duplicate runs (AF-14 and AF-18) shows similar metal produced in each. However, accumulations of slag in the reservoir is highly variable from 10% to 50% of the metal.

Metal produced in bench reactor run AF-20 (SiO_2/Al_2O_3 = 1.22, 80% stoichiometric carbon, 15 ℓ/min CO sweep) was not significantly different from metal produced in previous runs at lower SiO_2/Al_2O_3 ratios. Comparison of this ingot, in which the burden consisted only of kaolinite and coke, with no bauxite, with earlier ingots confirms the previously indicated trend toward more accumulation of slag in the metal reservoir at higher SiO_2/Al_2O_3 ratios, possibly due to formation of a less viscous slag. However, separation of the slag and SiC from the liquid metal within the reservoir was more efficient in earlier runs, at lower SiO_2/Al_2O_3 ratios, than in AF-20.

Metallic product samples from pilot reactor runs VSR-35 and VSR-39 were submitted for phase analysis in order to determine the effects of a change in the temperature profile within the reservoir on the metal product. Phases were identified by optical microscopy, based upon phase compositions determined previously by electron microprobe. Modal compositions were determined by counting 450-550 grains on each sample.

Samples VSR-35 (1), VSR-35 (2), and VSR-35 (3) were taken from the base, middle, and top of the metal ingot formed in the reactor reservoir. Modal compositions of the three samples are listed in Table 3. The major phases are three-phase eutectic microstructure (Al-Si-$FeSiAl_5$), SiC, and hypereutectic Si, with minor to trace amounts of $FeSi_2Al_4$/

$FeSiAl_5$ and $TiSi_2(Al, Fe)/TiSi_2(Al)$ intermetallics. The metal is coarse-grained, with intermetallic crystals up to 3 mm in length and SiC plates up to 10 mm in length. As in metal product from previous runs, the top of the ingot is highly porous due to drainage of low melting liquid into intercrystalline cracks developed by thermal contraction of the metal. The base of the metal ingot consists of a 1-2 mm thick zone of mixed SiC and slag with interstitial metal. The SiC has been derived by reaction from the slag, rather than by reaction with the graphite reservoir. There has also been no reaction between the metal and the reservoir wall.

The three-phase eutectic microstructure exhibits a dendritic morphology which is typical of the alloy purification and product remelt samples, but which has not previously occurred in the primary metal ingots. This morphology consists of branched dendritic crystals of either Al or $FeSiAl_5$ with interstitial metal of eutectic composition. Individual dendritic crystals of Al are up to 1 mm in length, whereas individual dendritic crystals of $FeSiAl_5$ are up to 0.5 mm in length; the latter are occasionally in crystallographic continuity with hypereutectic $FeSiAl_5$ forming a peritectic reaction rim on the surface of intermetallic $FeSi_2Al_4$ crystals, indicating nucleation of eutectic $FeSiAl_5$ on previously formed hypereutectic crystals. The conditions which determine whether the eutectic liquid crystallizes by cellular solidification, yielding dendrites, or by plane front solidification, yielding the platelets of Si and $FeSiAl_5$ in an Al matrix characteristic of previous metal ingots, are not known at this time.

SiC occurs as bladed crystals only, with no Al_4SiC_4 associated. The bladed crystals are up to 5 mm long in the lower part of the ingot, and up to 10 mm long in the porous zone near the top of the ingot.

The peritectic reactions resulting in the formation of $FeSiAl_5$ and $TiSi_2(Al)$ by reaction between the melt and $FeSi_2Al_4$ and $TiSi_2(Al, Fe)$, respectively, have not progressed to the extent typical of earlier metal ingots. This difference is probably the result of a different thermal history for the VSR-35 ingot. The large intermetallic $FeSi_2Al_4$ and $FeSiAl_5$ crystals grow by coalescence of small spherical grains as well as by primary crystallization from the melt.

Two samples from Run VSR-39, designated top one-third and bottom one-third, were taken from the center of the cylindrical ingot at approximately one-third and two-thirds of the distance from the top of the ingot. The phase composition of the metal samples is also listed in Table 3.

Both metal samples consist of Si and $FeSi_2Al_4$ intermetallics in a matrix of eutectic microstructure. In the lower sample SiC occurs as discontinuous radial aggregates of fine platelets. In the upper sample large bladed SiC crystals up to 8 mm in length form an open framework, in which some of the interstices are filled with metal. $TiSi_2$(Al, Fe) is intergrown with SiC in both samples, suggesting co-crystallization of the two phases over a limited temperature range, with continued crystallization of SiC at higher and/or lower temperatures.

Task completion is 60%.

Task No. 11: Supportive Mechanical Engineering

Machining fabrication and engineering design was supplied as needed. A total of 83 man-hrs were expended.

Task completion is 60%.

B. ALLOY PURIFICATION - PHASE C

Task No. 1: Pilot Unit Installation

The crystallization unit assembly was completed. Top heating system and top ceramic cap pieces were installed. The combustion system electrical and mechanical revisions were carried out. Tamper unit modifications were completed and installed.

Equipment modifications and installation were completed and the shakedown run was made during the last week in May.

The back-up tamper was used while repairs were being made to the new device. The new tamper will be used with the crystallizer false bottom in the third quarter.

Task completion is 100%.

Task No. 2: Effects of Pilot Operating Parameters

No runs were made in this quarter.

Task completion is 25%.

Task No. 3: Pilot Unit Modifications

No progress.

Task completion is 0%.

Task No. 4: Supportive Analytical

No runs were made in this quarter. Therefore, no samples were analyzed.

Task completion is 25%.

Task No. 5: Supportive Mechanical Engineering

Mechanical design and fabrication supporting Task No. 1 required 204 man-hrs.

Task completion is 60%.

C. PURIFICATION TO COMMERCIAL GRADE ALUMINUM - PHASE C

Task No. 1: Pilot Unit Installation

Two product removal units (kettles) equipped with jet ejectors for vacuum tapping aluminum from the sump region of the cell have been completed. These units consist of a steel shell, lined with two inches of insulating block (HW-177) and three inches of insulating castable (SJP). Molten Al will be tapped into a graphite mold positioned within the refractory-lined kettle. Molten metal will be transferred from the cell to one of two available kettles through a preheated fiberfrax-lined steel pipe. The completed unit is equipped with manual and automatic vent valves and a high intensity viewing lamp.

During the second quarter, aluminum buswork was installed between the cell electrodes and the DC rectifier. This buswork has been designed for 15,000 amps (DC) service. It consists of three 1-in thick, 12-in high aluminum pieces connected to the four anode electrodes. A similar buswork arrangement was installed between the cathode electrodes and the rectifier.

Buswork was also connected from the cell electrodes to the 30 KVA AC transformer. AC power will provide heat to the cell to maintain the temperature of the bath, when the DC rectifier has been switched off. Under normal DC operating conditions, the AC buswork will be electrically isolated to prevent damage to the transformer.

Water-cooling jackets have been incorporated into the shell of the cell to remove heat generated during the electrolytic process, and to maintain a desirable freeze line of bath in the refractory insulation. A piping system to individually supply and meter H_2O to each of the jackets in the shell has been completed. With this system, municipal water is filtered, regulated, and metered to each circuit. The flow rate and temperature of H_2O to each section of the shell can be monitored and controlled during normal operation.

The system for supplying molten bath ($LiCl-NaCl-AlCl_3$) at temperatures above 650°C to the cell has been completed. A 25 KVA AC power supply was installed for melting bath in the inconel chamber. A portable tapping station has been constructed so that the transfer of molten bath to the cell can be controlled and regulated in a safe manner.

Inconel strips suitable for inserting into the corners of the cell are currently being fabricated. These strips will be connected in series to heat the cell to start-up temperature (660°C) with DC power.

Task completion is 70%.

Task No. 2: Effects of Pilot Operating Parameters

No Progress.

Task completion is 0%.

Task No. 3: Pilot Unit Modifications

No Progress.

Task Completion is 0%.

Task No. 4: Support Pilot Operations

No Progress.

Task completion is 50%.

Task No. 5: Supportive Analytical

No Progress.

Task completion is 25%.

Task No. 6: Supportive Mechanical Engineering

Assistance was provided for Task No. 1. A total of 79 man-hrs were expended.

Task completion is 80%.

PHASE C THIRD QUARTER PROGRAM

Administrative

Cost substantiation data will be submitted to DOE regarding the projected cost overrun.

Technical

Reduction: A special technical report addressing the feasibility of the low pressure, combustion-heated reduction process will be issued. Pilot reactor VSR-3 will be further modified to enhance the possibilities of tapping molten alloy. Burdens containing alumina hydrate, clay, and coke will be evaluated. Separation of metal from carbides using molten salt fluxes will be tested. The pilot (large bench) crucible reactor will be converted to a small submerged arc unit. Studies will continue in the bench reactor on the effects of carbon stoichiometry on alloy yield. Calculations will be made on the effects of pressure on the combustion-heated blast furnace concept. Improved heat and mass balances will be determined for a blast-arc with midwestern coal, and both a blast-arc and submerged arc with low ash, low-iron coal. Phase identification analyses of pilot and bench reactor product samples will be continued. The SOLGASMIX-REACTOR computer program will be expanded to handle recycle for the Al-Si-C-O-Fe system.

Alloy Purification: The pilot unit will be operated on a simulated reduction alloy with a nominal composition of 66% Al, 30% Si, 3% Fe, and 1% Ti.

Purification to Commercial Grade Aluminum: Installation of the pilot cell should be completed. The bench scale unit will be operated to correlate metal penetration of the membrane with electrolyte composition.

Cost Summary

Expenditures for the second quarter of Phase C totaled $397,027. Distribution was $261,230 for Reduction, $49,585 for Alloy Purification, and $86,212 for Purification to Commercial Grade Aluminum. Cumulative spending for Phase C was $880,227. Total cumulative spending through the first

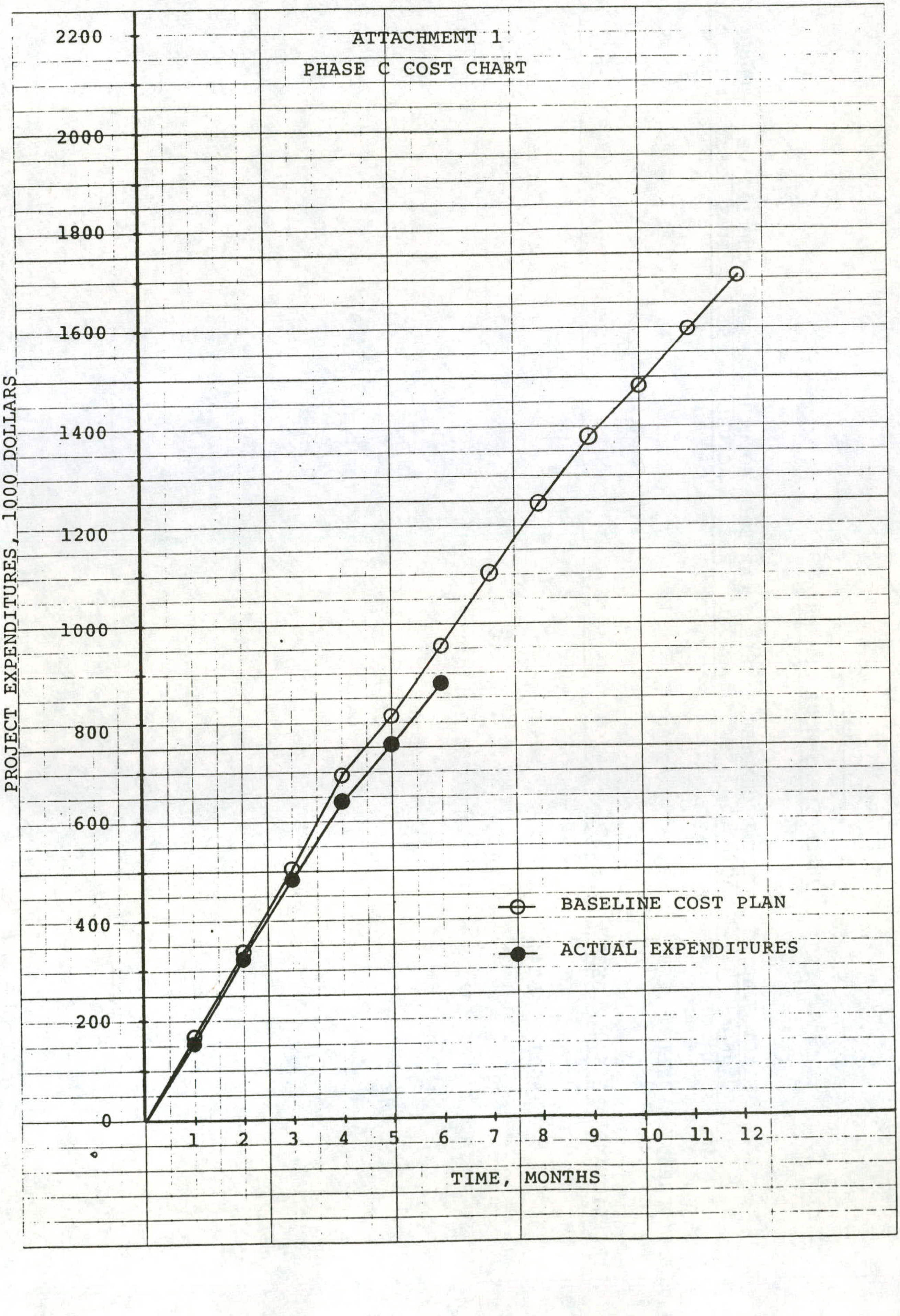
34 months of the contract was $4,276,241. Actual spending for Phase C is compared to estimated spending in Attachment 1.

Assigned Personnel

The actual man-hrs expended by engineers and technicians for the second quarter of Phase C are shown in Attachment 2 and compared to estimated man-hrs. For engineers, actual was 5.5% above estimated. For technicians, actual was 0.9% below estimated. Total cumulative man-hrs were 1.0% above estimated.

Task/Milestone Schedule

Attachment 3 shows the task-time relationship for the three major tasks from initiation in DOE fiscal year 1977 (fourth quarter) through completion of the current contract in DOE fiscal year 1981 (first quarter). Completed milestones are noted by filled-in circles.



ATTACHMENT 1

PHASE C COST CHART

ATTACHMENT 2

PHASE C MAN-HOUR SUMMARY

	FIRST QUARTER		SECOND QUARTER		THIRD QUARTER		FOURTH QUARTER		CUMULATIVE		CUMULATIVE
	ACTUAL	EST.	ACTUAL	EST.	ACTUAL	EST.	ACTUAL	EST.	ACTUAL	EST.	% DEVIATION
ENGINEERS	3227	2705	2560	2780					5787	5485	+5.5
TECHNICIANS	6464	6539	6227	6262					12691	12801	-0.9
TOTAL	9691	9244	8787	9042					18478	18286	+1.0

ALUMINUM COMPANY OF AMERICA

ALCOA LABORATORIES

Attachment 3

Direct Reduction Project

Task/Milestone Schedule

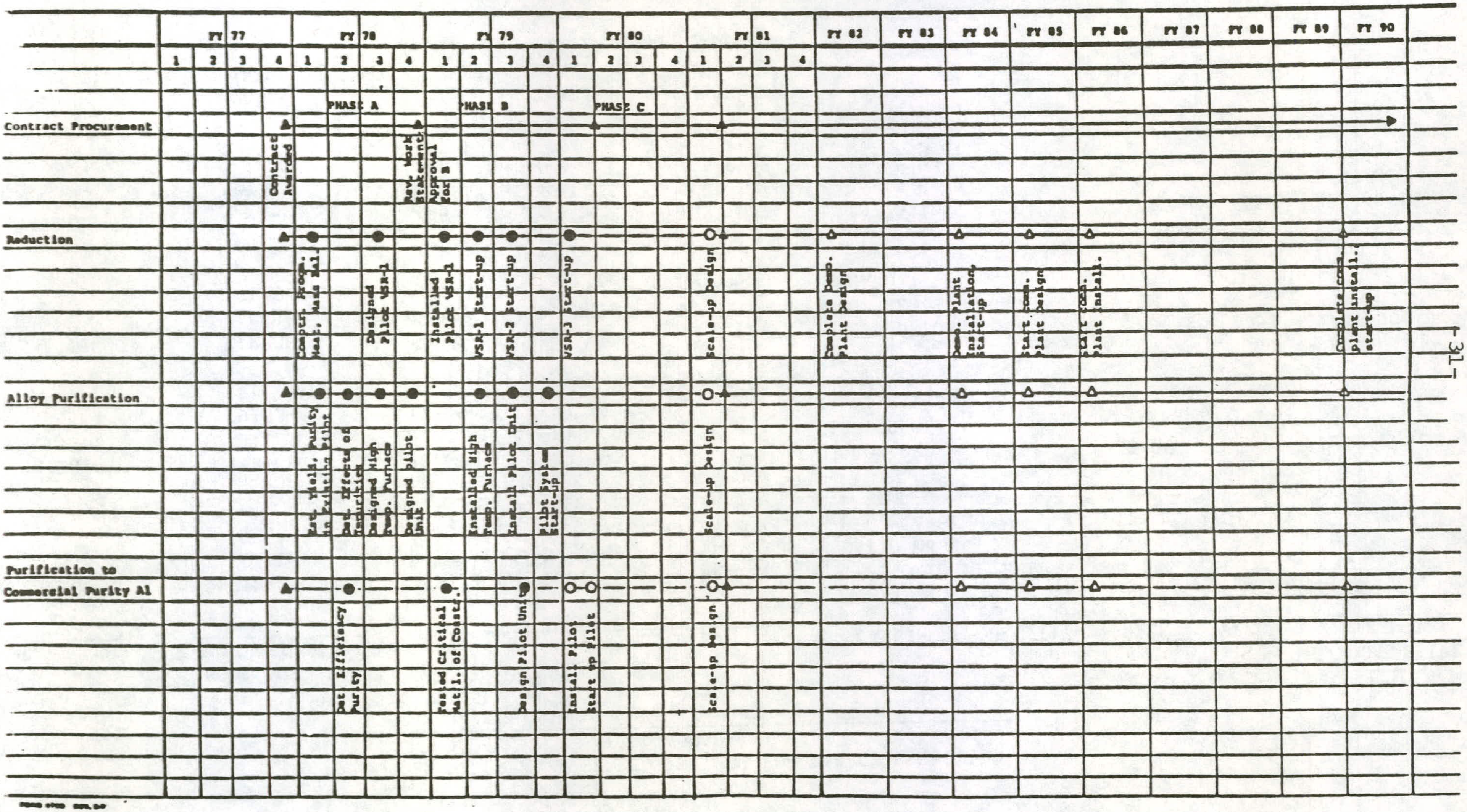


TABLE 1

BURDEN MATERIAL PREPARED IN 1980 FOR DIRECT REDUCTION
(74D1450601)

EXPT. NO.	BOOK AND PAGE NO. METHOD	RAW MATERIALS USED			CARBON	QUANTITY	SIZE	YIELD	FIRED COMPRESSIVE STRENGTH
5A1106	EIRICH BALLS 19185-35	288.13 H. CLAY (57.626%)	100.855 B. BXTE (20.171%)	111.015 MET. COKE (22.203%)	.788 OXIDE RATIO @80% RED. CARB.	500#		63%	
5A1107	EIRICH BALLS 19185-40	76.61 H. CLAY (76.61%)	23.39 MET. COKE (23.39%)		80% RED. CARB. SiO_2/Al_2O_3 RATIO	100#	3/8" x 6M		212#/sq in
5A1117	EIRICH BALLS 19185-42	285.95 H. CLAY (57.19%)	99.55 B. BXTE. (19.91%)	114.50 MET COKE (22.90%)	.788 OXIDE @80% RED. CARB.	500#	3/8" x 6M	59%	178#/sq in
5A1126	EIRICH BALLS 19185-48	264 H. CLAY (52.8%)	107 B. BXTE. (21.4%)	129 COKE BREEZE (25.8%)	.787 OXIDE RATIO @80% RED. CARB.	500#	3/8" x 4M	55%	287/sq in 1/4" dia
5A1149	SMALL DISK	4.375 H. CLAY (35%)	5.875 WARR. COAL (47%)	2.250 CALC. Al_2O_3 (18%)		12.5#			
5A1167	EIRICH BALLS	265 H. CLAY (53.0%)	129 PROP. #10 M. COKE (25.8%)	107.5 CALC. Al_2O_3 (21.5%)		500#	3/8" x 4M	56%	728#/sq in 1/4" dia

TABLE 2

COMPOSITION OF REACTANTS AND PRODUCTS FROM VSR-38

	AL	SI	FE	TI	$\frac{AL}{AL + SI}$
METALLIC CONTENT OF ORE PELLETS 5A1106 WITH CARBON AND OXYGEN REMOVED.	54.0	37.6	3.95	2.84	.59
CONTENT OF FILTERED* ALLOY FROM VSR-38	68.4	23.2	4.94	1.86	.75

*Unfiltered ingot was not present in the catchbasin from VSR-38 for comparison. Furthermore, the metallic content retained in the packed bed could not be accurately sampled or analyzed.

TABLE 3

PHASE COMPOSITION OF VSR-35 AND VSR-39 METAL INGOT (VOLUME %)

	VSR-13(1) BASE	VSR-13(2) MIDDLE	VSR-13(3) TOP	VSR-39(1) BOTTOM 1/3	VSR-39(2) TOP 1/3
AL	37%	42%	36%	49%	44%
Si (1)	19	20	24	16	19
$FeSi_2Al_4$	7	9	11	6	6
$FeSiAl_5$ (2)	7	6	8	<1	<1
$TiSi_2$(Al, Fe)	10	6	8	5	1
$TiSi_2$(Al)	1	1	1	-	-
SiC	19	18	13	24	31

(1) Included both eutectic and hypereutectic Si

(2) Includes both eutectic and intermetallic $FeSiAl_5$; estimated 80% of this is in eutectic microstructure.

TABLE 4

EFFECT OF SiO_2/Al_2O_3 CARBON STOICHIOMETRY AND CO SWEEP ON METAL PRODUCTION

Run	Ore SiO_2/Al_2O_3 Wt. Basis	Consumed Charge / CO	% Total C	Wt. Metal / Consumed Charge	Reaction Zone Wt.	Metal Producing Temperature
AF7	0.6	---	100 %	0.210	---	2050
AF12	0.784	1.815	80.55	0.164	1280 gm	2050
AF13	0.502	2.199	80.0	0.161	1341	2055
AF14	0.993	2.035	80.0	0.177	759	2060
AF15	0.788	1.950	80.0	0.191	718	2065
AF16	0.784	1.042	80.55	---	558	2030
AF17	0.784	0.579	80.55	---	362	2140
AF18	0.993	1.393	80.0	0.195	927	2060
AF20	1.224	0.730	80.0	0.209	122	2100
AF21	1.224	0.491	80.0	---	200	2060
AF22	1.224	1.173	85.0	---	150	2090
AF23	1.224	---	85.0	0.239	334	2075
AF24	0.469	---	80.0	0.325	---	2060
AF25	0.68	---	85.0	0.282	475	2020
AF26	0.785	---	84.5	0.274	927	2020
AF27	0.502	---	84.7	0.334	661	2035
AF28	0.785	---	91.3	0.294	127	2140

CONSUMED CHARGE = TOTAL ORE FED - REMAINING LOOSE CHARGE IN THE AUGER

TABLE 5

CARBON STOICHIOMETRY OF ORE BURDEN

Run	Ore SiO_2/Al_2O_3 Wt. Basis	% C Ore	% C for SiC	% Total C	Carbon Source
AF7	0.6	71.0	58	100	met and pet coke
AF12	0.784	80.55	67	80.55	met coke
AF13	0.502	64.24	51	80.0	char and pet coke
AF14	0.993	63.75	76	80.0	char and pet coke
AF15	0.788	56.74	67	80.0	char and pet coke
AF16	0.784	80.55	67	80.55	met coke
AF17	0.784	80.55	67	80.55	met coke
AF18	0.993	63.75	76	80.0	char and pet coke
AF20	1.224	72.62	84	80.0	met and pet coke
AF21	1.224	72.62	84	80.0	met and pet coke
AF22	1.224	72.62	84	85.0	met and pet coke
AF23	1.224	72.62	84	85.0	met and pet coke
AF24	0.469	---	49	80.0	pet coke
AF25	0.68	77.67	63	85.0	met and pet coke
AF26	0.785	74.94	67	84.5	char, met, and pet cok
AF27	0.502	64.24	51	84.7	char and pet coke
AF28	0.785	91.30	67	91.3	char and met coke

TABLE 6

METALLOGRAPHIC PHASE ANALYSIS IN VOLUME %

			Uncoalesced Metal				
Run	% Total Carbon	Ore SiO_2/Al_2O_3 Wt. Basis	Slag	Si	SiC	Al_4SiC_4	Si/Al Wt. % Basis
AF7	100	0.6	--	16	12	2	--
AF12	80	0.784	9	15	15	--	--
AF13	80	0.502	6	19	17	1	--
AF14	80	0.993	2	18	22	--	--
AF15	80	0.788	3	16	11	--	0.48
AF18	80	0.993	5	10	42	--	0.60
AF20	80	1.224	5	27	15	--	--
AF23	85	1.224	--	8	64	--	1.37
AF26	84.5	0.785	--	12	51	1	0.62
AF27	84.7	0.502	--	11	45	1*	0.99

* Al-Si carbide verified by both x-ray diffraction and metallography

** $Al_4Si_2C_5$ identified by x-ray diffraction

TABLE 6 (Continued)

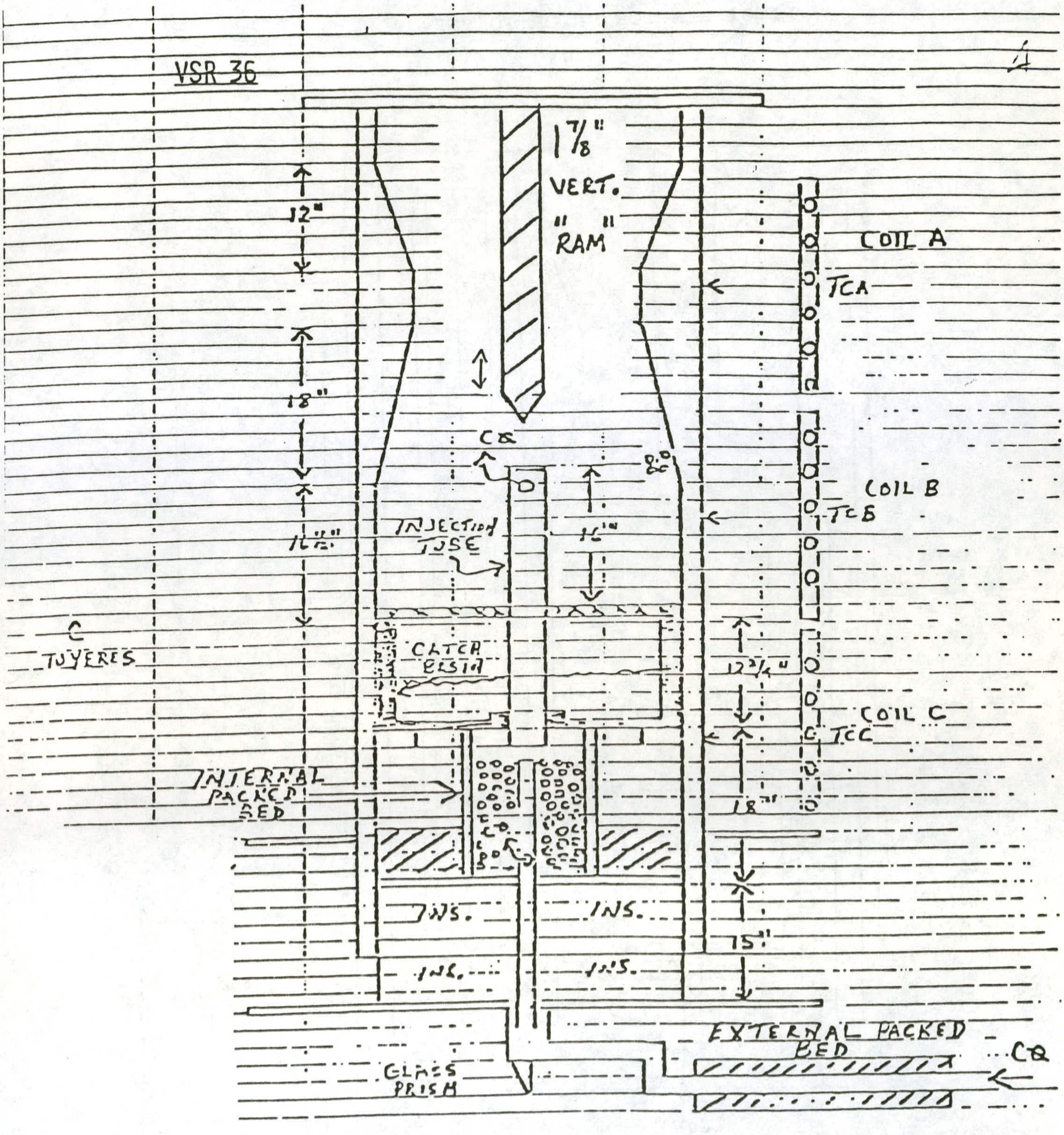
METALLOGRAPHIC PHASE ANALYSIS IN VOLUME %

	Metal Below Slag Layer					Metal Above Slag Layer					Top Metal				
Run	Slag	Si	SiC	Al_4SiC_4	Si/Al Wt. % Basis	Slag	Si	SiC	Al_4SiC_4	Si/Al Wt. % Basis	Slag	Si	SiC	Al_4SiC_4	Si/Al Wt. % Basis
AF7	2	18	31	9	0.43	2	14	30	2	0.46	—	27	10	2	0.45
AF12	10	21	18	—	0.59	—	15	31	—	0.84	—	20	15	—	0.61
AF13	5	15	23	3**	0.38	1	18	16	6	0.39	1	20	11	2	0.51
AF14	20	9	53	—	—	—	24	9	—	0.92	—	25	2	—	0.66
AF15	16	16	23	—	0.58	—	—	—	—	1.08	—	24	11	—	0.44
AF18	15	6	60	—	0.86	—	—	—	—	—	2	22	14	—	0.53
AF20	—	—	—	—	—	5	19	27	—	0.80	2	25	28	—	0.80
AF23	—	15	38	—	0.80	—	13	23	—	0.53	—	24	1	4	0.42
AF26	—	11	52	2	1.13	—	18	17	4	0.46	—	18	22	7	0.44
AF27	—	12	45	6*	0.99	—	17	27	7*	0.39	—	6	23	9	0.42

* Al-Si carbide verified by both x-ray diffraction and metallography

** $Al_4Si_2C_5$ identified by x-ray diffraction

FIGURE 1
PILOT REACTOR PRIOR TO VSR-36



*VERTICAL STROKE "RAM" AND SHAFT TAPER ADDED

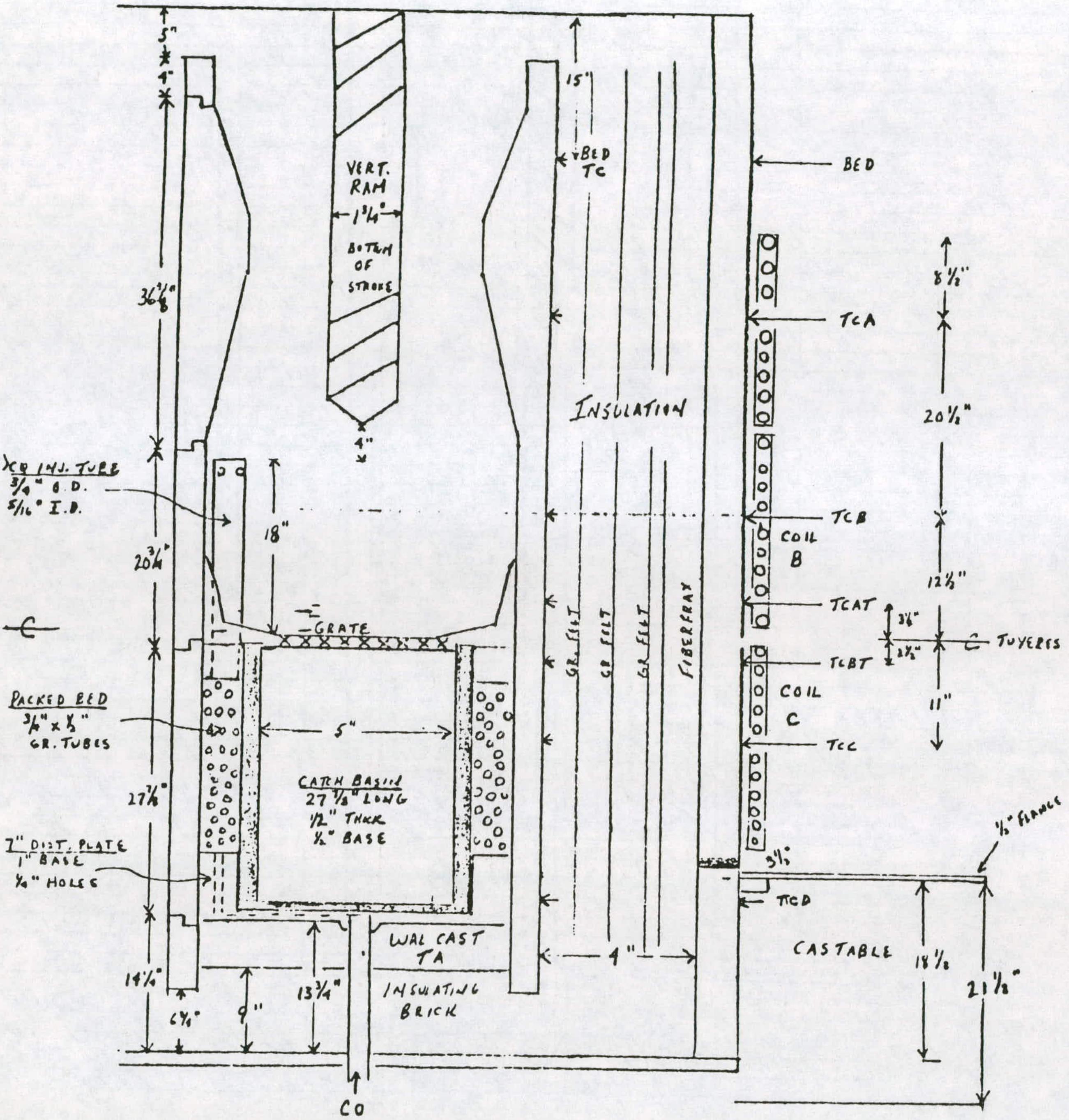
*EXCELLENT BED MOVEMENT (7KG ORE/HR)

*MOVEMENT MAINTAINED AT 100 SCFH OF CO INJECTION

*6KG INGOT

FIGURE 2

PILOT REACTOR PRIOR TO VSR-38



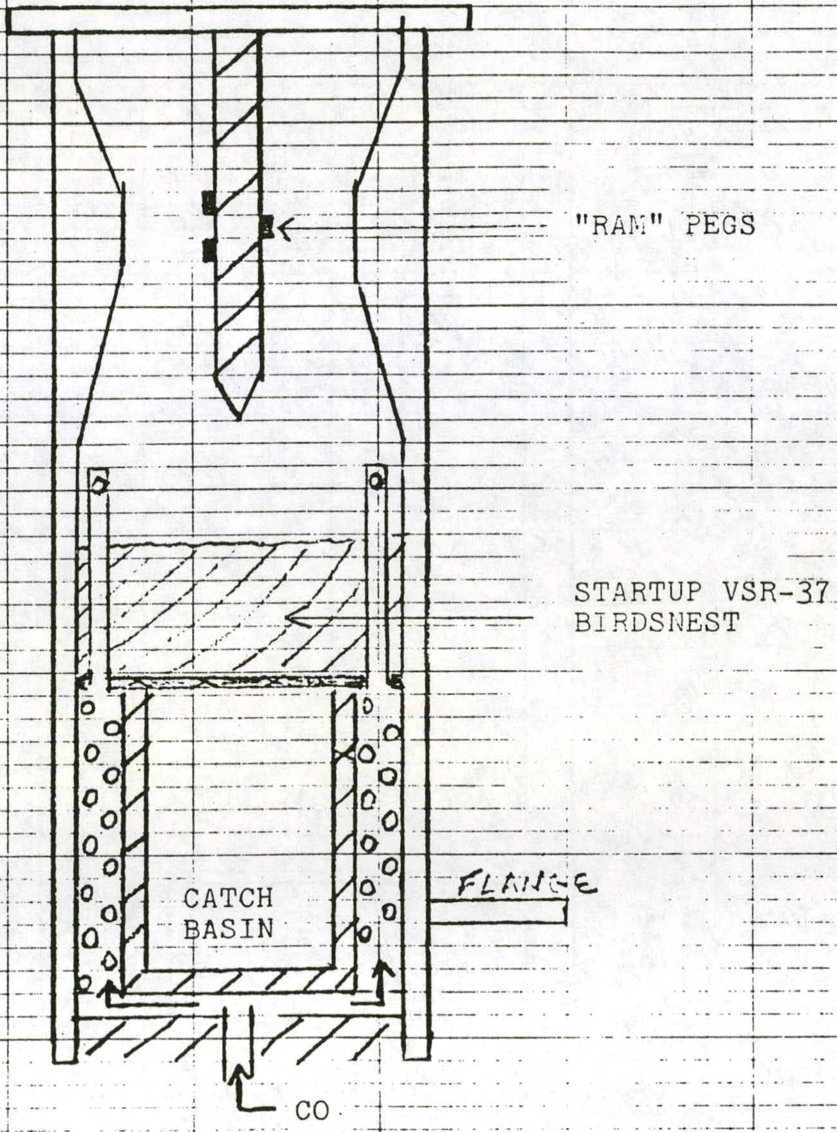
HEIGHT 1 BLOCK = 1"
WIDTH 4 BLOCKS = 1"

A) SUSCEPTOR THICKNESS 25/32"
B) " I.D. 8 1/8"
C) CATCH BASIN 1/4" BELOW 2ND SUSCEPTOR PIECE SHIPLAP

D) SHELL
O.D. 19.5"
I.D. 17.5"

FIGURE 3

VSR-38 START-UP



*HORIZONTAL PEGS ADDED TO RAM

*STARTUP WITH "BIRDSNEST" FROM VSR-37 TO DEMONSTRATE COMMERCIAL STARTUP AFTER SHUTDOWNS OR DELAYS

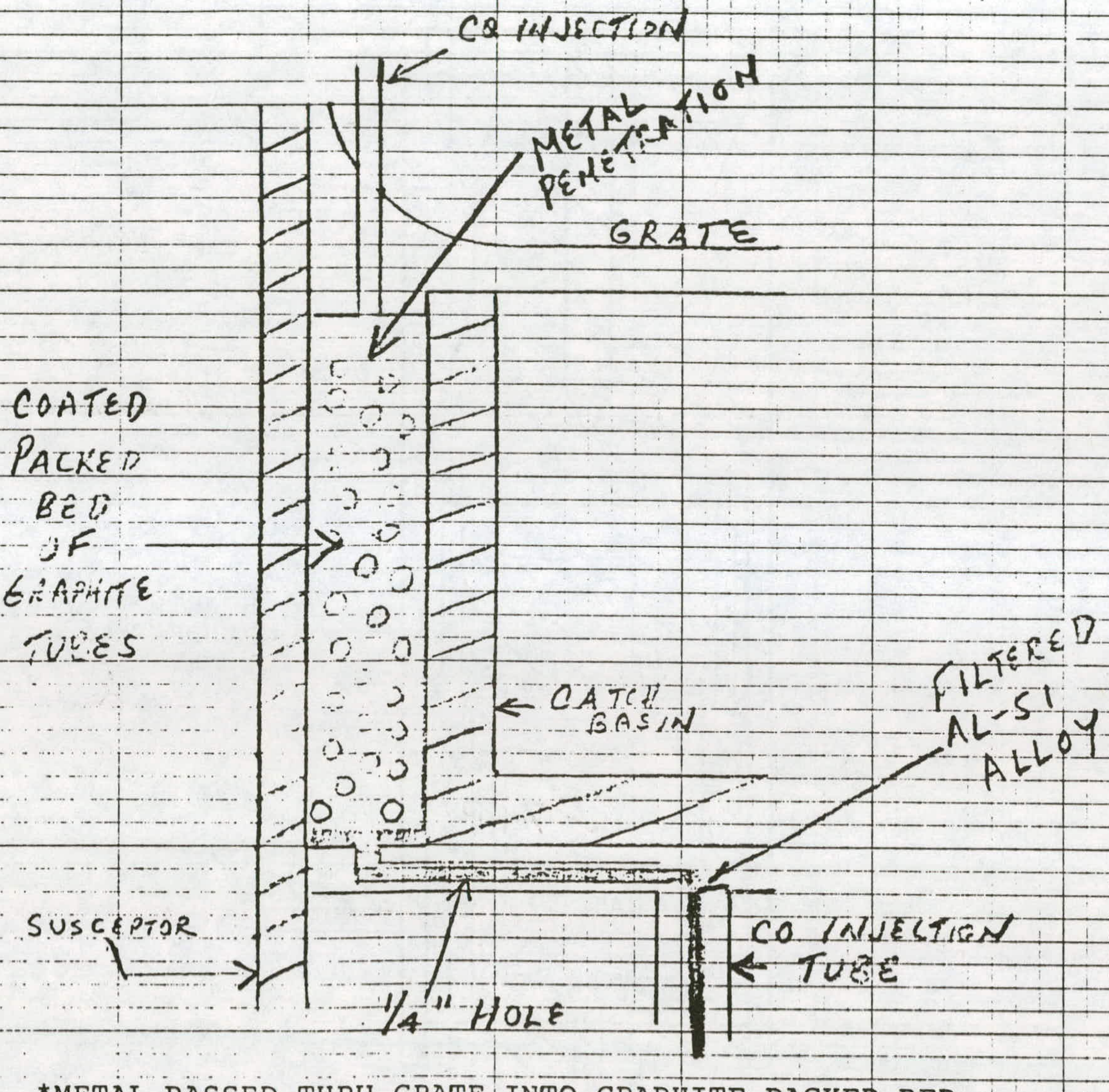
*NO CO INJECTION

*SOLIDS MOVEMENT EXCEEDED 8.7 KG PER HR

*55 KG OF ORE CONSUMED

FIGURE 4

VSR-38 AUTOPSY



*METAL PASSED THRU GRATE INTO GRAPHITE PACKED BED

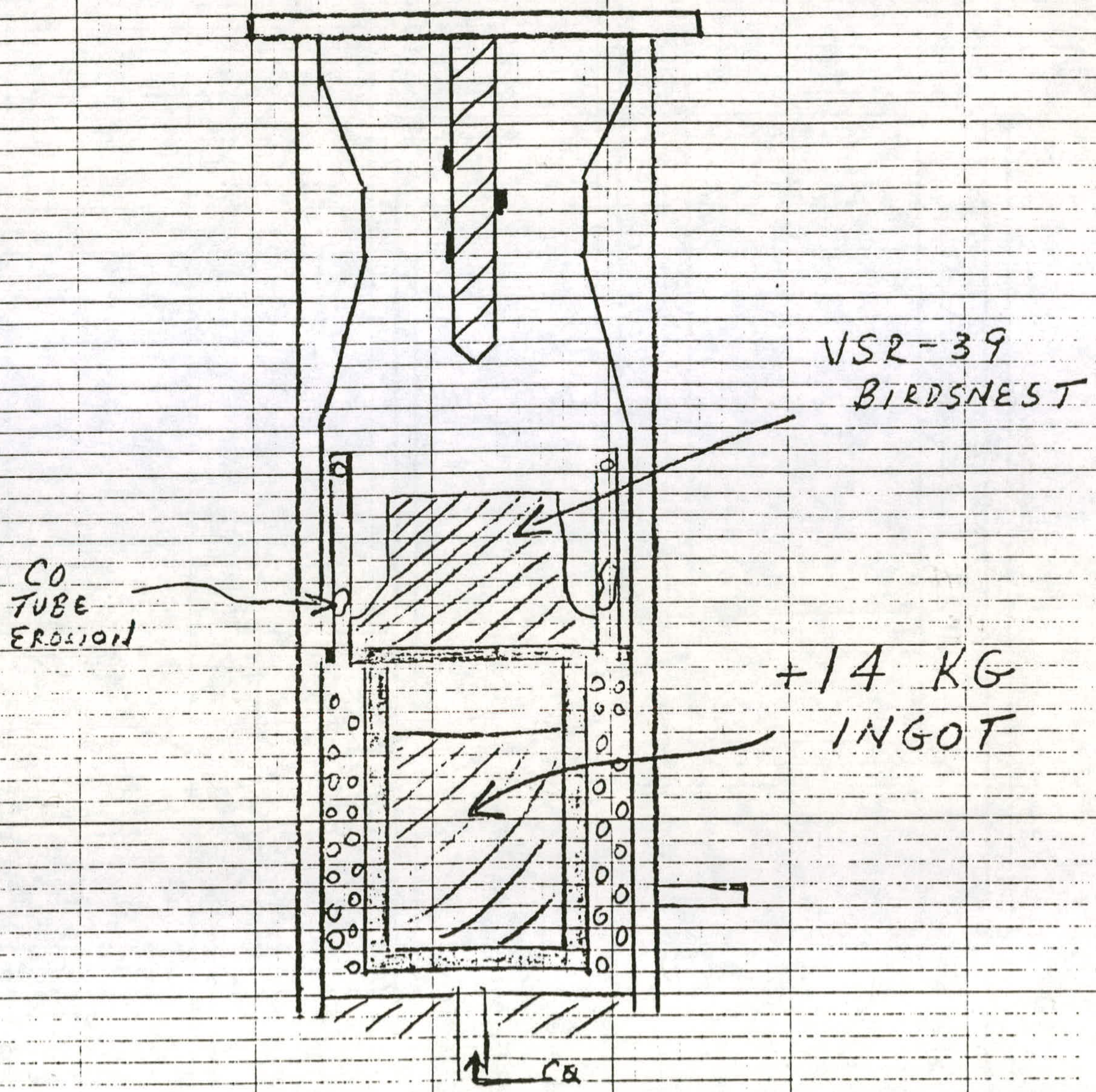
*SIC FILTERED OUT

*"PURIFIED ALLOY" PASSED THRU 1/4 INCH HOLES IN DISTRIBUTOR TO REACTOR BOTTOM

*ALLOY CHARACTERISTICS "VERY CLEAN"

FIGURE 5

VSR-39 AUTOPSY



*RECORD ALLOY PRODUCTION WITH 100 SCFH OF CO INJECTED INTO STAGE I

*A BED MOVEMENT OF 7 KG PER HR MAINTAINED AT 200 SCFH OF INJECTED CO

*CO INJECTION INTO STAGE III DURING LAST 2 HOURS OF OPERATION. BIRDSNEST EROSION

FIGURE 6

VSR TAPPING LAYOUT

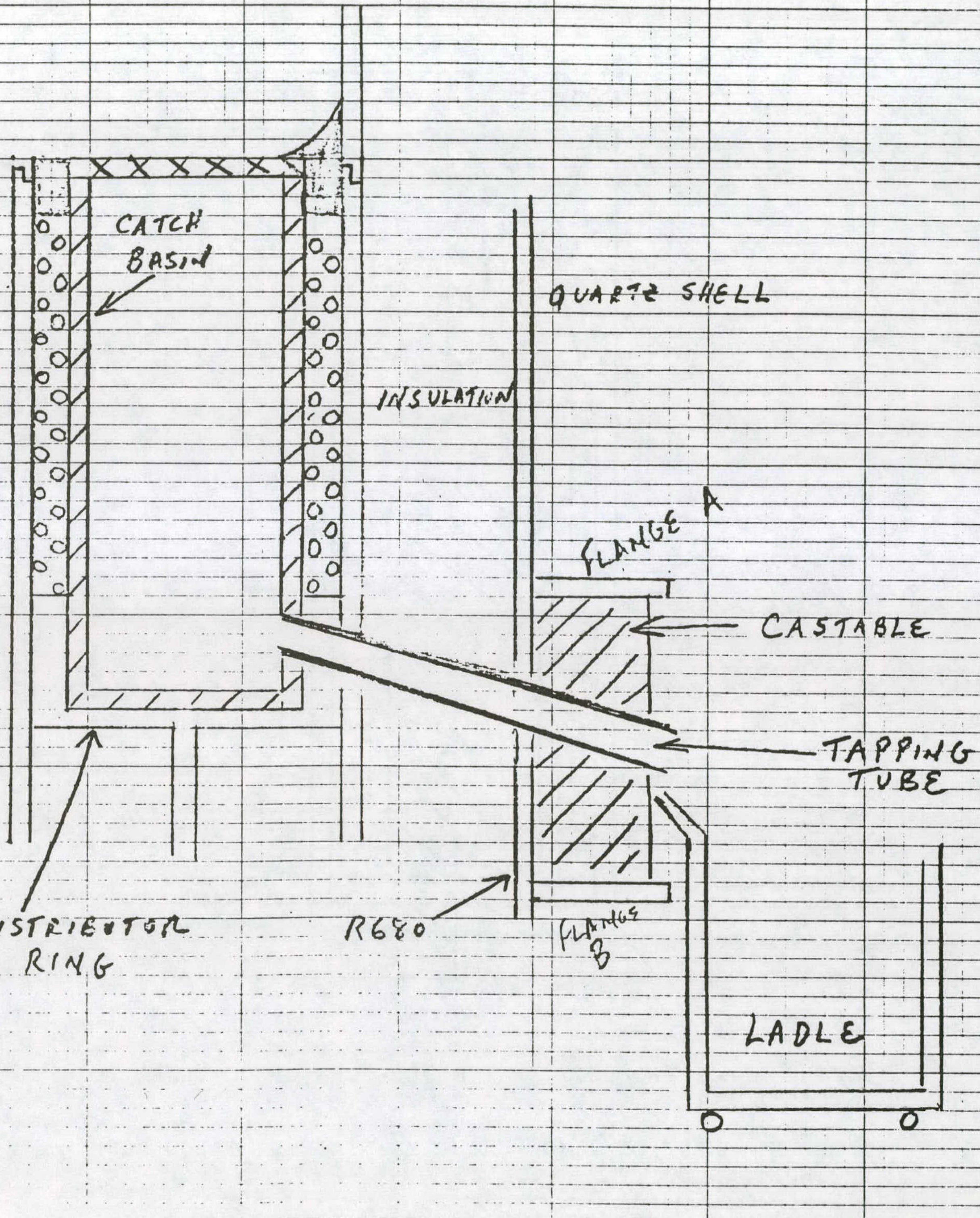
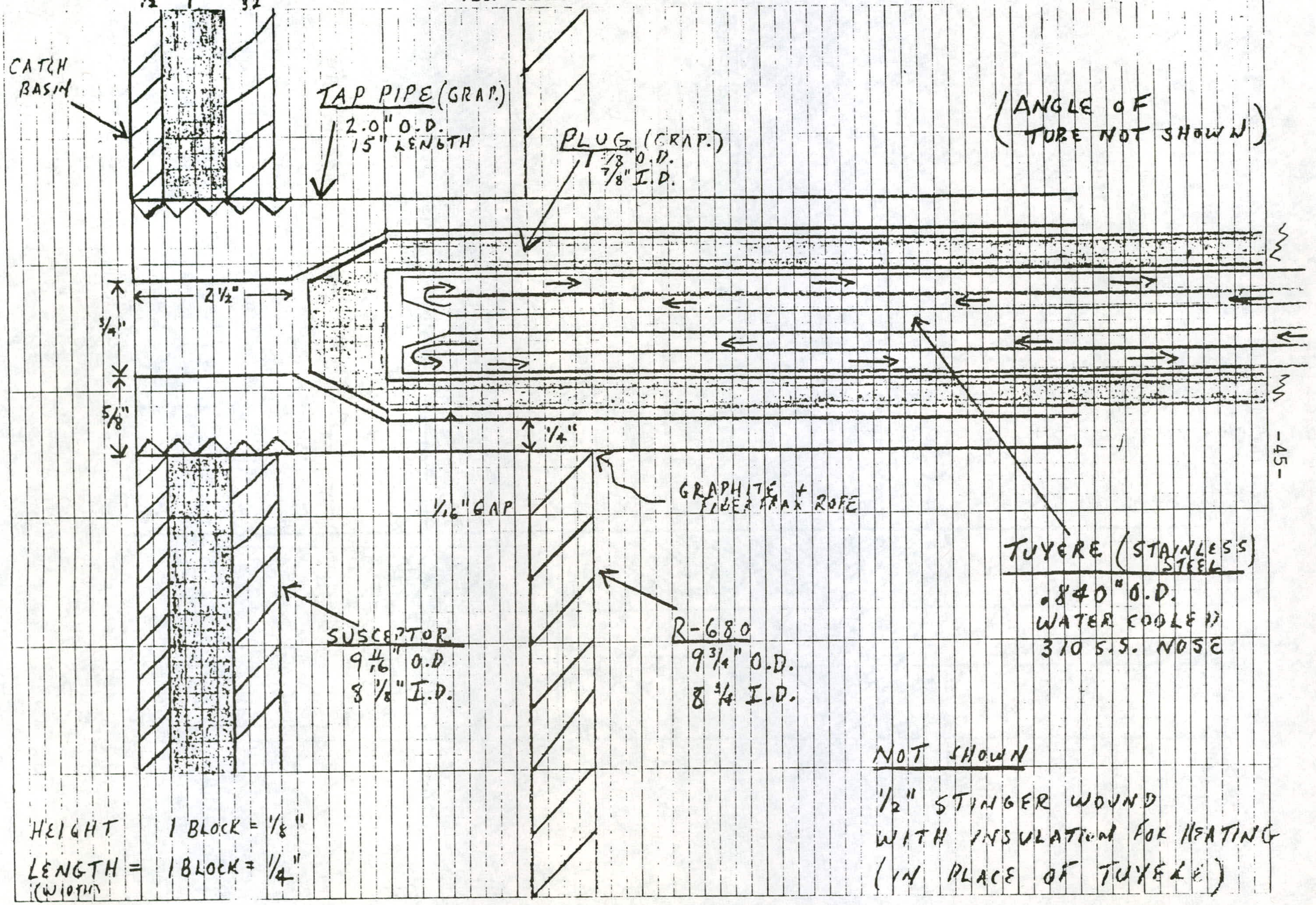


FIGURE 7
VSR TAPHOLE DESIGN



AF-16

80-4-10

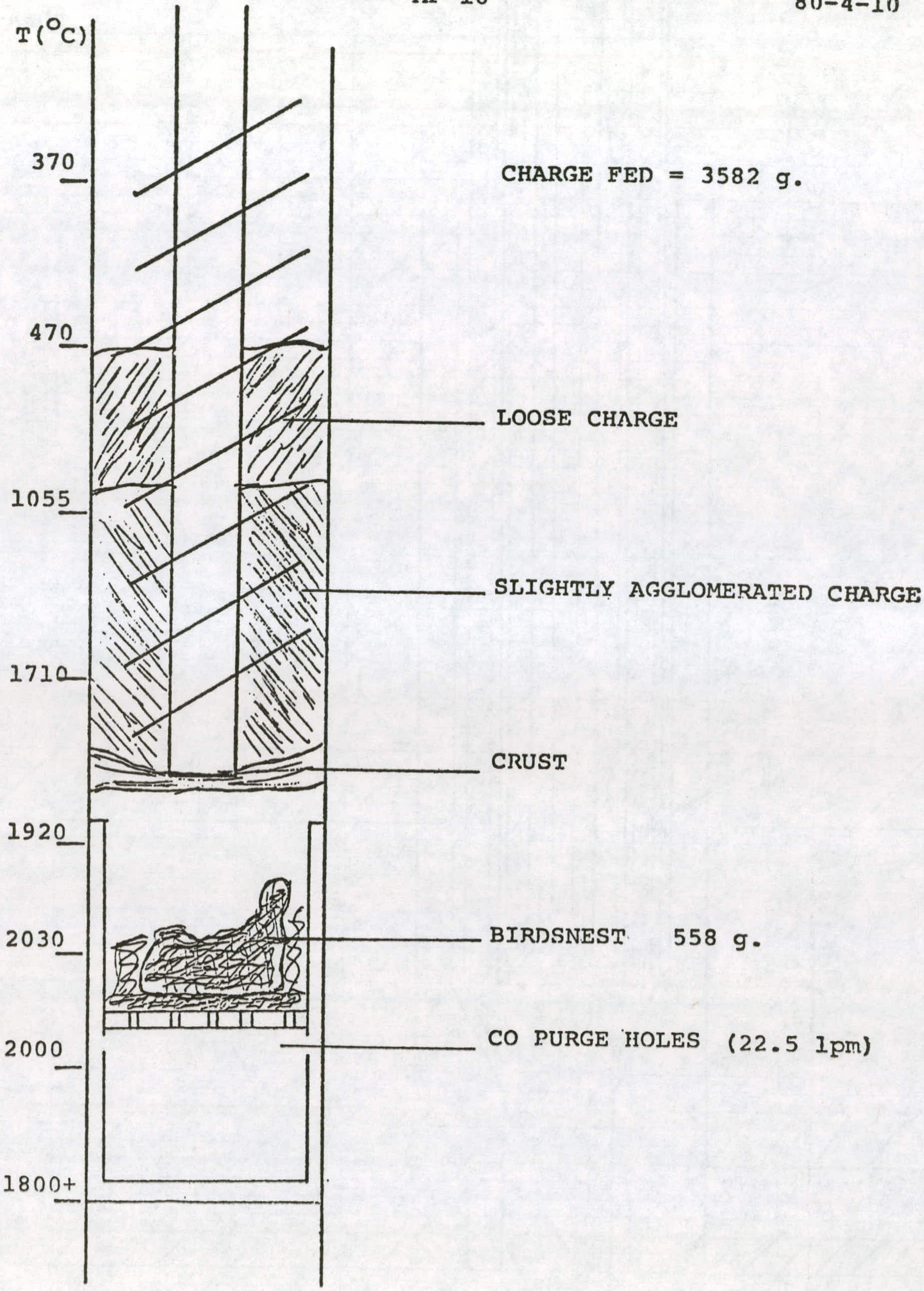


FIGURE 8

AF -17 80-4-15

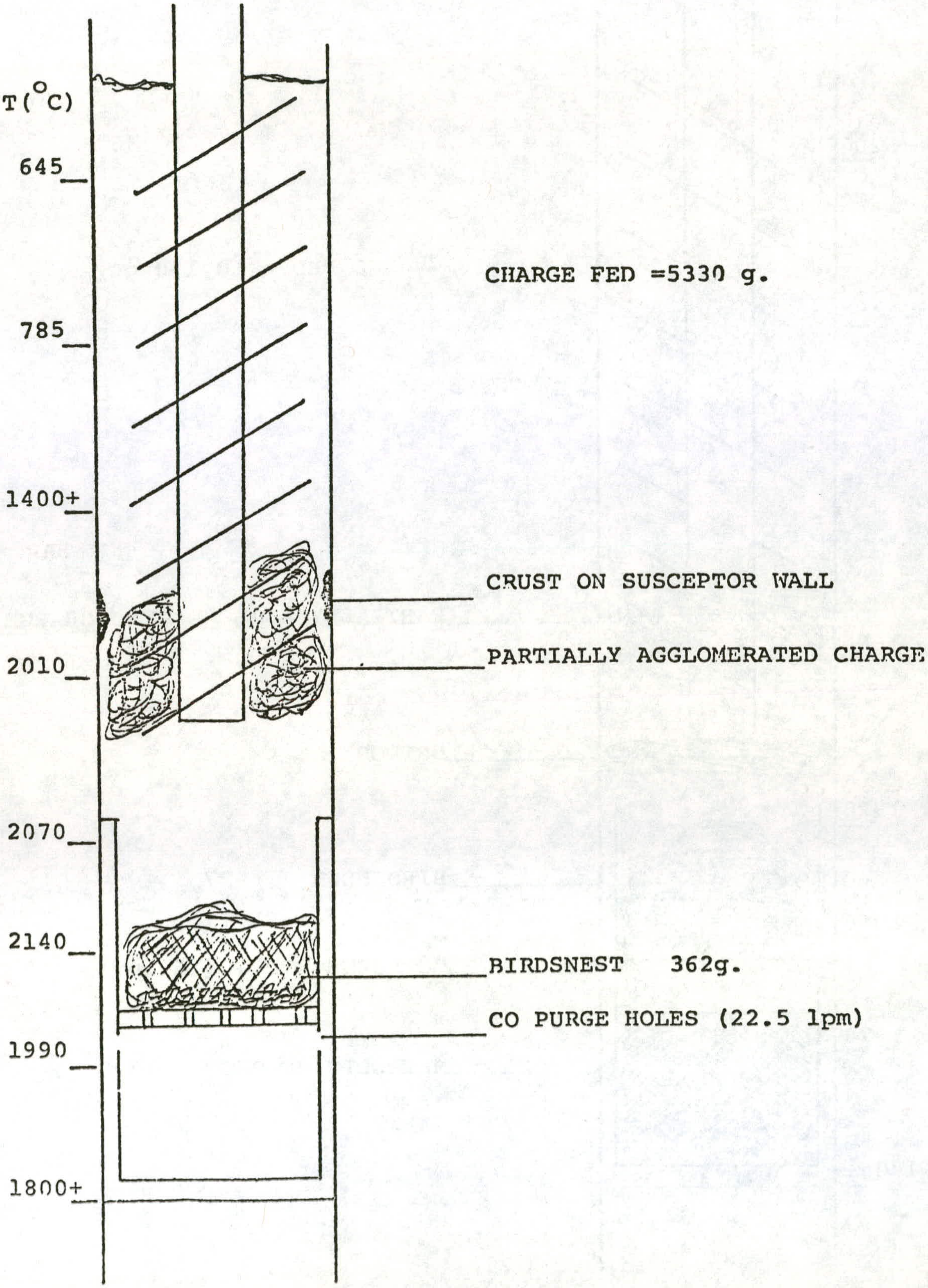


FIGURE 9

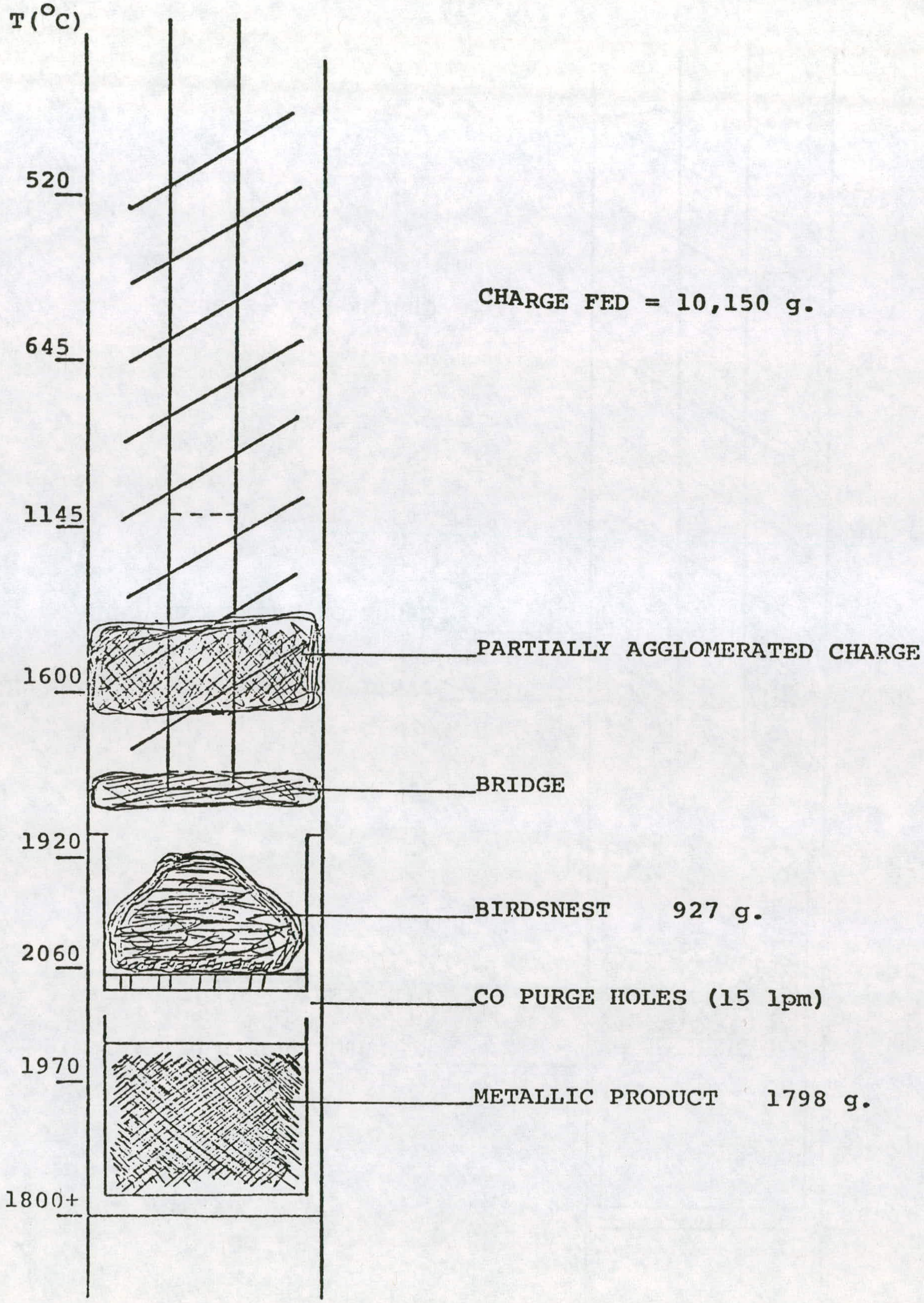

T(°C)
520
645
1145
1600
1920
2060
1970
1800+
CHARGE FED = 10,150 g.
PARTIALLY AGGLOMERATED CHARGE
BRIDGE
BIRDSNEST 927 g.
CO PURGE HOLES (15 lpm)
METALLIC PRODUCT 1798 g.

FIGURE 10

AF-20 80-5-7

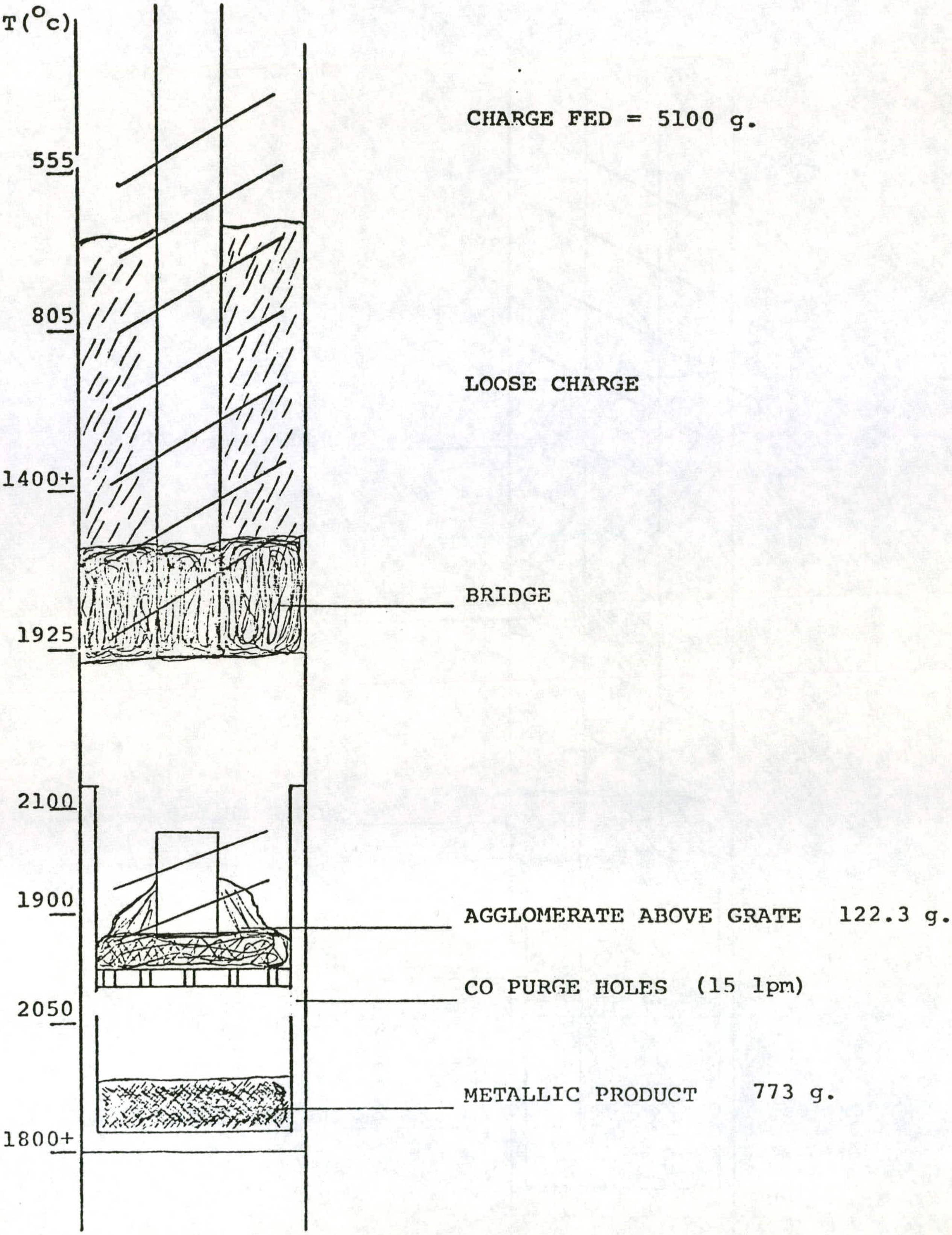


FIGURE 11

AF-21

80-5-14

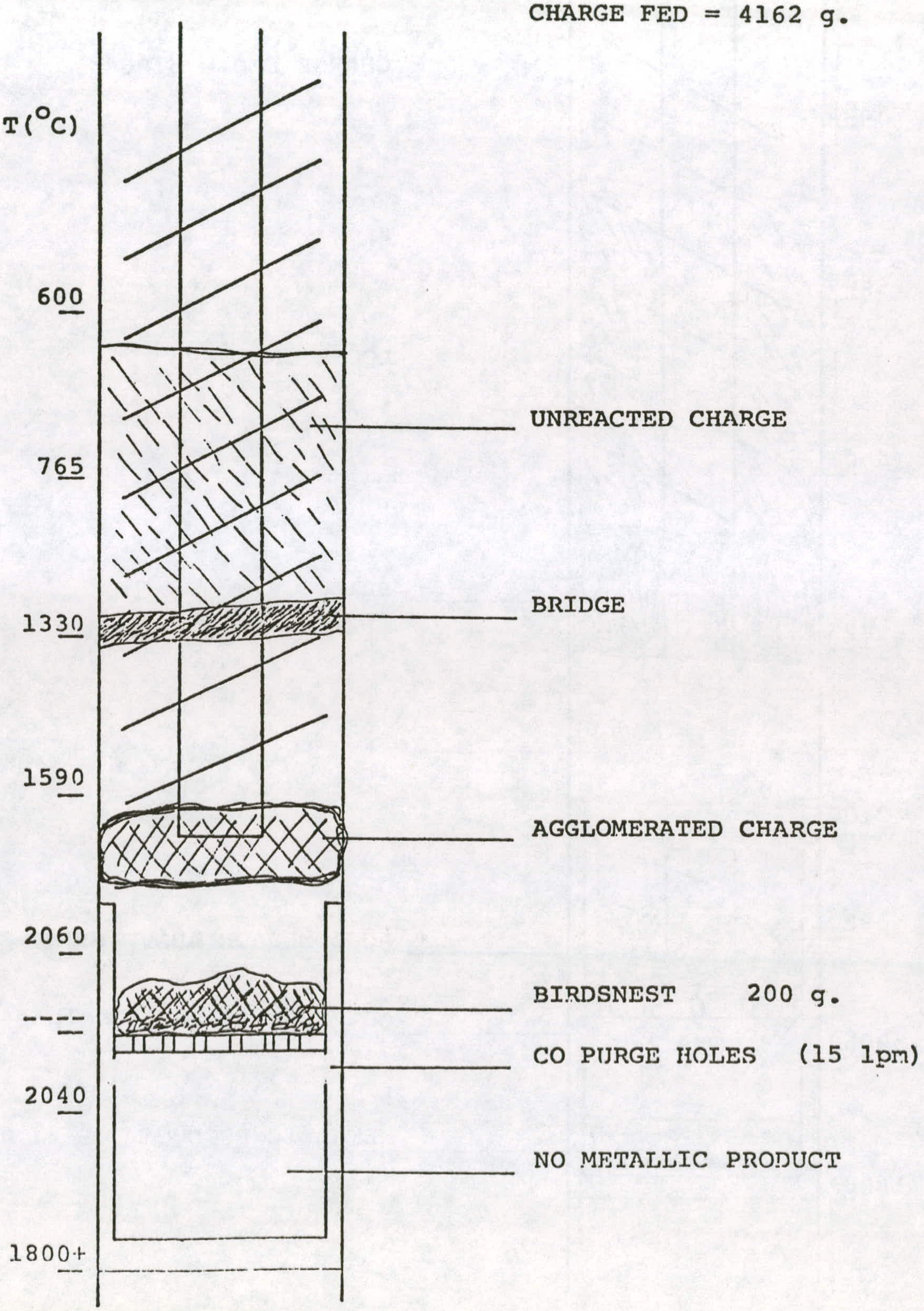


FIGURE 12

AF-22 80-5-20

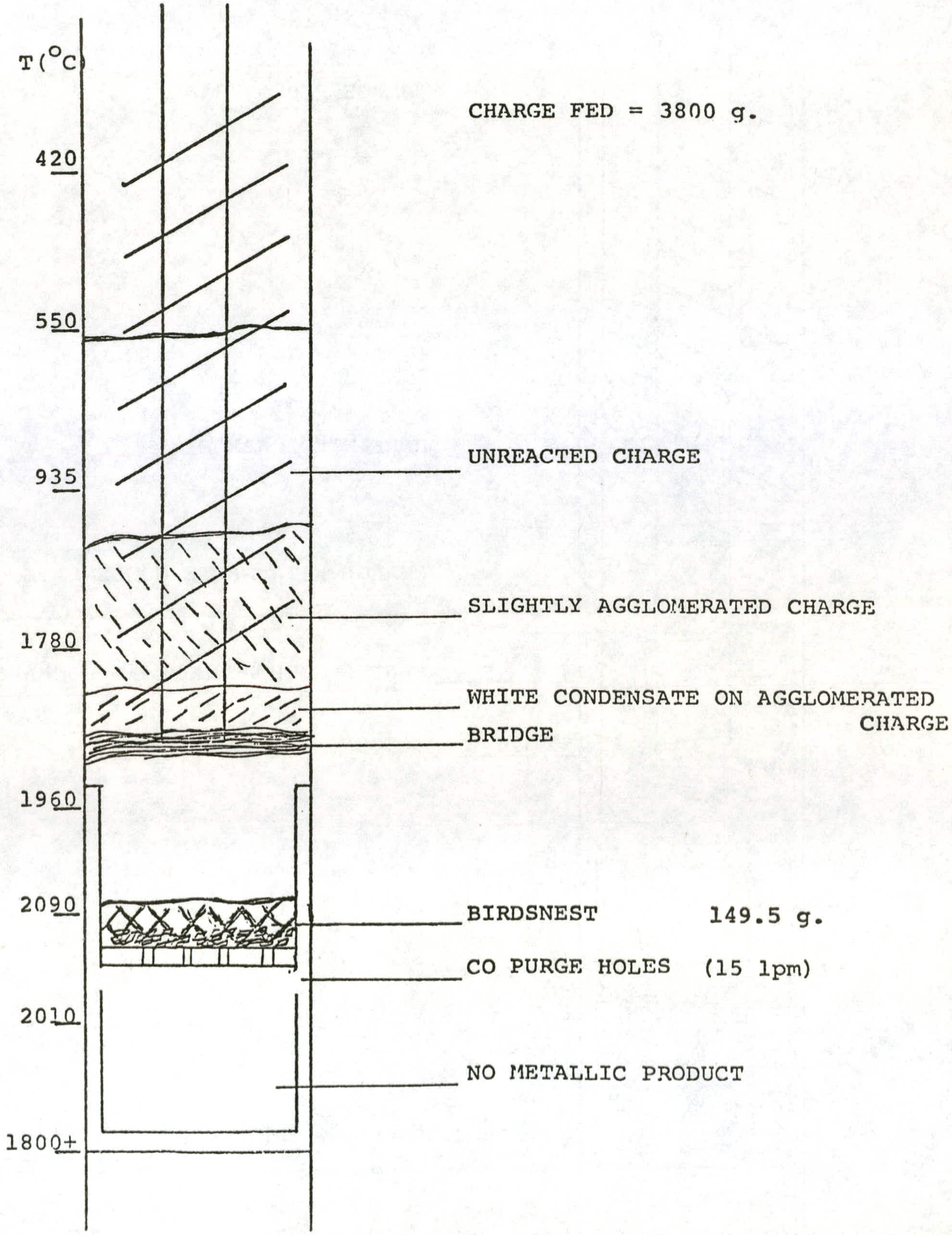


FIGURE 13

AF -23 80-5-22

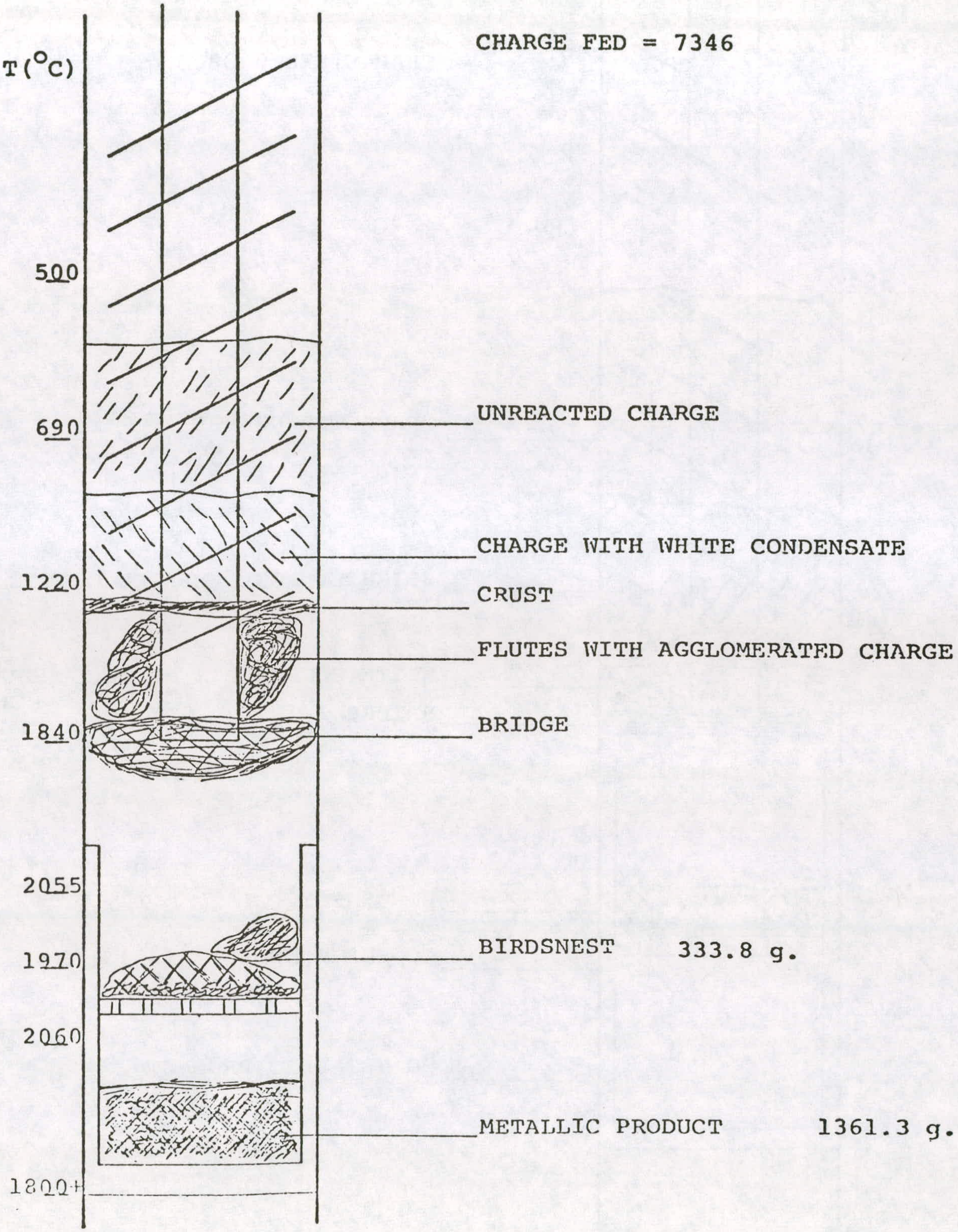


FIGURE 14

AF-24 80-5-30

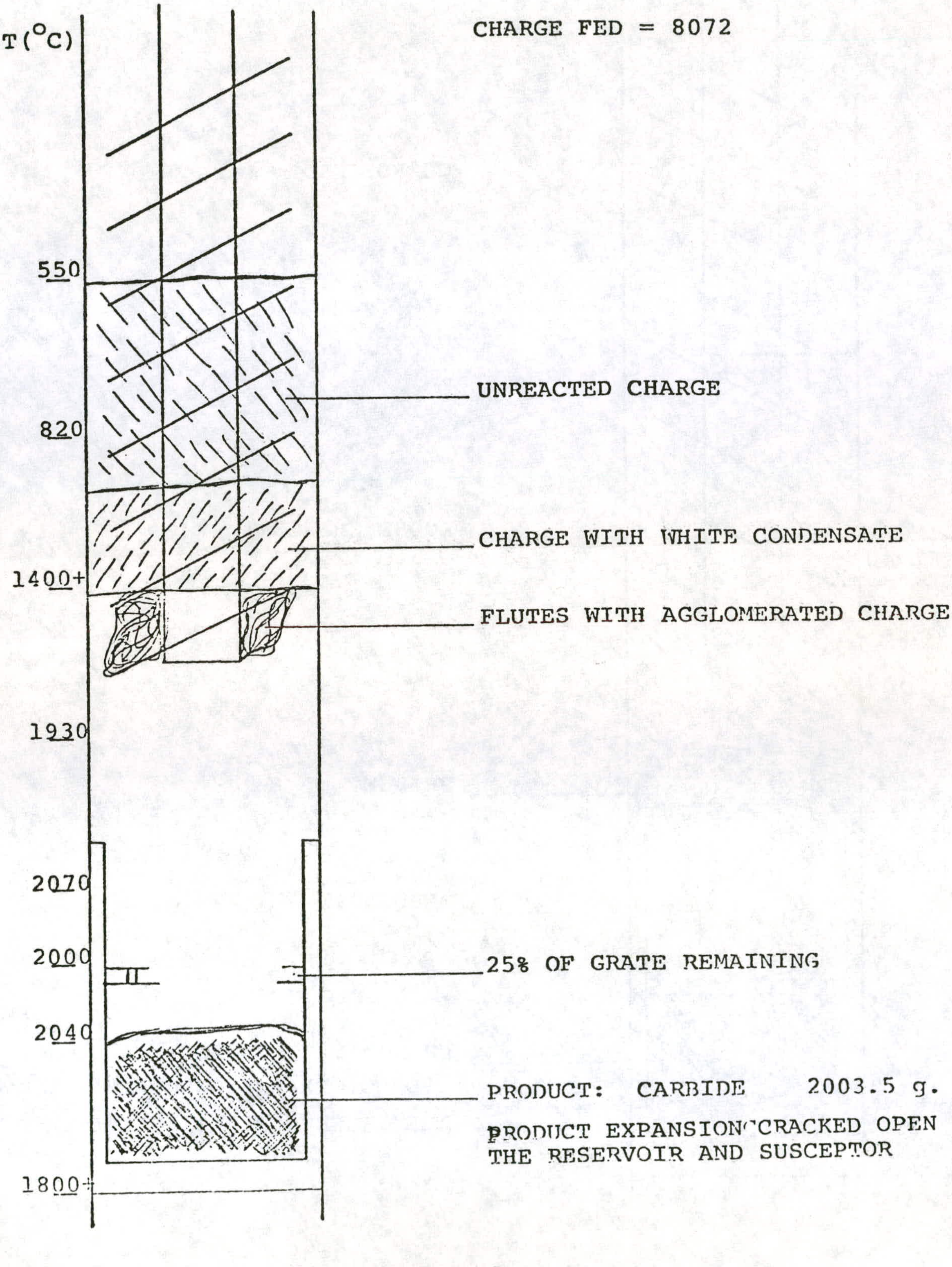
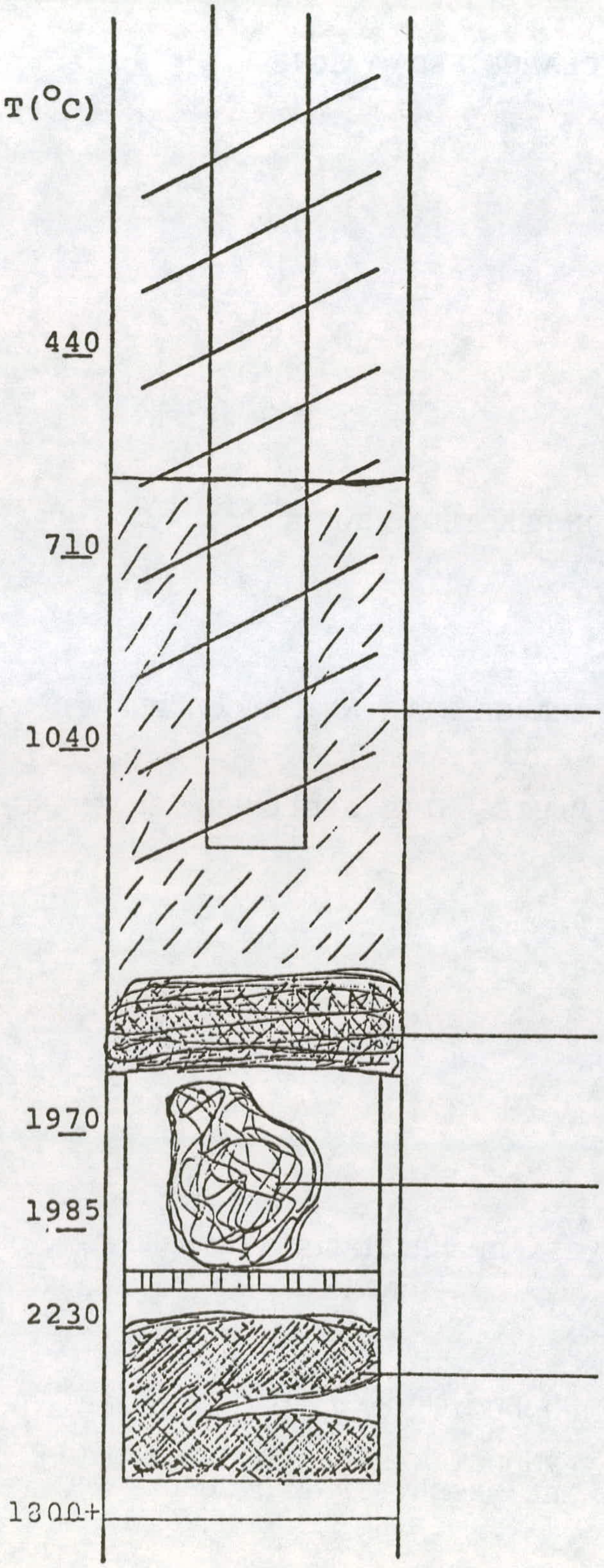


FIGURE 15

AF-25 80-6-11



CHARGE FED = 9455 g.

LOOSE CHARGE

BRIDGE

BIRDSNEST 474 g.

PRODUCT: CARBIDE 2110 g.
PRODUCT EXPANSION CRACKED OPEN
THE RESERVOIR AND SUSCEPTOR

FIGURE 16

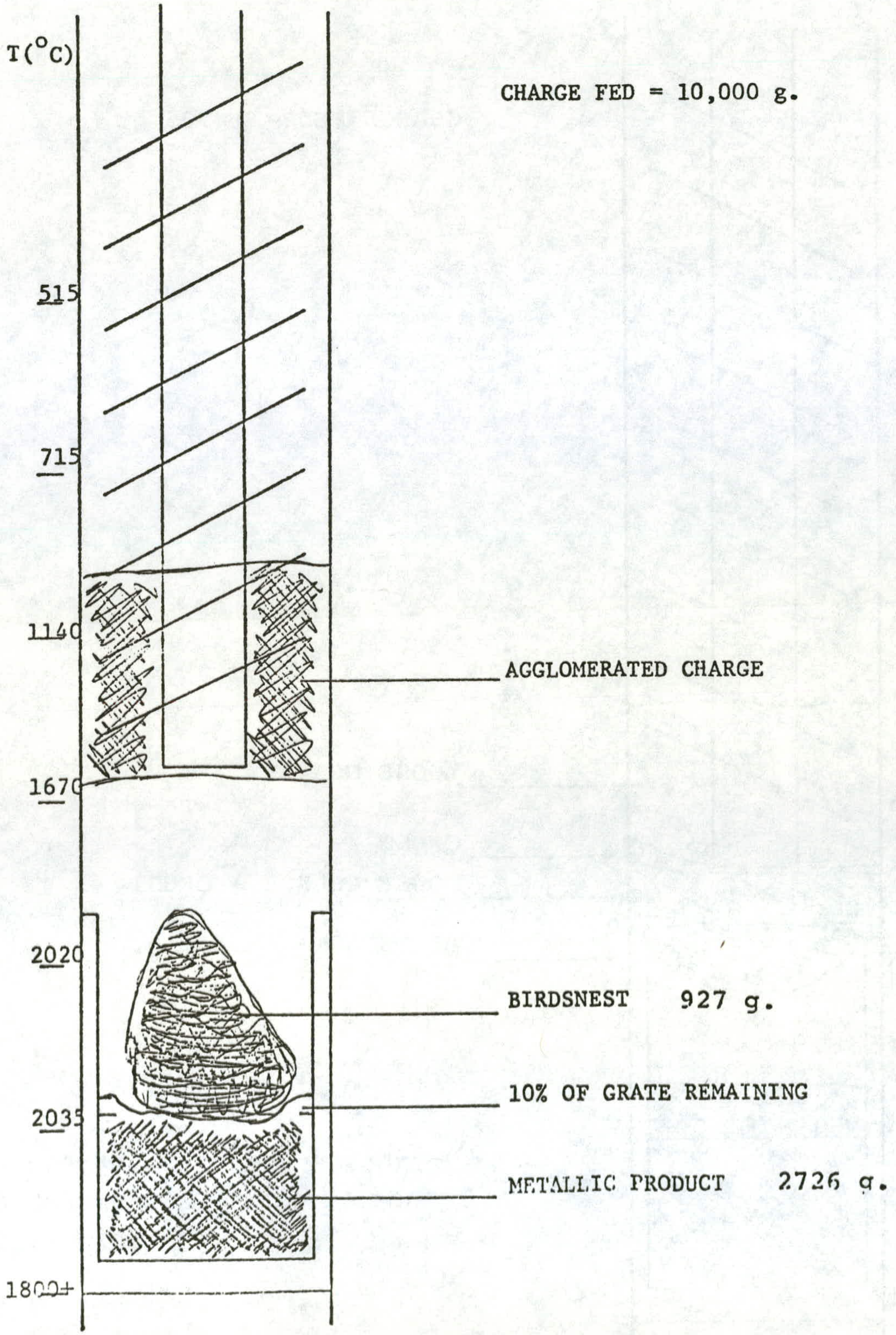

T(°C)
515
715
1140
1670
2020
2035
1800+
CHARGE FED = 10,000 g.
AGGLOMERATED CHARGE
BIRDSNEST 927 g.
10% OF GRATE REMAINING
METALLIC PRODUCT 2726 g.

FIGURE 17

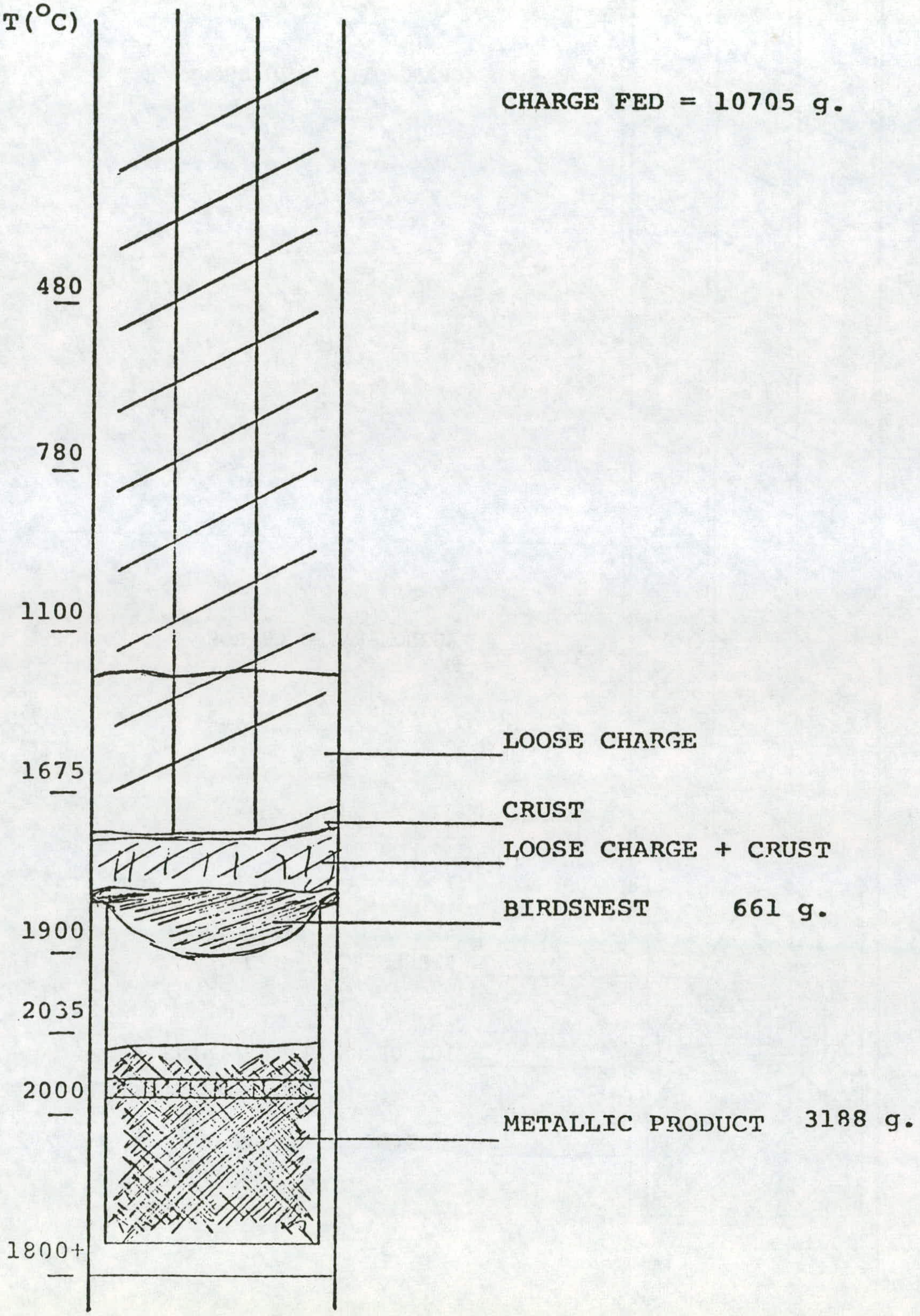

T(°C)
480
780
1100
1675
1900
2035
2000
1800+
CHARGE FED = 10705 g.
LOOSE CHARGE
CRUST
LOOSE CHARGE + CRUST
BIRDSNEST 661 g.
METALLIC PRODUCT 3188 g.

FIGURE 18

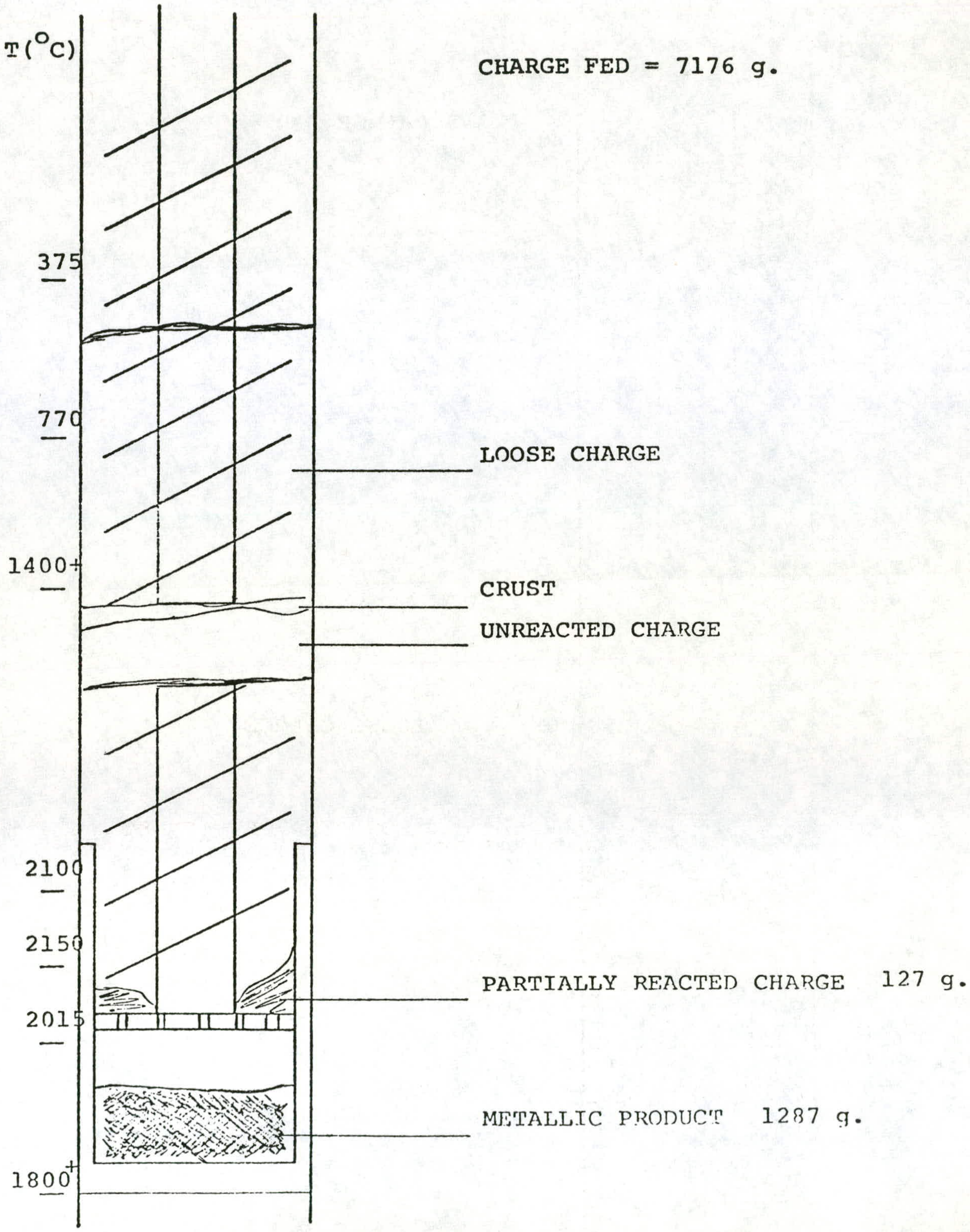


FIGURE 19

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