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OFFICE OF NAVAL RESEARCH

Contract Nonr 4381(00)

Task No. NR 356-461

Technical Report No. 15

Bench Scale Production of $C_2B_4H_8$
(nido-4,5-dicarbahexaborane-8)

Robert E. Williams, J. F. Ditter, J. D. Oakes, and F. James Gerhart

AEROJET-GENERAL CORPORATION

Space-General Plant

CENTER FOR RESEARCH AND EDUCATION
1117 West 35th Street
Los Angeles, California 90007



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26 October 1967

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BENCH SCALE PRODUCTION OF $C_2B_4H_8$ (nido-4,5-dicarbahexaborane-8)

by

Robert E. Williams, J. F. Ditter, J. D. Oakes and F. James Gerhart

INTRODUCTION

During the past two years one of our aims has been to build up a capability for preparing ample quantities of the smaller closo-carboranes. Until very recently, as noted below, the only good method of synthesizing these closo-compounds was via the nido-carborane, $C_2B_4H_8$ (prepared from pentaborane-9 and acetylene), so a prerequisite capability had to include synthesis of "bench scale" quantities of $C_2B_4H_8$. The end result has been that we can now produce over 90 grams of $C_2B_4H_8$ per five-day week, and this is accomplished by batch processing a total of about 200 grams of pentaborane-9. The reaction is carried out at 215°C in the gas phase in an excess of acetylene. Each run consumes about 40 hours reaction time, so considering the additional time required to fill, empty, and clean the reactor tanks, we are able to process two batches per week.

During initial phases of this particular task we were plagued by the fact that nearly one-half of the runs terminated by explosions (all contained in the reactor tanks), and when this occurred no $C_2B_4H_8$ was produced. Naturally, the cumulative yield from all the batches was disappointingly low. In time, however, we learned how to prevent the explosions and how to make the runs proceed smoothly. This was accomplished by (a) limiting the quantity of pentaborane-9 per run, (b) by cleaning the reactor tanks between runs, (c) by separating heaters and reactors to avoid "hot spots" and (d) by adding acetylene intermittently instead of "all at once." Needless to say, we also avoided any materials of construction or peripheral equipment which contained

silver, mercury, or high percentages of copper which could form explosive acetylides and thus initiate gas phase explosions.

Following these remedial actions and after about one year of "explosionless" operation, we considered scaling up our capability six to eighteenfold by using 300-liter reactor tanks instead of the present 34-liter units, and in line with this plan we had a preliminary engineering and cost analysis conducted (see Appendix A). Included in the future plans was a simple automatic acetylene-feed system, utilizing a pressure demand valve in place of intermittent manual injection. Also to be included was a method of rapid renovation of reactor tanks by installing an "in situ" steam cleaning system so that we could process three batches per week instead of two.

Our original plan was to issue a Technical Report covering the operation and yields obtained from the much larger and presumably more efficient equipment. Very recently, however, we have discovered a new method of synthesizing the smaller closo-carboranes, which suddenly eliminates the need for larger amounts of their erstwhile precursor, $C_2B_4H_8$. This new method will be in a subsequent Technical Report and is not to be confused with our recent ONR Technical Report #13, which details the virtually quantitative synthesis of $C_2B_3H_5$ (~ 40%), $C_2B_4H_6$ (~ 20%), and $C_2B_5H_7$ (~ 40%) from $C_2B_4H_8$. We are of course not dismantling our present 136-liter processing equipment (four 34-liter tanks) because $C_2B_4H_8$ remains of synthetic importance and of interest to numerous investigators, and perhaps at a later date, it may come into consideration again for scaled up synthesis. However, inasmuch as there is no likelihood of our undertaking this task in the near future, this technical report essentially details the present "state of the art."

BACKGROUND

Weiss^{1,2} synthesized the first nido-(nest) carborane and determined its empirical formula to be $C_2B_4H_8$. Good¹ then isolated a sufficient quantity for structural analysis, and Williams¹ deduced its structure from

the ¹¹B n.m.r. spectrum and its similarity to B₆H₁₀. Onak^{1,3} later improved the synthesis and raised the yield to useful proportions. We then used Onak's best data³ as the basis for scaling up the synthesis by over two orders of magnitude, using four 34-liter stainless steel tanks in place of one-liter glass bulbs.

EQUIPMENT DESCRIPTION

Equipment, primarily purchased as military surplus, was assembled as detailed in figure 1. All parts were of stainless steel and materials which contained high concentrations of copper, silver or mercury were assiduously avoided, since they can induce formation of acetylides, which in turn might initiate gas phase explosions. Commercial grade acetylene was utilized, and provision was made for removal of the acetone. This was done by passing the acetone laden acetylene through pre-dried, activated charcoal (Barnebey Cheney KE). The charcoal as purchased contains ~ 3% water which is easily removed before use by applying vacuum while the tank containing the charcoal is heated. Routine regeneration (removal of acetone) from the charcoal is accomplished in the same fashion. The temperature of each reactor tank could be determined by a thermocouple inside the tank, and temperature control was effected by standard crude Robertshaw "home-oven" type controllers, pre-calibrated with the thermocouples. Explosions above 240° and low reaction rates below 210° necessitated a fair degree of control.

Four 250W (at 110V) strip heaters were operated at 55V, and these provided sufficient heating capability. An aluminum sheath separated the strip heaters and the tanks to prevent direct contact of heater and reactor and the consequent hot spots, which were found to induce explosions.

EXPERIMENTAL RESULTS

A typical successful preparation proceeded as follows: B₅H₉ was allowed to expand into the four previously evacuated tanks to its vapor

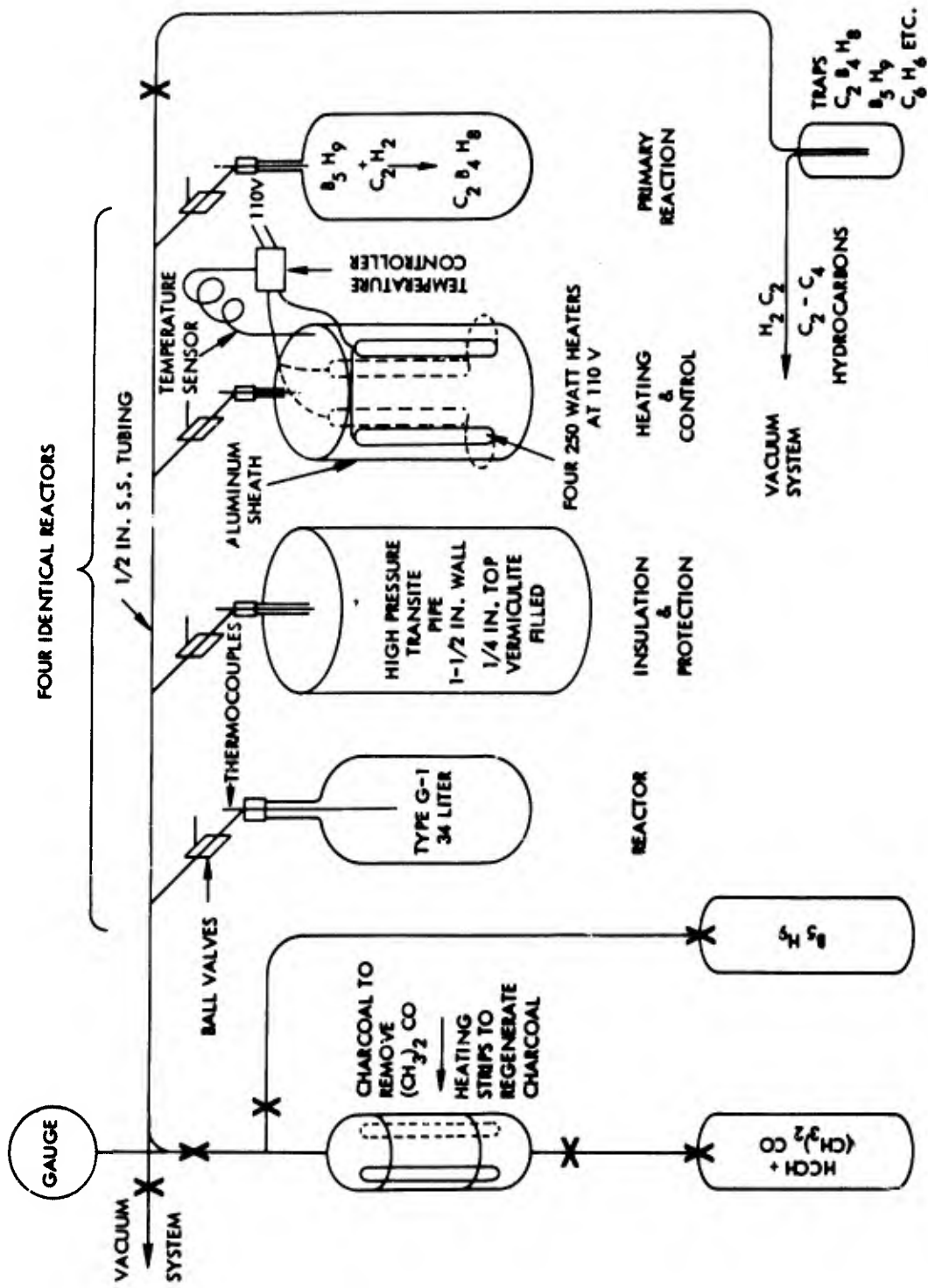


Figure 1. Details of Assembled Equipment

pressure of 150-200 mm. (Determined by the ambient outside temperature), and acetylene was then added to bring the pressure to about 20 psia. The heaters were turned on and the reactor tanks brought up to 225°C (internal temperature). After 18 hours of heating the pressure had declined, and more acetylene was added to bring the total pressure to 30 psia. Subsequently, 6 hours later, more acetylene was added to bring the pressure once again back to 30 psia. Heating overnight then resulted in a final pressure on the order of 18 psia. Fractionation of products through a -117°C trap removed the $C_2B_4H_8$ and by-product benzene as well as small amounts of the $CH_3CB_5H_8$ isomers and allowed the hydrogen and unreacted acetylene to be pumped off. Our best yields of $C_2B_4H_8$ have approached 40%.

DISCUSSION

The initial problem of explosions was the only major difficulty encountered in the scaled up synthesis of $C_2B_4H_8$. Continued use of the same reactor tank without cleaning was found to give considerably decreased yields of product, even if explosion did not occur, and eventually, moreover, continued re-use would always lead to an explosion. To eliminate this problem it was necessary to clean the tanks after each reaction to remove the solid, nonvolatile materials accumulated on the walls of the tank. These solids either initiated explosions catalytically or else absorbed excessive amounts of pentaborane-9 during filling at room temperature. In any event the ceramic-like material which forms on the walls as a by-product of the reaction could until recently only be removed by the abrasive action of sand and water while the four tanks were being rotated or rolled on a ball mill type apparatus which turned at 20 rpm. By filling the tanks half way with sand and then adding water the majority of this solid debris could be removed within a few hours, and it could be completely removed if the tanks were allowed to rotate overnight. This procedure has now been replaced by cleaning with steam introduced at 100 psi for 20-60 minutes. Moreover, the steam cleaning procedure made possible the considered scale-up to 300-liter vessels. The specter of having to load four 300-liter tanks with sand was not a pleasant one to contemplate.

One tank was lined with teflon to assess whether such lining would facilitate water cleaning following reaction or function undesirably with B_5H_9 as a fuel-oxidizer mixture during reaction. The teflon coating did facilitate cleaning to a minor extent, but is not considered worth the additional cost.

Cleanliness cannot be over emphasized. We have tried doubling the initial B_5H_9 charge and obtained about the same quantity of products as realized from a normal run. Perhaps the "by-product junk" accumulates roughly twice as fast and acts to reduce the overall yield proportionately. The yield roughly approximates the yield obtained when the tank is used twice with normal amounts of reagents. One is almost forced to conclude that the highest possible yields would be realized only if a continuously "clean" system could be constructed. Such systems may be imagined, but none appear to be practical.

As to the problem of obtaining reduced yields by adding acetylene all at once, we found that by maintaining the acetylene at as low a level as possible during reaction the yields could be increased. This was accomplished by injecting the acetylene intermittently instead of in a single initial charge, suggesting that a pressure demand system which could continuously add acetylene and could maintain the reactors at about atmospheric pressure should be close to optimal. Such a control system had been slated for the 300-liter reactors. We intend to add it to our currently used smaller system in the near future.

REFERENCES

1. Onak, T.P., Williams, R.E., Weiss, H.G., J. Am. Chem. Soc. 84, 2830 (1962).
2. Weiss, H.G., Shapiro, Isadore, Patent No. 3086996 (1963).
3. Onak, T.P., Drake, R.P., Dunks, G.B., Inorg. Chem. 3, 1686 (1964).

APPENDIX A

DISCUSSION OF ENGINEERING AND COST ANALYSIS

A few comments are in order concerning the following preliminary engineering and cost analysis. Dr. Milne, who made the analysis, proposes that it would be more practical and less expensive simply to add more 34-liter reactor units than to supplant them with 300-liter tanks. The primary reason pertained to the difficulty and expense of cleaning the larger tanks. Subsequent to his survey, however, we have demonstrated the practicability of "in situ" steam cleaning, so the use of the larger tanks with "built in" capability for steam cleaning appears more promising.

Also, Dr. Milne was unaware that increasing the initial charge of pentaborane-9 by any great extent might lead to explosions.

The suggested mixing fan is open to question. So far as we know the 5-10°C improvement that would be effected in temperature homogeneity is probably not worth the added complexity of a fan. Getting vacuum tight seals around the rotor shaft, particularly at the relatively high temperature of 215°C is not a simple matter.

Although this preliminary engineering and cost analysis will not be acted upon and is partially obsolete in the light of recent knowledge, it should serve as a basis upon which a more refined analysis could be made when and if a greater demand for $C_2B_4H_8$ ever arises. Updating it at this time for this technical report seems superfluous.

SPACE-GENERAL CORPORATION

INTEROFFICE CORRESPONDENCE

TO: R. E. Williams

CC:

DATE: July 10, 1967

SUBJECT: Carborane Production Facilities

FROM: John R. Milne

This summarizes the results of a preliminary design study, as you requested, for increasing the production capabilities at CRE for carboranes. Present capacity is limited by two factors: reactor space and work schedules. Certain problems would arise in changing either or both.

Increasing the reactor space available by using one 295-liter vessel instead of the four 35-liter vessels could increase capacity by a factor of only a little over 2, at most. But this would require a capital investment of more than twice that of the present installation. In order to use this approach, a new method for cleaning scale from the vessel would have to be developed. Also, a larger-sized vessel would be inherently more subject to failure because of attendant uncertainties in reaction rates. Still another disadvantage would be that the expected operating costs would be higher due to the cleaning problem. All of these factors indicate that increased reactor space can be obtained more economically by increasing the number of 35-liter reactor-ovens in use.

Rescheduling men and equipment could result in another factor of two increase in production capacity. Because rescheduling manpower for Saturday and Sunday operations is presumed to be impracticable, equipment rescheduling with automatic control of acetylene addition was considered. Duplication of the present schedule of acetylene additions would require a timer-operated solenoid valve in addition to a suitable pressure control. Such a system would be feasible at reasonable cost. However, the modulation of pressure by means of a solenoid valve probably would be superfluous. That is, simple pressure control should work equally well or better without modulation; its usefulness could be determined quickly by trial. A more sophisticated, preprogrammed control system for the addition of acetylene also was considered. Whether or not the cost of such a controller could be justified depends on (1) the expected value of experimentation conducted under special schedules of pressure control, and (2) whether an adequate step-wise system of pressure control can be achieved with the timer, solenoid valves, and simple pressure regulators.

It is thus apparent that a factor of two or more increase in production capacity could be achieved at essentially the same unit of costs. The limit on capacity is set essentially by product-handling capability and manpower availability for operation, cleaning, and maintenance when reaction space and scheduling are optimized. Reduction of unit costs might be achieved by changing process conditions; for instance, by increasing the pentaborane initially introduced into the system or by developing a continuous flow process.

To: R. E. Williams

(2)

From: J. R. Milne

Recommendations

On the basis of the study made, I recommend:

1. Installing a pressure control system for the addition of acetylene, as shown in Figure 1*, using a simple pressure-regulator instead of the cam-programmed controller shown;
2. Scheduling of production operations as shown in Schedule B;
3. Studying the feasibility of process changes as follows:
 - (a) Use of more pentaborane in the initial charge,
 - (b) Shortening reaction time sufficiently to get three batches per week,
 - (c) Variation in schedule of acetylene additions, and
 - (d) Continuous flow reaction; and
4. Studying improved methods for cleaning reaction vessels.

Costs

The estimated costs of the system studied are listed in Tables 1*, 2, and 3 and are summarized here:

(1)	Large reactor-oven	\$1,768
(2)	Cleaning setup for same	1,205
	Totals (1) and (2) ...	<u>\$2,973</u>
(3)	Simple acetylene control system	389
(4)	Programmable acetylene control system	1,037
	Totals (1), (2), and (3)	<u>3,362</u>
	Totals (1), (2), and (4)	<u>\$4,010</u>

While these figures are not exact, they should provide an adequate basis for planning. The entire problem of cleaning, however, adds an undesirable uncertainty to the total cost of a large reactor. No action towards building such a system should be undertaken before this problem is resolved.

* Figures and Tables within this Appendix are referred to as Figures A-1 through A-7 and Tables A-1, A-2, and A-3.

To: R. E. Williams

(3)

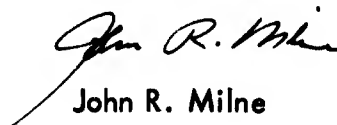
From: J. R. Milne

Sketches

Most of the attached sketches are reasonably self-explanatory. Figure 1 shows an oven-reactor system, using a 295-liter vessel. Surrounding the vessel is a radiation shield which supports heaters on the outside and is supported by a large transite duct. The temperature probe should be shielded to avoid large (as much as 50°C) errors in estimation of gas temperature. A fan at the top circulates hot air within the oven-reactor system. While the fan shown within the reactor itself is optional, I believe it could help markedly in improving both yields and over-all rates. An alternative mode of construction for the hot-air circulating system which might be considered, is that shown in Figure 3. A plan view of the reactor showing the arrangement of strip heaters is shown in Figure 4 and a plan view of the motor and blower support is shown in Figure 5.

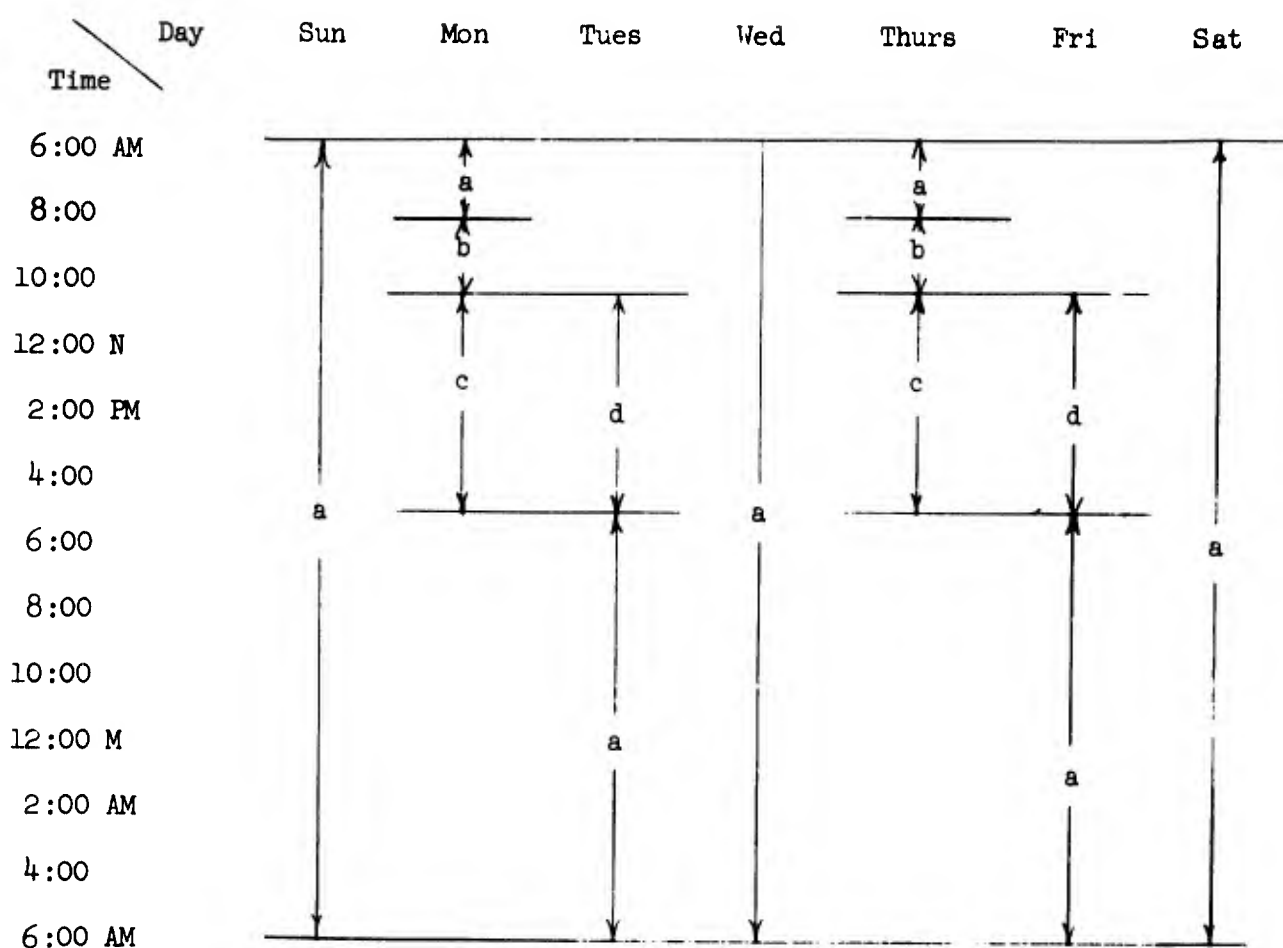
The wiring circuit for the reactor heaters is shown in Figure 6. It is important that the three heater circuits be well distributed spatially because the heater control switches only two of the three.

Figure 7 shows an arrangement for a cleaning system which would use hot soda ash (sodium carbonate) solution or a proprietary cleaning agent (such as Oakite) pumped at about 300 psi through a nozzle. While some of its drawbacks are obvious, this system could be made to work. The alternative of using several reactors, hoisting them in and out of the oven for cleaning, would be less feasible.


John R. Milne

JRM:bb
Enclosures

SCHEDULE A



- Key:
- a. Operate reactors, shutoff C_2H_2 when finished - 40 hrs, min.
 - b. Remove products, shutoff heat, flush with nitrogen - 20 hrs.
 - c. Disconnect, clean, and dry - $6\frac{1}{2}$ hrs.
 - d. Make connections, test for leaks, test safety devices, evacuate to 1 mm, add pentaborane, heat, and turn on C_2H_2 - $6\frac{1}{2}$ hrs.

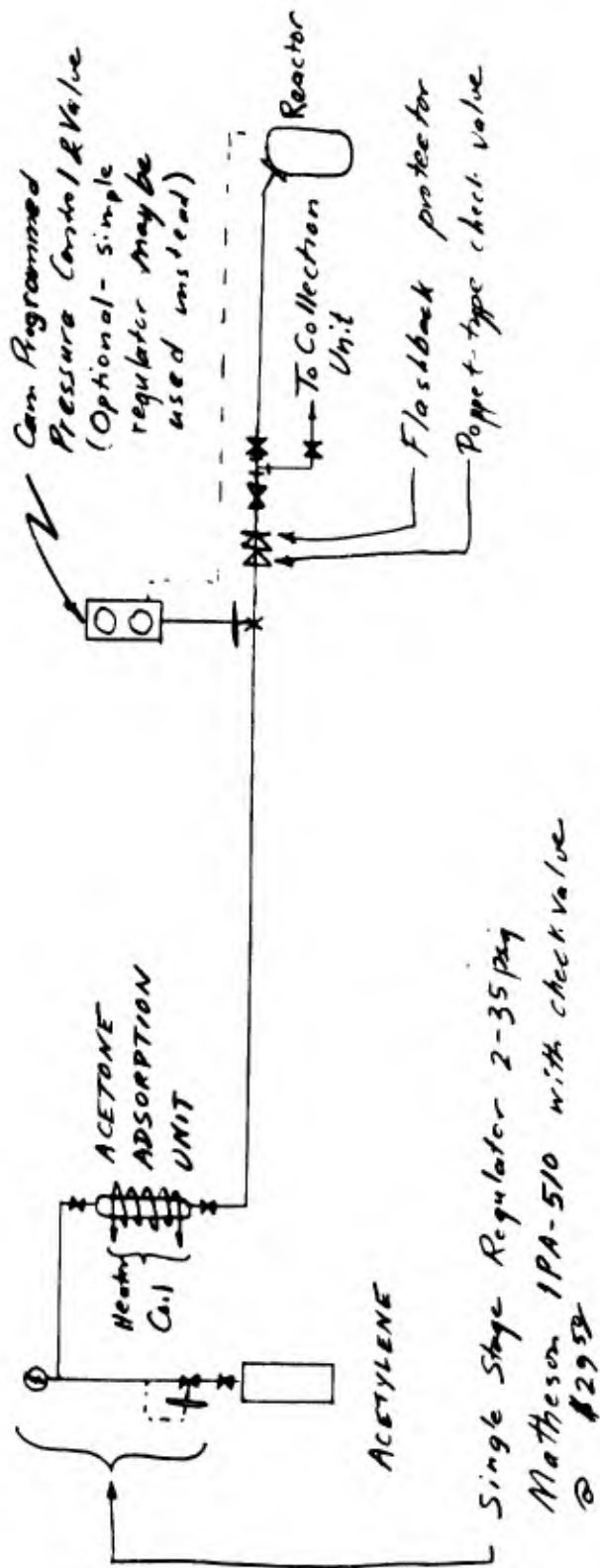


Figure A-1. Acetylene Feed System

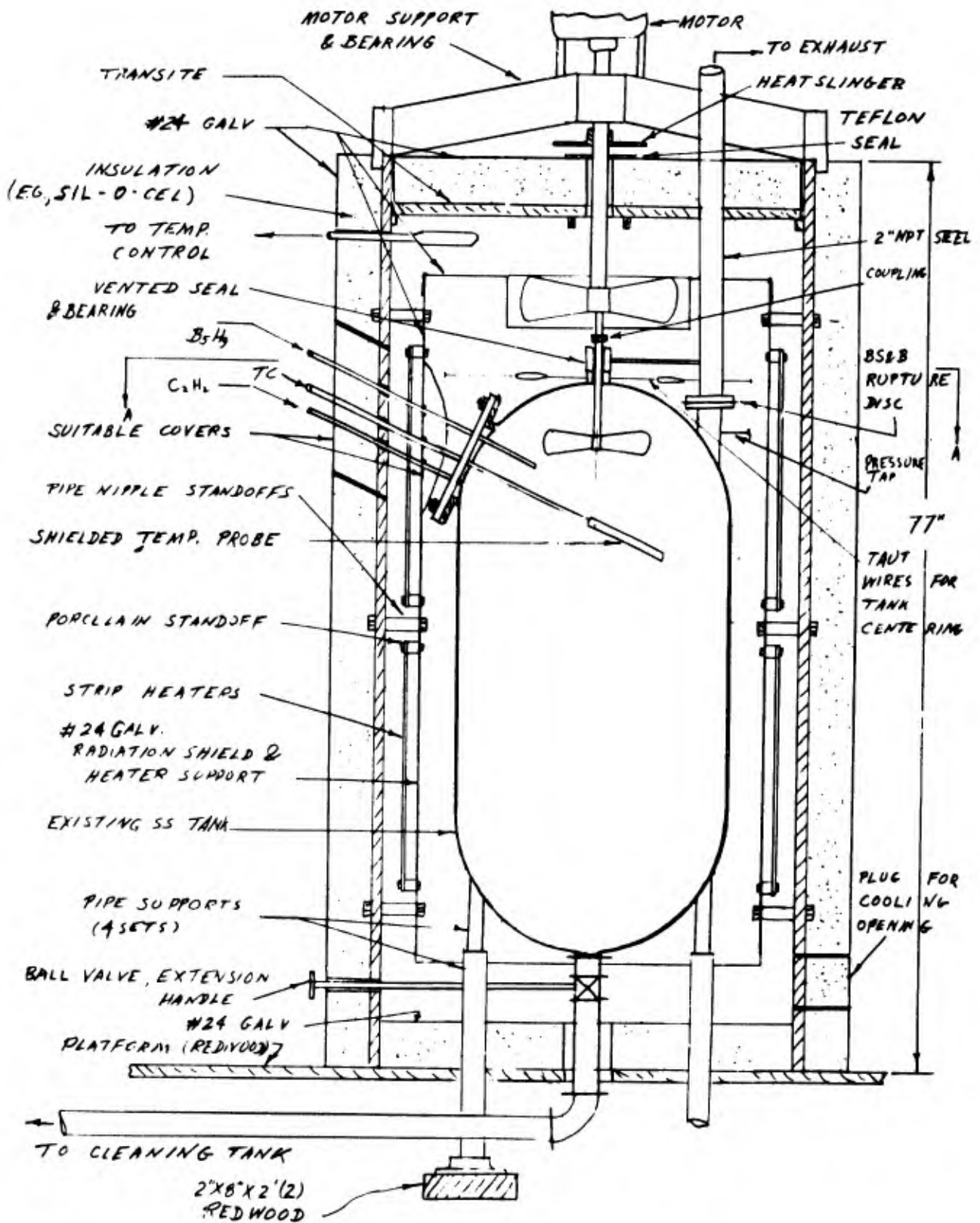


Figure A-2. Carborane Reactor Elevation

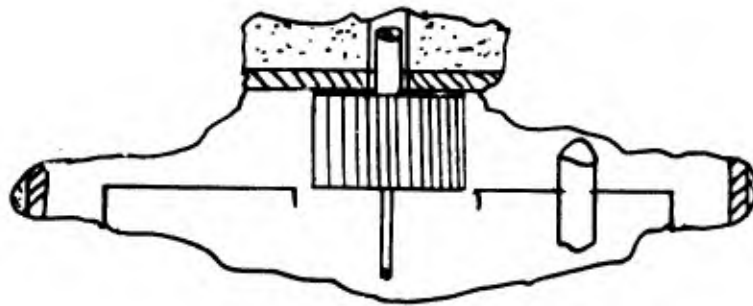


Figure A-3. Alternate Construction, Using Centrifugal Blower Instead of Fan

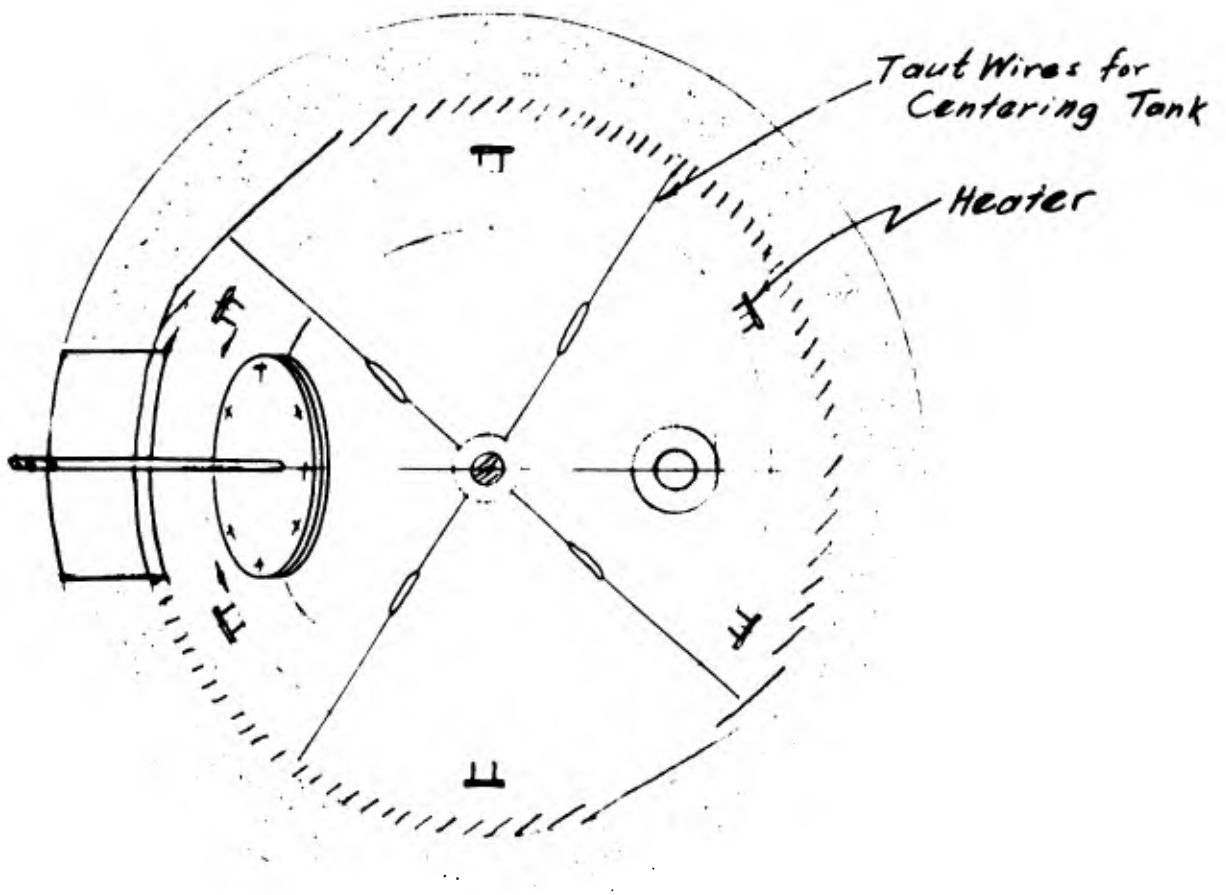


Figure A-4. Carborane Reactor, Plan Section A-A

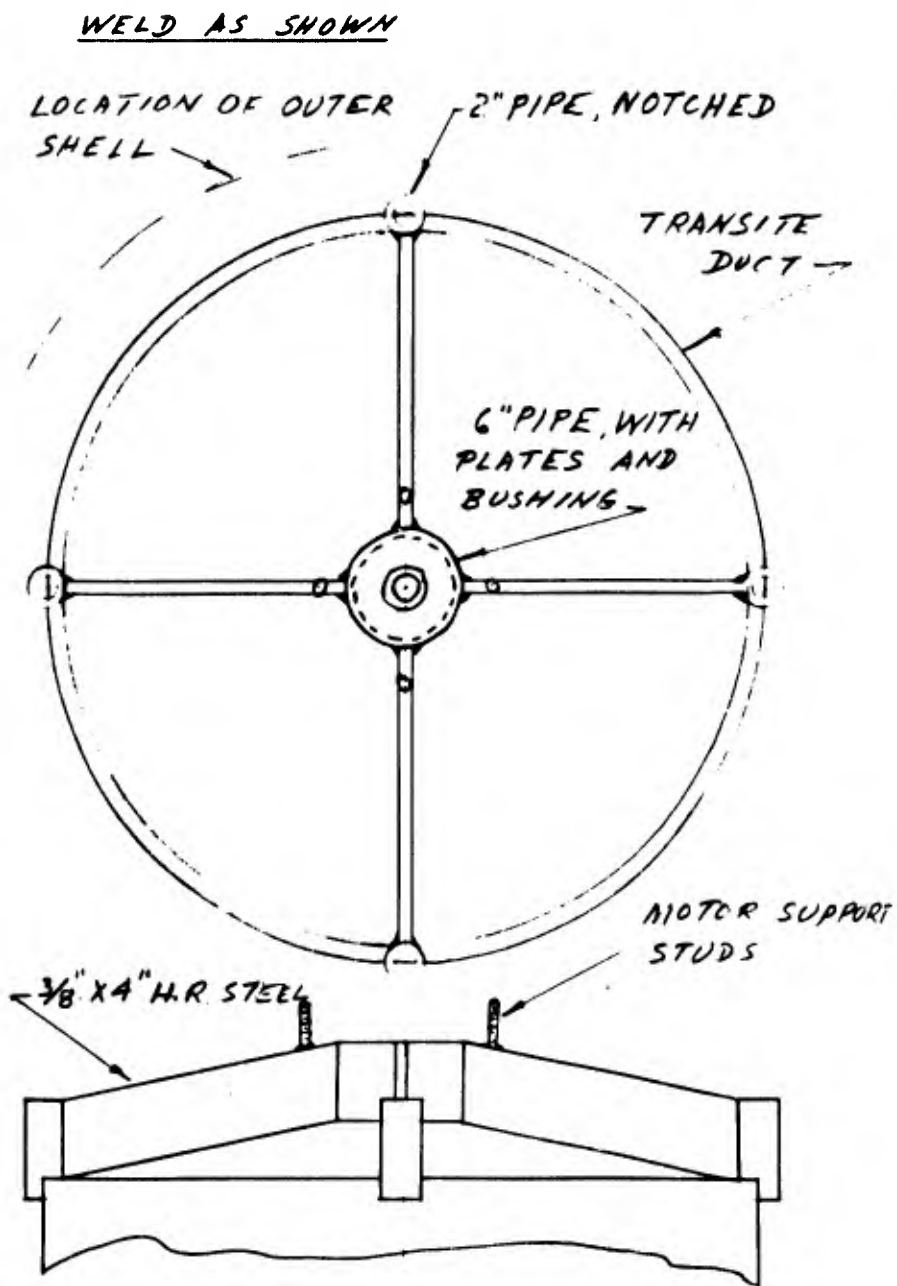


Figure A-5. Motor and Blower Support

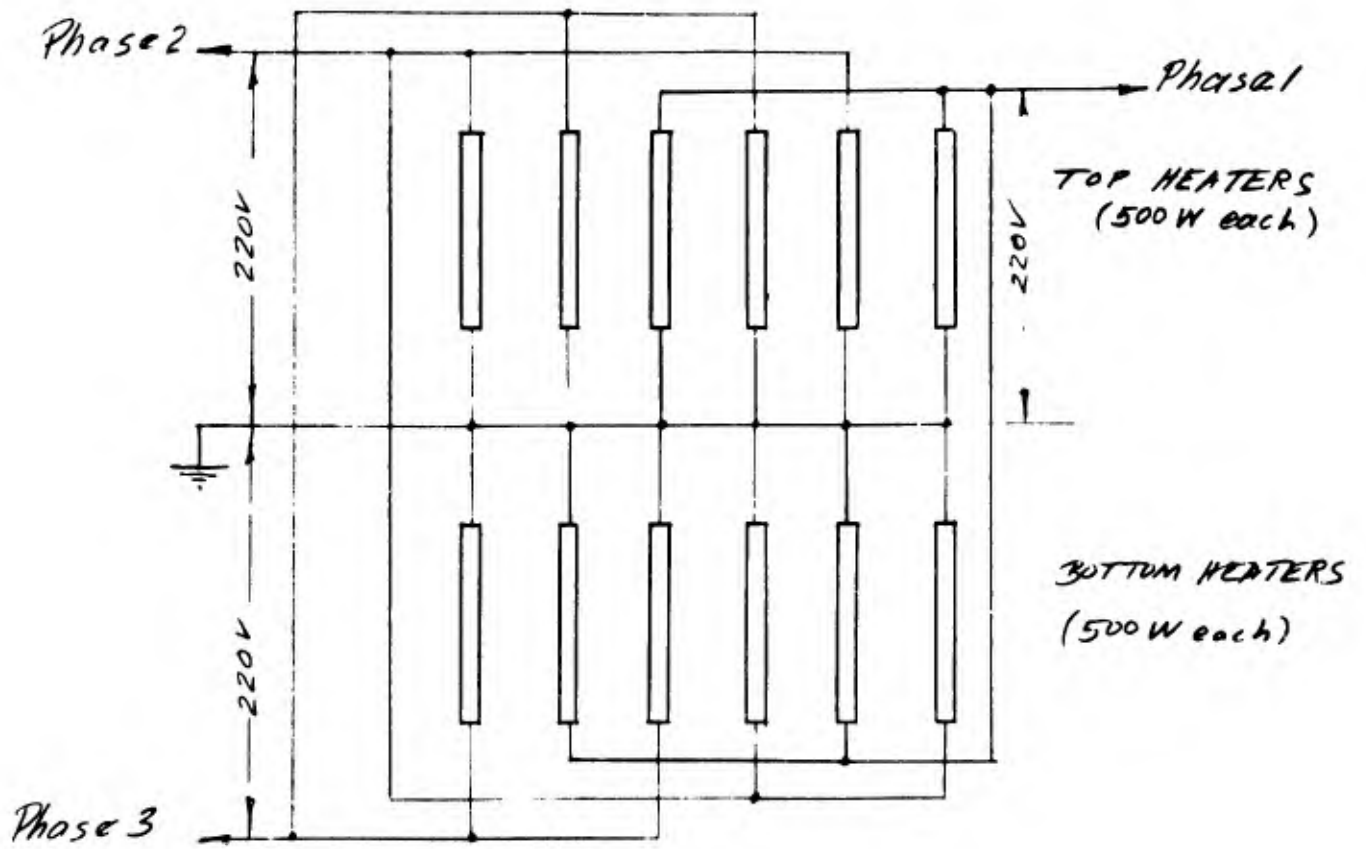


Figure A-6. Reactor Heater Wiring Circuit

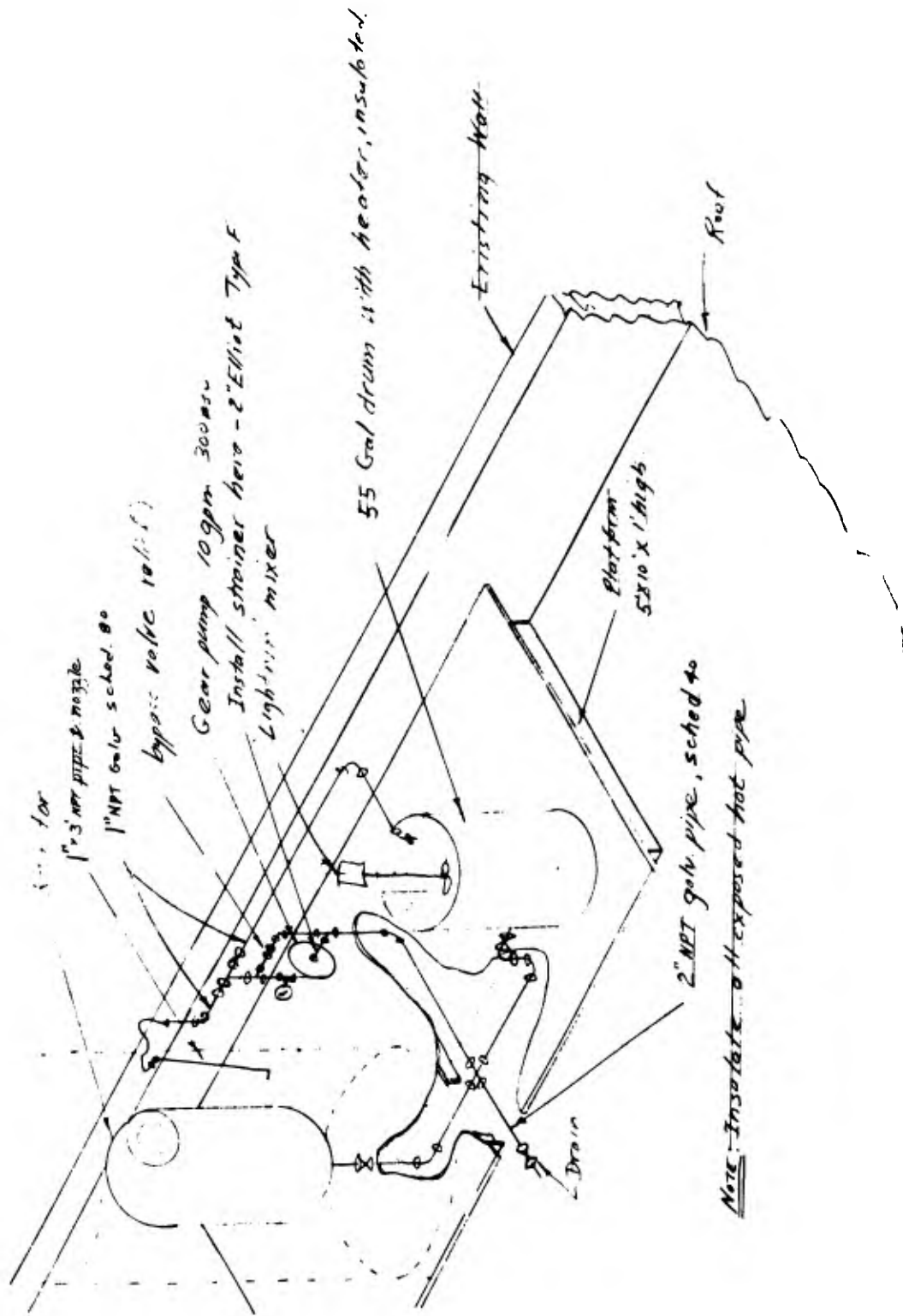


Figure A-7. Reactor Cleaning Setup

Table A-1

Reactor & Oven	Mfg.	Model	Specs	No. Regd	Est. Cost	Net	Net + *
					\$	\$	15%
1. Platform, 100 bd. ft. 4 man hours			Redwood	1	\$ 20 16	\$	\$
2. Reactor		On hand	On hand				
3. Access port flange	Ladish		8"	1	89		
4. Blind companion flange	Ladish		8"	1	97		
5. Shielded thermowell				1	25		
6. Pipe supports				4			
7. Transit duct, 36"	Johns		34" wall	1	158		
8. Insulation, sil-o-cel	J-M		Coarse	14 bags	69		
9. Valve handle extension				1	3		
10. Galve. sheet iron			#24	200 ft. ²	60		
11. Rupture disc. assembly	BS&B		10psi	1	50		
12. Vented seal				1	50		
13. Temp. controller	Robertshaw	SE5050-001	200°-550°F	1	25		
14. Thermocouple & temp. rec.			On hand				
15. Heaters, 500 watt, 220V	Acrawatt	TEEM	1½" x 23"	12	50		
16. Blower & motor					350		

Table A-1 (Continued)

Reactor & Oven	Mfg.	Model	Specs	No. Regd.	Est. Cost	Net	Net + * 15%
17. Blower & motor support					\$ 15	\$	\$
18. Hardware, misc.					20		
19. Labor:							
Welding 4 hrs					40		
Sheet metal 16 hrs					96		
Electrical 16 hrs					144		
Other 40 hrs					160		
Total						\$ 1537	\$ 1768

* 15% for taxes, freight, and contingencies

Table A-2

Cleaning Setup	Mfg.	Model	Specs	No. Regd.	Est. Cost	Net	Net + * 15%
1. 55 Gallon drum				1	\$ 25	\$	\$
2. Lightning Mixer with motor	Mixing Equip. Co.			1	280		
3. Drum Heater, SS	Wiegand	KTFLS-312	12KW	1	132		
4. Contactor for heater	Teton	MC330	3 pole 30A	1	24		

Table A-2 (Continued)

Cleaning Setup	Mfg.	Model	Specs	No. Reg'd.	Est. Cost	Net	Net + * 15%
5. Thermostat for heater			70-500°F	1	\$ 25	\$	\$
6. Pipe, Galv. 2" sched. 40				10 ft	40		
1" sched. 80				10 ft	16		
Insulation					50		
7. Hose 1" x 5 ft.				1	5		
8. Gear pump with bypass	Roper	F	300psi (2HP) N10gpm	1	150		
9. Fittings					52		
10. Valves, gate			2" CI	3	36		
Valves, gate			1" steel	1	6		
Valves, globe			1" steel	1	6		
11. Strainer	Elliott	Type F	2"	1	87		
12. Plumbing & wiring					114		
Total						\$ 1048	\$ 1205

* 15% for taxes, freight, and contingencies

Table A-3

Acetylene Control	Mfg.	Model	Specs	No. Regd.	Est. Cost	Net	Net + * 15%
1. Regulator, Single stage	Matheson	1PA-510	2-35psig	1	\$ 30	\$	\$
2. Valve, Teflon seat, packless	Dahl	GT/303 + P2 + BJ + R15	SS 1/8" NPT	1	37		
3. Time schedule controller	Taylor	147RA136	7 day	1	649		
4. Valves	Hoke			5	50		
5. Acetone adsorber				1	50		
6. Flashback protector				1	10		
7. Check valve	Circle Seal			1	5		
8. Plumbing & wiring					50		
9. Indicating pressure controller	Taylor	395RF1011	0-30psia	1	85		
10. Air filter	Taylor	89517		1	9		
11. Air regulator	Taylor	415323		1	12		
Total, Programmed Control (Omitting Item 9)						\$ 902	\$ 1037
Total, Nonprogrammed Control (Omitting Item 3)						\$ 338	\$ 389

* 15% for taxes, freight, and contingencies

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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Chemistry Branch, Office of Naval Research	
13. ABSTRACT The production of $C_2B_4H_8$ (nido-4,5-dicarbahehexaborane-8) has been scaled up to approximately 90 grams per week utilizing about 200 grams of B_5H_9 and excess acetylene.			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
carborane						
<u>nido</u> -carborane						
dihydrocarborane						
<u>nido</u> -4,5-dicarbahexaborane-8						
<u>stapho</u> -2,3-dicarbahexaborane-8						
pentaborane-9						
acetylene						