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FINAL REPORT

MICROCONTACTOR UTILIZING HIGH-DENSITY, METALLIC SUPEROXIDES

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ABSTRACT

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A laboratory prototype microcontactor that used high-density, metallic superoxides in revitalizing a sealed cabin atmosphere was designed, fabricated, and tested at General Dynamics/Electric Boat. A microcontactor is a device for producing finely divided superoxide particles by grinding a block of high-density material, and then providing intimate contact between the finely divided superoxide particles and a dynamic air stream.

Preliminary laboratory tests were made to determine the grinding characteristics of high-density KO_2 (115 lb/ft^3), such as grinding rates, product size distribution, power requirements as functions of grinder speed, and force of KO_2 against the cutting tool. Semi-quantitative tests were made to investigate KO_2 reaction rates as functions of time, moisture concentration, and particle size.

Based on the preliminary laboratory tests, a one-man capacity microcontactor was designed and fabricated. Test runs were made with the microcontactor under various inlet conditions of moisture and CO_2. Test results showed that the microcontactor concept, using potassium superoxide, and probably other superoxides, for air revitalization is feasible. No over-production of oxygen was encountered, as is commonly the case when using canisters of the superoxide. The microcontactor matched respiratory quotients (R.Q.) between 0.6 and 1.1 required by man. Steady-state conditions with respect to O_2 and CO_2 concentrations were obtained after only a few minutes of operation.

Author

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SECTION ONE

INTRODUCTION

This publication reports on the work performed under Contract NASw-551, which encompassed the design, fabrication, and testing of a laboratory microcontactor which uses high-density, metallic superoxides to revitalize a sealed cabin atmosphere. The device provides intimate contact between finely divided superoxide particles and a dynamic air stream. Its primary components are: 1) a grinder for producing the finely divided particles from a high-density block of superoxide, and 2) a reaction-filtration zone (see Figure 5-5).

The following advantages (as compared with a granular bed) result from this approach:

1. Storage volume for the superoxide chemical is reduced by a factor of about 3.
2. Chemical utilization is practically complete because of intimate contact of a small quantity of small, particle-size chemical with a relatively large volume of air.
3. Control of O_2 and CO_2 in the cabin air is improved by supplying freshly ground superoxide as needed and by controlling humidity level in the reactor zone.
4. A lower pressure drop results because of the small quantity of chemical exposed at one time.

Air revitalization in a manned space vehicle requires that carbon dioxide and water vapor produced by man be removed and the oxygen consumed by man be replenished. Metallic superoxides can aid in fulfilling these requirements. These chemicals react with water vapor in the air to release oxygen. The metallic hydroxide reaction product then combines with carbon dioxide to produce the metallic carbonate and/or bicarbonate. Trade-off studies indicate that the metallic superoxide approach to air revitalization is practical up to approximately 30 man-days in flight. Beyond this point, the resulting weight penalty favors a regenerative system. For these longer missions, a superoxide system can be used for emergency back-up.

SECTION TWO

REVIEW OF METALLIC SUPEROXIDES FOR AIR REVITALIZATION

2.1 GENERAL

At present, there is considerable interest in metallic superoxides due to the high potential that these compounds have as possible air revitalization materials. In the past, interest was mainly directed toward an attempt to understand the behavior of these "abnormal oxides." Petrocelli and Kraus (1963) made a review of the work done to date on the superoxides and ozonides of various inorganic materials. Table 2-1 shows the characteristics of four metallic superoxides which have potential as O_2 supply/CO_2 removal chemicals.

The major portion of the research and development work to date has been performed using potassium superoxide (KO_2). This is due to its availability, low cost, and proven application as an emergency oxygen source. For the same reasons, the scope of the work performed under the present contract was limited to the application of high-density potassium superoxide.

Potassium superoxide, KO_2, has been commercially available for about 30 years as an oxygen source and has been used for emergency breathing apparatus by industry, fire departments, and the military. The superoxide is manufactured by spraying molten potassium into dry air, forming a yellow "fluff" which is later compressed into cakes under 1800 psi pressure. The cakes are then crushed to the desired particle size. The canary yellow solid is commercially produced in the 2-4 mesh size with a bulk density of about 41 lb/ft^3. During manufacture, there is some contamination with K_2CO_3 and KOH by reaction with traces of CO_2 and water vapor in the air. The product, as packed in commercial canisters, contains less than 2% total of these two impurities. The guaranteed minimum available oxygen of this material as stated by the vendor is 32% by weight (total theoretical available oxygen = 236 standard cc per gram KO_2 in reaction with H_2O, or 34% by weight). The minimum guaranteed purity is, therefore, about 94%.

TABLE 2-1

CHARACTERISTICS OF VARIOUS METALLIC SUPEROXIDES (PETROCELLI AND KRAUS)

Compound	Formula	$\frac{\text{Lb } O_2}{\text{Lb Compound}}$	$\frac{\text{Lb Base Generated}}{\text{Lb Compound}}$	Remarks
Lithium Superoxide	LiO_2	0.61	0.62	1
Sodium Superoxide	NaO_2	0.43	0.73	2
Potassium Superoxide	KO_2	0.34	0.79	3
Calcium Superoxide	$Ca(O_2)_2$	0.46	0.71	4

Remarks

1. Unsubstantiated Russian claims for its preparation and stabilization at room temperature.
2. Commercial route for its preparation available. Expensive.
3. Commercial route for its preparation available. Widely used.
4. Produced in low yield. Highly desirable due to high melting point of $Ca(OH)_2$.

2.2 CHEMICAL REACTIONS

The main reactions which occur with KO_2 are as follows:

1. $2KO_2 + H_2O \longrightarrow 2KOH + 3/2\ O_2$
2. $2KOH + CO_2 \longrightarrow K_2CO_3 + H_2O$
3. $KOH + CO_2 \longrightarrow KHCO_3$

The following hydrating reactions also compete with the above reactions:

4. $KOH + 3/4\ H_2O \longrightarrow KOH \cdot 3/4\ H_2O$
5. $KOH + H_2O \longrightarrow KOH \cdot H_2O$
6. $KOH + 2H_2O \longrightarrow KOH \cdot 2H_2O$
7. $2K_2CO_3 + H_2O \longrightarrow 2(K_2CO_3 \cdot 1/2\ H_2O)$
8. $2K_2CO_3 + 3H_2O \longrightarrow 2(K_2CO_3 \cdot 3/2\ H_2O)$

The human respiratory quotient (R.Q.), i.e., the volume or molar ratio of CO_2 expired to the O_2 consumed by man, is usually taken as 0.82. If reactions 1 and 2 above are the only reactions occurring, then the resulting R.Q. is 0.67, i.e., too much oxygen is produced by reaction 1 in order to produce sufficient KOH for reaction 2 to remove all the CO_2 produced, or from the other point of view, too little CO_2 is removed by reaction 2 for the amount of oxygen supplied by reaction 1. On the other hand, if reactions 1 and 3 are the only reactions occurring, then the resulting R.Q. is 1.33, i.e., too little oxygen is produced in relation to the CO_2 removed or, again from the other point of view, too much CO_2 is removed in relation to the O_2 produced.

Also, when the hydration reactions of KOH (4, 5, and 6) compete with reactions 2 and 3, the R.Q. is reduced. Therefore, to match man's R.Q. of 0.82 efficiently by utilizing KO_2 alone, the formation of both the carbonate and bicarbonate must occur, and the hydration of KOH must be minimized.

2.3 RECENT LITERATURE

Bovard and Sinisgalli (1958) investigated the use of KO_2 canisters for maintaining the atmosphere in a closed system. They used a small alcohol lamp in a 7 ft^3 drum as the system for one 22-hour test. The gas in the drum was circulated through a canister containing 375 grams of 4-8 mesh KO_2. They concluded that the use of KO_2 would be satisfactory as a source of oxygen and absorbent for carbon dioxide and water in closed systems housing humans. They felt more experimental work was required to optimize such a system.

Manned tests were conducted with two men enclosed in a 210 ft^3 chamber for 6.3 and 7 hours (Bovard, Mausteller, and Batutis, 1959). The atmosphere in these two tests was controlled by circulating the air through a KO_2 canister. It was concluded that KO_2 would satisfactorily control a large closed system atmosphere.

Bovard (1959) reviewed the use of potassium superoxide and sodium chlorate candles for controlling a closed chamber atmosphere. This review included two previous investigations made by Mine Safety Appliance (MSA) Research Corp.

Keating and Weiswurn (1960) demonstrated with human subjects in a sealed environment that a passive air regeneration system using potassium superoxide will provide air revitalization without using blowers, fans, or electric power. Their result of a one-man, 168-hour test indicates that approximately 0.33 lb of KO_2 per man-hour is required to passively regenerate a sealed environment. The KO_2 was able to maintain suitable O_2, CO_2, and relative humidity concentrations with no additional scrubbing chemical or driers. Two of the tests were terminated due to eye irritation of the subject from KO_2 powder in the air.

Optican (1962) evaluated the effects of the various operating parameters on KO_2 canisters for life support systems in a manned space vehicle. An annular, screen-type canister was developed to reduce the pressure drop through the KO_2 bed. The role of CO_2 concentration in

establishing the CO_2 adsorption rate and the O_2 generation rate was shown, as well as the roles of absolute humidity and catalysts in establishing the O_2 generation rate. He was unable to prove the feasibility of using KO_2 alone for both O_2 generation and CO_2 adsorption in a volume ratio matching man's R.Q.

A program to investigate the use of potassium and sodium superoxide for oxygen control in manned space vehicles was recently completed (Kunard and Rodgers, 1962). This study used the canister approach, i.e., passing the moist, CO_2-enriched air through a canister of KO_2 granules (usually 4-6 mesh). The results showed that there is no reaction of dry CO_2 with KO_2. This indicates that the CO_2 does not react directly with the KO_2, but only with the reaction product, KOH.

With high concentrations of CO_2 and water vapor, they found that the initial oxygen production rate was high. As the reaction proceeded, the O_2 production rate decreased while the water adsorption rate remained essentially constant. This indicated that hydration of the reaction products was competing with the reaction of KO_2.

They concluded that by controlling the inlet CO_2/H_2O mole ratio at 2/1, the ratio of CO_2 uptake to O_2 release could be controlled at about 0.82, matching the human R.Q. They also found that the absolute water concentration was the controlling factor in the rate of oxygen production. A high initial water concentration in the inlet stream resulted in a high initial rate of oxygen evolution. This rate dropped with time, even though the water concentration was maintained at the original high level, since the surface was becoming coated with the reaction products. After the KO_2 particles become coated, the evolution rate becomes dependent on the diffusion rate of oxygen out of, and water into the granule.

Kunard and Rodgers concluded that it is desirable to operate a KO_2 bed with the lowest water concentration which will provide suitable O_2 evolution. This will prevent mushing of the canister bed, due to overloading with water, and will also prevent over-production of O_2.

The recent Boeing test (NASA, 1964) used sodium superoxide (NaO_2) successfully for controlling the atmosphere of the sealed chamber. This was essentially a complete 150 man-day test. Some LiOH was used as a backup to remove CO_2, as required. Six beds of the superoxide were used with a total of about 900 lb of NaO_2. The total volume of the beds alone was about 38 ft^3. Chamber air was mixed with dried (silica-gel) air to give the desired inlet humidity. The average inlet humidity of the air was 0.0042 $\frac{\text{lb } H_2O}{\text{lb dry air}}$ (30 $\frac{\text{grains } H_2O}{\text{lb dry air}}$).

During the 29.37 days of operation, the oxygen concentration ranged from 19.0 to 22.8% with a time-weighted average of 20.89%. The CO_2 concentration ranged from 0.30 to 0.87% with a time-weighted average of 0.717%. The yield of oxygen was calculated as 96.7% of theoretical. LiOH was required for 5.7% of the total CO_2 removed.

As indicated by this brief review, the work to date has shown that KO_2 can be used effectively for generating oxygen and removing carbon dioxide in a closed environment. The remaining problem appears to be one of finding the most efficient means of using the chemical.

Electric Boat's proposed approach was to design a microcontactor unit capable of continuously producing fine mesh particle size KO_2. Fresh reactant surface would thus be continuously created and exposed. It was felt that the advantages of using small particle size KO_2 had not received full consideration. It was postulated that the rate of conversion of reactant to product increases by decreasing the particle size. The decrease in product film thickness, with the consequent decrease in resistance to reactant diffusion, was also expected to enhance the conversion of the solid reactant.

SECTION THREE

PRELIMINARY GRINDING RATE STUDIES

3.1 GENERAL

One of the main functions of the microcontactor concept was the continuous creation of small, fresh particles from a block of high-density KO_2. Potassium superoxide is available in densities reaching about 120 lb/ft^3. Both unsintered and sintered types are available. Unsintered KO_2 is prepared by compressing to the desired density the very fine (-200 mesh) yellow "fluff" formed by spraying molten potassium into dry air. The sintered material is prepared by compressing the same starting material to an intermediate density and then crushing to a particle size of about 20 mesh. These particles are sintered and then compressed in a mold to the final desired density. Sintered KO_2 is a darker yellow color than the unsintered.

The relationship between mesh size, average diameter, average outer surface area, and total outer surface area of KO_2 particles is shown in Table 3-1. In designating the average diameter and outer surface area, it was assumed that the particles are spherical in shape. Mesh size designates the number of openings per inch in a screen, with particle diameter based on values given for the Tyler standard seive series. The column on the extreme right of the table illustrates how the total outer surface area of the KO_2 particles (per unit weight of material) increases as the particle size decreases.

3.2 LABORATORY TEST GRINDER DESIGN

The method selected for manufacturing the required small particles featured a device for grinding the high-density KO_2 block which would be held in place by spring-loading at constant pressure against a suitable grinder. After reviewing possible grinding methods, the use of a slowly revolving cutting tool appeared to be the most feasible. A small test device was designed to determine the grinding characteristics, i.e., product particle sizes, grinding rates, and the spring loading and

TABLE 3-1

RELATIONSHIP OF MESH SIZE, AVERAGE DIAMETER, OUTER SURFACE AREA AND TOTAL OUTER SURFACE AREA OF HIGH-DENSITY KO_2 PARTICLES

Mesh (openings/in.)	Particle Size Range (in.)	D_{avg} (in.)	D_{avg} (micron)	Particle Outer Surface Area* ($ft^2 \times 10^6$)	Total Outer Surface Area of KO_2 Particles with $\rho = 120\ lb/ft^3$ $\left(\frac{ft^2}{lb\ KO_2}\right)$
4-6	0.131 - 0.185	0.158	4010	544	3.81
6-8	0.093 - 0.131	0.112	2840	274	5.36
8-10	0.065 - 0.093	0.079	2010	136	7.55
10-14	0.046 - 0.065	0.0555	1410	67.2	10.85
14-20	0.0328-.0.046	0.0394	1000	33.9	14.95
20-28	0.0232-0.0328	0.0280	711	17.1	21.5
28-35	0.0164-0.0232	0.0198	502	8.55	30.3
35-48	0.0116-0.0164	0.0140	355	4.27	42.8
48-65	0.0082-0.0116	0.0099	251	2.14	61.0
65-100	0.0058-0.0082	0.0075	191	1.23	80.5
100-150	0.0041-0.0058	0.0050	127	0.545	120.
150-200	0.0029-0.0041	0.0035	89	0.267	172
200-270	0.0021-0.0029	0.0025	63.5	0.136	242
270-400	0.0015-0.0021	0.0018	45.7	0.0707	335

*Calculated assuming the particle is a non-porous solid sphere.

power requirements for proper design of a laboratory prototype unit. A schematic of the test device is shown in Figure 3-1. The grinding test device was driven by a 2-rpm, gear-head, a-c motor coupled to the shaft of a carbon steel spindle. The spindle passed through the clear plexiglass, particle collector and was attached to a tool holder. This tool holder held two tungsten carbide cutters, each measuring one-half the diameter of the tool holder. They were mounted radially, 180° apart from each other and each had a 7° relief angle. The KO_2 sample block was held in place by a holder mounted to the frame. A moving retainer, which incorporated two holding pins, held the KO_2 block inside the holder. A follower with a square shaft prevented the retainer and KO_2 block from rotating but allowed vertical movement as the KO_2 block was forced against the cutting tool. A platform was attached to the top of the follower shaft to support weights which simulated various spring pressures. Photographs of the assembled unit are shown in Figure 3-2. and Figure 3-3. At the completion of the runs with the 2-rpm motor, modifications were made to increase the grinding rate by replacing the 2-rpm motor with a 7-rpm motor and by adding two more cutter blades, making a total of four, each placed 90° apart.

3.3 TEST PROCEDURE

The grinding device and the two KO_2 sample blocks, sintered and unsintered, each 115 lb/ft^3 were placed in a dry box. The grinding device was leveled so as to have the face of the cutting tool in a true horizontal position. The dry box was allowed to come to equilibrium before the KO_2 block was removed from its sealed container. With the holder loosened and free to slide up and down on the support frame, the KO_2 sample block was placed in the holder and its pin holes lined up with the pins of the retainer. The holder was then leveled to assure a true horizontal alignment of the surface of the KO_2 block with the face of the cutting tool. About 1/8 in. of the block was exposed below the edge of the holder. The holder was then bolted in this position and the desired weight was placed on the platform.

The grinder was operated for two to five minutes with the particles collecting on the tool holder and in the particle collector. At the

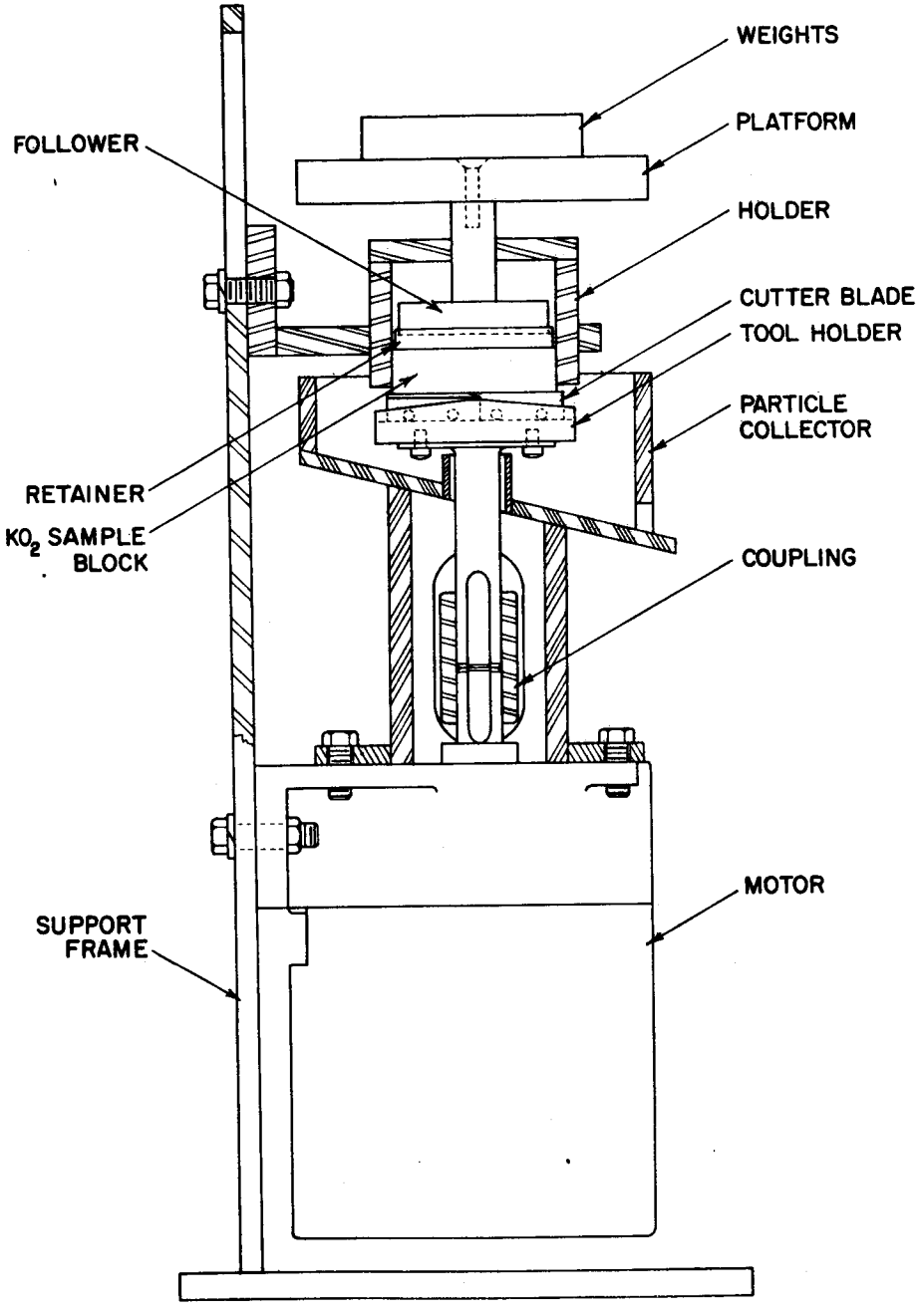


FIGURE 3-1 SCHEMATIC OF LABORATORY GRINDER TEST DEVICE

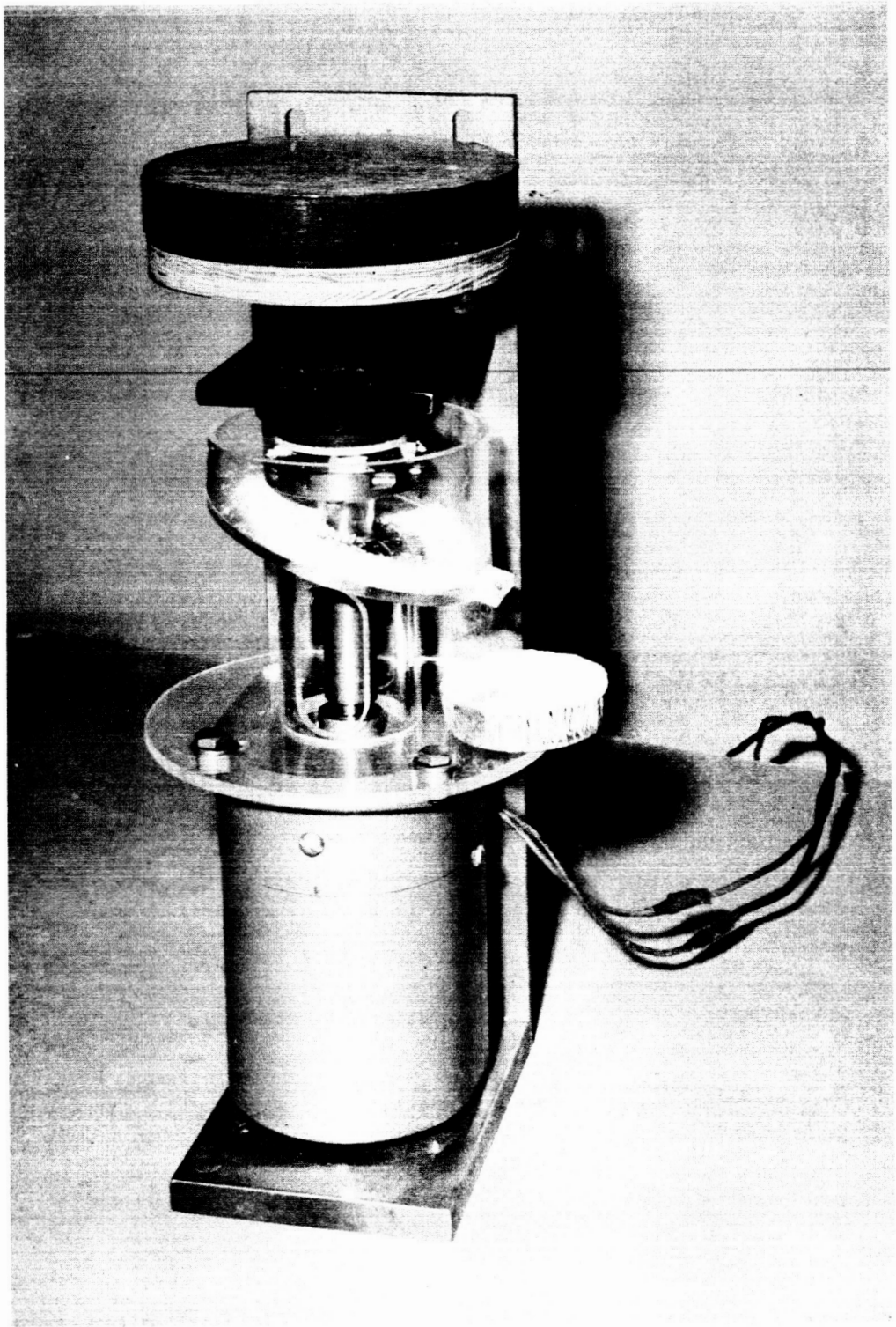


FIGURE 3-2 LABORATORY GRINDER WITH EPOXY/TALC TEST BLOCK

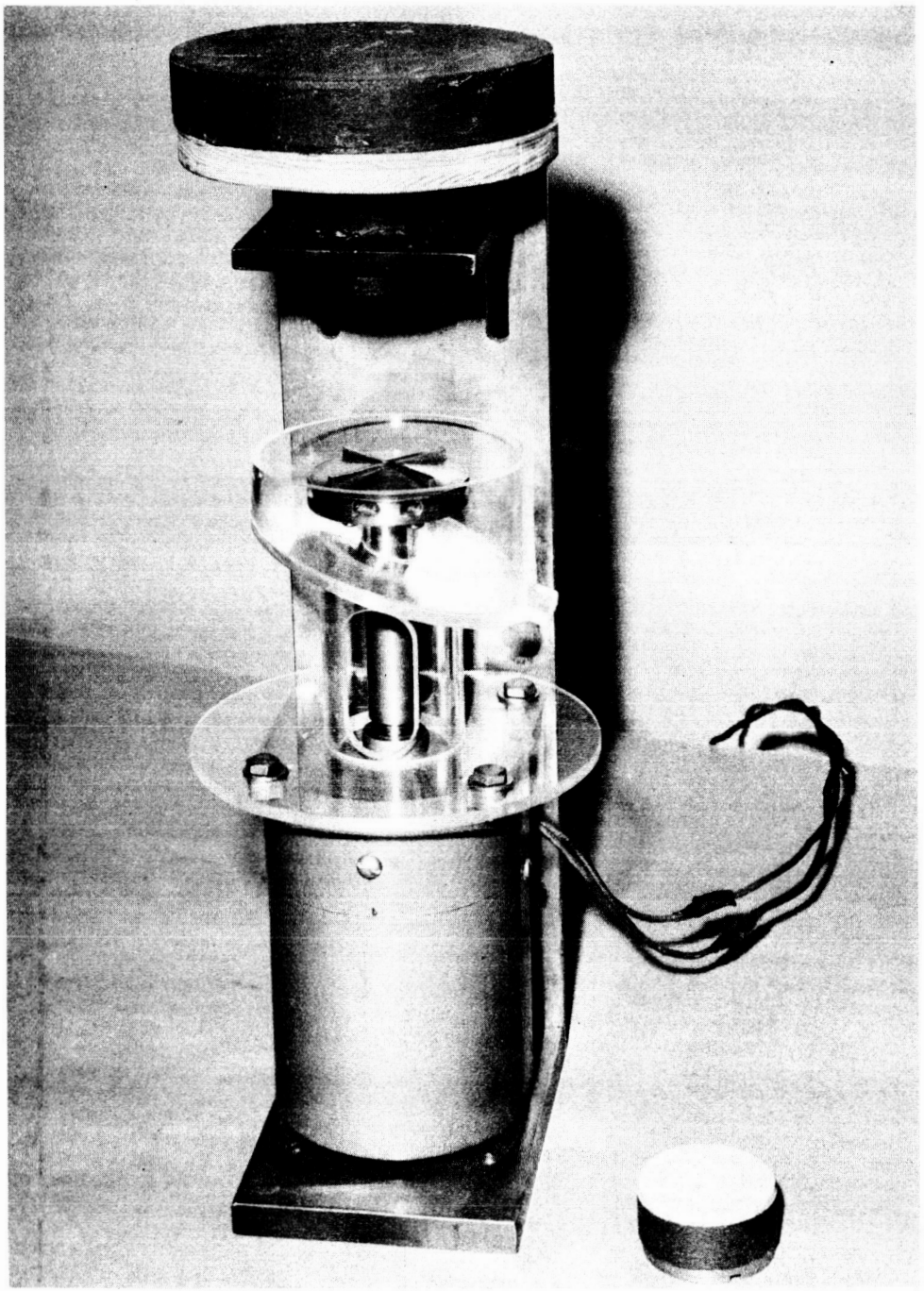


FIGURE 3-3 LABORATORY GRINDER WITH 2 RPM MOTOR AND FOUR CUTTING BLADES

end of the allotted time, the motor was shut off and the particles collected and weighed. A screen analysis was then made on the product obtained.

Starting torque measurements were made by removing the plastic particle collector and observing the average force necessary to start the cutter blades moving against the KO_2 block at various loadings. A rigid metal rod was inserted into a set-screw hole in the side of the cutting tool head so that the required force could be applied and measured at a fixed distance from the center of the cutter assembly. Readings were taken using a calibrated, portable, spring-type scale applied tangentially to the direction of rotation. To minimize the shaft friction inherent in the system, a small Teflon disc was placed between the top of the motor shaft and the bottom of the cutter spindle. A coupling was fastened to the motor shaft with a set-screw and the cutter spindle was allowed to rotate freely in the upper part of the coupling.

Grinding rate and torque measurement tests were made at various weight loadings, using both the sintered and the unsintered KO_2 samples and a dummy block made of (40%/60%) epoxy/talc. The grinding experiments were first conducted on the dummy epoxy/talc block to check out the operation of the assembly. After completing runs with the 2-rpm motor, the experiments were repeated using the 7-rpm motor and the four blades.

3.4 GRINDER TEST RESULTS

The results of all the grinding tests are summarized in Tables 3-2, 3-3, and 3-4, and Figures 3-4 and 3-5.

The runs using the 2-rpm motor and the two cutting blades experienced some difficulty in aligning the block on the cutting blade. The four blades positioned 90° apart, eliminated this alignment problem during the runs using the 7-rpm motor.

A 1/4-in. diameter hole was drilled in the center of each of the test blocks during the 7-rpm runs to eliminate the near-zero blade velocity

TABLE 3-2

SUMMARY OF GRINDING TESTS - RATE (LB/DAY)

Material	Wt. on Blade (lb)	2-rpm, 2-blade	7-rpm, 4-blade	7-rpm 4-blade 1/4-in. center hole
Epoxy/Talc	6	0.15	0.03	0.04
Dummy Block	11	----	0.20	0.22
	16	0.18	0.39	0.63
	21	0.30	0.86	1.08
	26	0.40	----	1.57
Unsintered	6	0.25	----	1.3
KO_2 Block	11	0.25	----	2.3
	16	----	----	3.7
Sintered	6	0.41*	----	1.2
KO_2 Block	11	0.23	----	3.2*

*Piece broken off block, probably a high value.

TABLE 3-3

SUMMARY OF SCREENING ANALYSIS

(SIZE DISTRIBUTION IN PERCENT BY WEIGHT OF PRODUCT)

Type of High-density KO_2 Used	Tests at 2-rpm Using 2 Blades			Tests at 7-rpm Using 4 Blades			
	Mesh Size	Percent of Product Wt. (6-lb Force)	(11-lb Force)	Mesh Size	Percent of Product Wt. (6-lb Force)	(11-lb Force)	(16-lb Force)
Unsintered	+20	0	0	+20	1.2	1.2	0
	-20 + 60	50	19	-20 + 40	2.1	10.9	3.6
				-40 + 60	5.7	7.4	6.1
	-60 + 80	15	9	-60 + 80	6.9	7.6	8.4
	-80	35	72	-80 + 100	12.5	10.4	11.0
				-100	71.6	62.5	70.9
Sintered				+20	1.1	8.7*	--
	+40	66	18	-20 + 40	5.9	20.0	--
	-40 + 80	19	32	-40 + 60	19.2	22.9	--
				-60 + 80	15.8	12.9	--
	-80	15	50	-80 + 100	16.5	12.0	--
				-100	41.5	23.5	--

*Several large pieces of the sintered block broke off, giving a higher proportion of larger particles.

TABLE 3-4

SUMMARY OF STARTING TORQUE RESULTS

Starting Torque with Unsintered KO_2 (4 Blades)

Weight on Blade (lb)	Starting Torque (lb-ft)
6	0.5
11	0.78
16	1.12

Starting Torque with Sintered KO_2 (2 Blades)

Weight on Blade (lb)	Starting Torque (lb-ft)
6	0.3
11	0.9

Starting Torque with Epoxy Block

Weight on Blade (lb)	Starting Torque (2 Blades) (lb-ft)	Starting Torque (4 Blades) (lb-ft)
6	0.15	0.25
11	0.25	0.6
16	0.58	1.0

at the center. This modification increased the grinding rate significantly as can be seen in Table 3-2.

The smaller particles which resulted from grinding the unsintered KO_2 block were in the form of fine granules, while the larger particles were short and needle-like in shape. The sintered material tended to fragment into the precompression particles (about 20 mesh) and showed more of a tendency toward crumbling. This may account for the higher ratio of larger particles.

3.5 DISCUSSION OF RESULTS

Both sintered and unsintered KO_2 gave a higher proportion of fine particles than was originally anticipated. Because the high-density material was so hard and required such a large force against the cutter blade, much of the energy was expended in particle size reduction.

Grinding rate results using the epoxy/talc dummy block indicated an exponential increase with increasing blade weight (see Figure 3-4).

Data for the 2-rpm runs were sufficient only to establish the fact that a small fraction of the desired 7-lb per day was achieved. These data were later supplemented with more complete information at conditions permitting a higher grinding level. The 2-rpm grinding rates were so much lower than anticipated that additional runs at this low speed were not made.

After modifying the testing device (increasing the speed to 7 rpm and doubling the number of cutting blades) grinding rates for both sintered and unsintered test blocks appeared to be almost directly proportional to the total weight on the blades over the range of weights tested. Data for comparing the two grinding conditions are shown in Figure 3-5.

Increasing the weight on the two blades of the 2-rpm grinder resulted in a further reduction of particle size for both types of KO_2. Using the four-bladed, 7-rpm grinder and the unsintered KO_2 block, increase in weight showed no significant further reduction of particle size over that obtained at 11 lb and 2-rpm. The higher proportion of larger

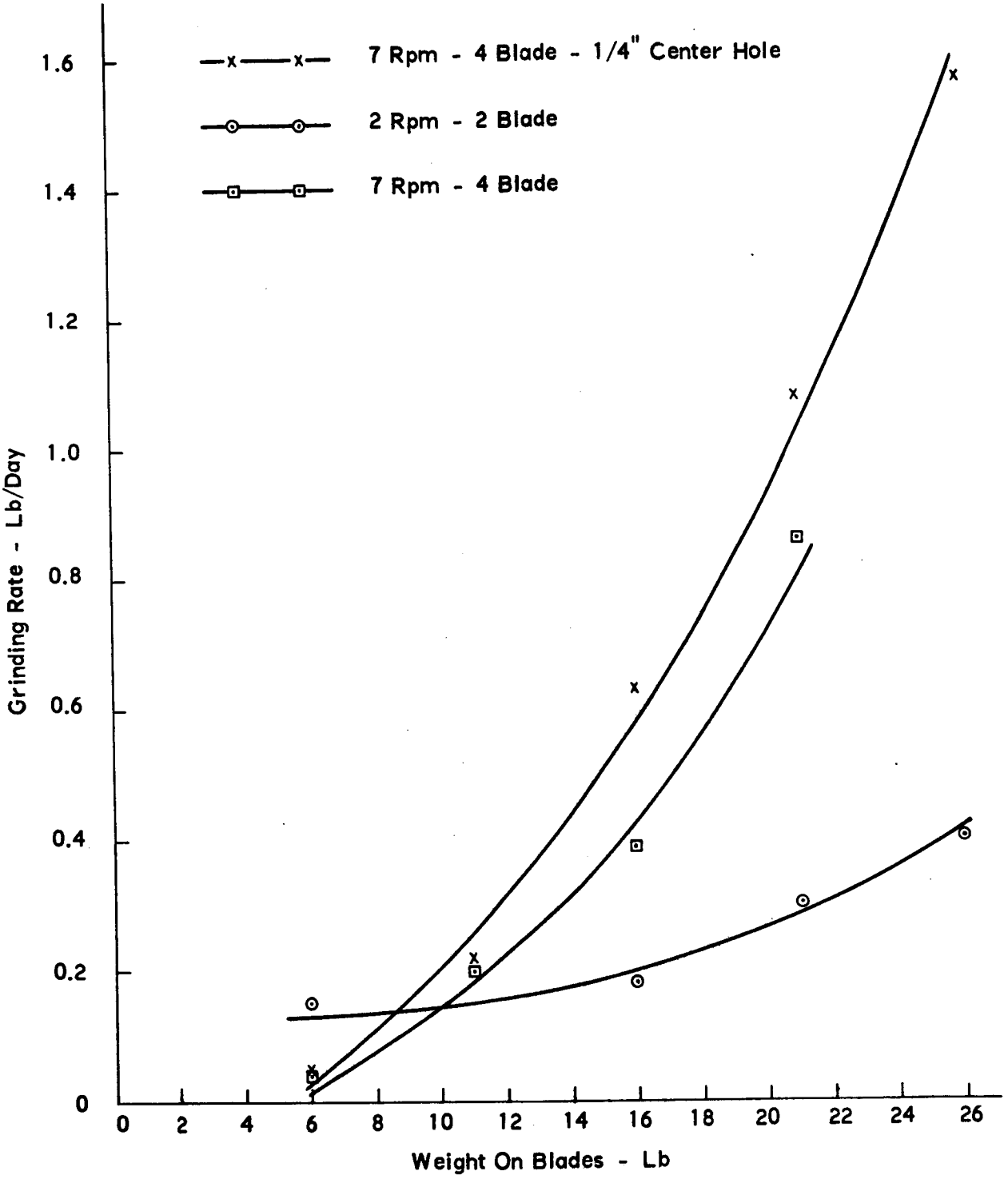


FIGURE 3-4 EFFECT OF WEIGHT ON BLADES ON GRINDING RATE FOR EPOXY/TALC SAMPLE BLOCK

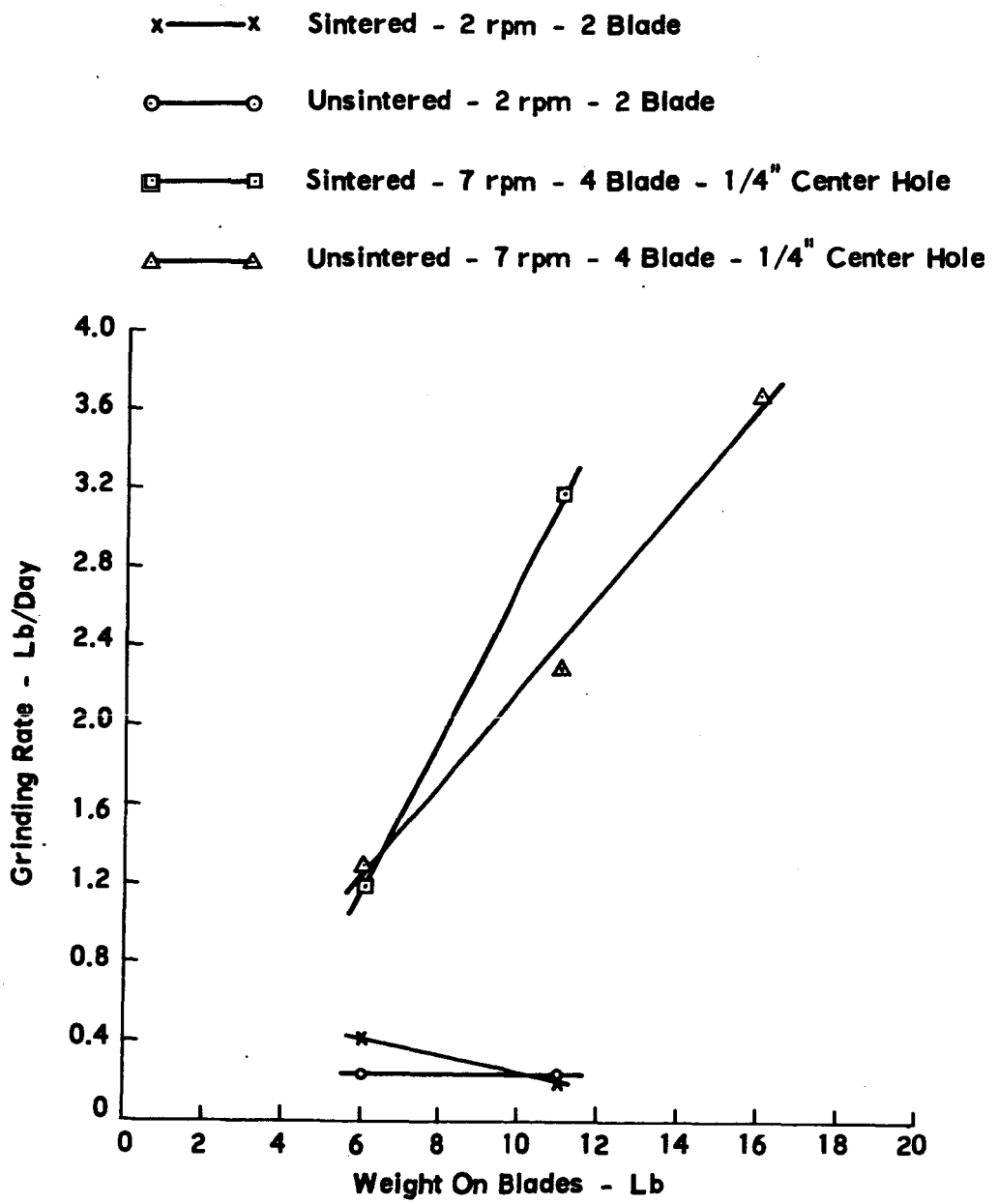


FIGURE 3-5 EFFECT OF WEIGHT ON BLADES ON GRINDING RATE FOR KO_2 SAMPLE BLOCKS

particles obtained when grinding the sintered material at 7 rpm can be attributed to the crumbling action noticed when grinding the sintered KO_2.

The starting torque appeared to be directly proportional to the pressure against the blade. However, it should be pointed out that the reported values are averages and that there was a considerable range in the experimental results obtained.

Although the particle sizes produced were much smaller than originally anticipated (20-40 mesh), these smaller particles would be expected to result in faster reaction rates as a result of increased surface area with all other things, such as porosity, being equal. A possible disadvantage may be a somewhat higher pressure drop across any filter used to remove the reaction products. The unsintered material appeared to have the better grinding characteristics since it resisted crumbling and was actually ground by the cutting tool.

As stated earlier, the grinding rates with the 2-rpm motor were much lower than anticipated. This was probably due to the hardness of the material which did not allow the cutting tool to cut as deeply as was assumed. After the motor speed was increased and the cutting blades increased from two to four, a higher grinding rate was achieved.

It was concluded that the grinding rate results obtained were sufficient to design a grinder device for a one-man microcontactor laboratory prototype. Grinding rates could be studied further in the prototype.

SECTION FOUR

PRELIMINARY RATE OF REACTION STUDIES

4.1 GENERAL

The only reaction rate data available are average rates of oxygen production during a test period of several hours. A major feature of the microcontactor approach was the anticipated high reaction rate that would occur in the reaction zone while the KO_2 particles were being swept through the system by the air stream. For this reason it was important that the reaction rates, as affected by the operating parameters, were well understood, especially during the short interval immediately following exposure to the moist air. This information was needed for proper design of the laboratory prototype. Semi-quantitative tests were therefore made to determine the extent of reaction as a function of time, moisture concentration, particle size, and carbon dioxide concentration. The test set-up was designed to simulate a fluidized bed reaction which allows intimate contact of the KO_2 particles and the inlet air.

4.2 TEST APPARATUS

The test arrangement that was used to investigate the rate of reaction of KO_2 is shown in Figure 4-1. The inlet gas was taken from the house CO_2 - air mixing tank, which allows the CO_2 level to be controlled at any desired concentration. House air was used to establish the zero point of the CO_2 analyzer. Both gas inlet streams were passed through individual flow regulators.

A humidifier which consisted of a mixing chamber that permitted the inlet gas to bubble through water at room temperature was used to add moisture to the house air-CO_2 supply. Since the house air supply was quite dry, it was not necessary to remove moisture from the inlet air for this test program. A by-pass control was provided so that a portion of the dry inlet gas mixture could be mixed with the humidified stream thereby allowing control over the desired range of humidity.

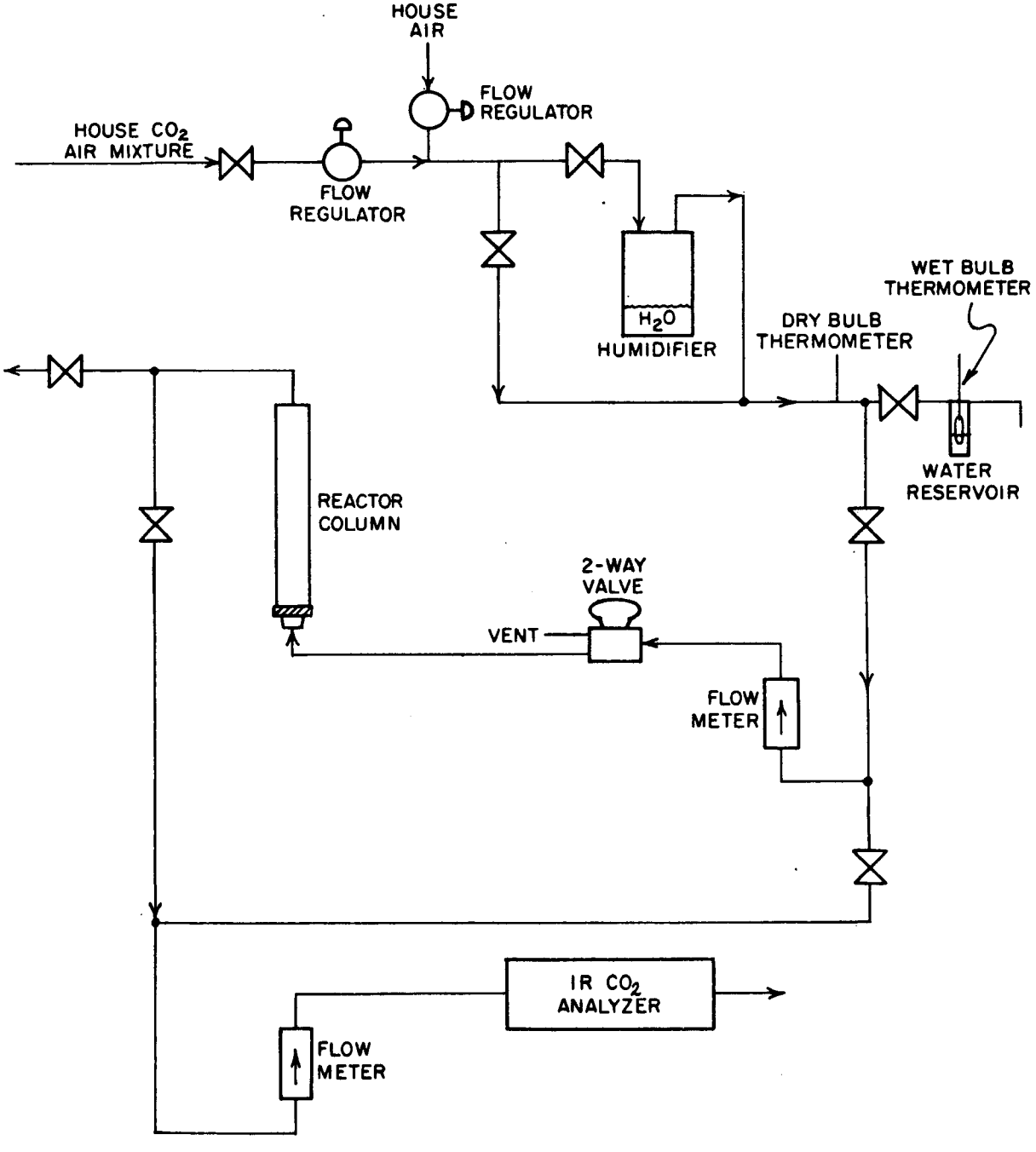


FIGURE 4-1 SCHEMATIC FOR RATE OF REACTION STUDIES

Humidity control, ranging from about 40 to 90 grains water per pound dry air (30% to 90% R.H. at room temperature), could be realized from this system at flow rates up to about 2 scfm. Absolute humidity values were used throughout, since the relative humidity expression is meaningful only when accompanied by the appropriate dry bulb temperature.

Downstream from the humidifier, a thermometer was inserted in the line to measure the dry bulb temperature of the inlet air. A wet bulb thermometer was inserted into a bleed line off the main gas line. Wet bulb measurements were made in the bleed line so as not to add water vapor to the air while the wet bulb temperature was being measured. The main gas line then passed through a flow meter, and then to a 2-way valve with one position to a vent and the other position to the reactor column. The reactor column was a 1-ft length of 1-in. ID plexiglass tubing. The reactor column inlet passed through a quick-disconnect adaptor which held a porous, stainless-steel filter (165 microns). Sample lines upstream and downstream from the reactor column passed through a small flow meter to a Lira CO_2 Infrared Analyzer.

4.3 TEST PROCEDURE

Because of the limited supply of high-density KO_2, the rate of reaction runs were made with low-density KO_2 (41 lb/ft^3 bulk density). This material was supplied in a 4 - 8 mesh size and was then ground with a mortar and pestle. The ground product was separated into the various particle sizes by using a set of mesh screens. Weighed samples of the various particle sizes were then prepared. All handling of KO_2 was done in a drybox except for the weighings, in which case the KO_2 was always in an air-tight sample bottle.

A run was initiated by setting the desired reaction column inlet moisture and CO_2 concentrations. These conditions were obtained by adjusting the various control valves.

When the desired conditions were attained, the run was started by placing the 2-way valve in the vent position and removing the adaptor and filter from the bottom of the reactor column. The test sample of KO_2

was placed on the filter and then the adaptor and filter were refastened to the bottom of the column. The 2-way valve was then placed in the column inlet position and the gas mixture was allowed to pass through the column. The air flow was adjusted such that all or most of the KO_2 particles were suspended in the air stream. At the end of the time increment, the 2-way valve was turned to the vent position and the adaptor and filter were quickly removed from the column. The filter and reaction products were transferred from the adaptor into the analysis reaction chamber. An analysis was then made on the partially reacted sample for the amount of remaining available oxygen.

4.4 KO_2 ANALYSIS

The amount of available oxygen from the partially reacted KO_2 sample was determined by the addition of an excess of water, then measuring the volume of gas produced in a gas buret. It was assumed that all the gas produced by the water reaction was oxygen.

The apparatus required for this analysis included a reaction chamber, an addition buret, and a gas buret with a compensator. All connections were of ground glass joints. After the KO_2 sample had been placed in the reaction chamber, the addition buret was inserted and the gas buret was connected to the addition buret. The level in the gas buret was adjusted to atmospheric pressure, then the stopcock of the addition buret was closed. The addition buret was filled with distilled water, which was then slowly added to the KO_2 sample in the reaction chamber. An excess of water (about 15 ml) was added. A magnetic stirrer was used intermittently to ensure good mixing and complete reaction. When the reaction was complete, as indicated by no further change in gas buret readings, the gas buret was leveled to atmospheric pressure and the volume recorded. Both initial and final gas buret readings were corrected to standard conditions and the percent reaction calculated on the basis of previously determined blanks.

4.5 TEST RESULTS

A summary of all the rate of reaction runs is shown in Table 4-1. Figures 4-2 and 4-3 show the relationship of percent reaction with the parameters of inlet moisture concentration and KO_2 particle size. Moisture is expressed as grains water per lb of dry air, and percent relative humidity is shown for convenience. These runs were all made with an inlet CO_2 concentration of 0.5% and a run length of 5 minutes. The amount of reaction increased with an increasing moisture concentration as was expected, but there was no apparent effect produced by different particle sizes.

Figure 4-4 shows the oxygen volume produced (cc/gm) as a function of time for various inlet air moisture concentrations. These curves are for 100 - 150 mesh KO_2 particles with an inlet CO_2 concentration of 0.5%. As would be expected, the higher moisture concentrations resulted in a higher rate of reaction, but overall the reaction rates were slower than originally anticipated.

The effect of CO_2 concentration on the rate of oxygen production is shown for two different carbon dioxide concentrations by Figure 4-5. An attempt was made to measure the change in CO_2 concentration as it passed through the reactor column but the infrared instrument was not sensitive enough to quantitatively measure the small difference.

4.6 DISCUSSION OF RESULTS

Apparently, KO_2 particle size in the range from 20 - 150 mesh has only a slight effect on the rate of reaction under the experimental conditions studied as shown by Figures 4-2 and 4-3. There are two possible explanations for this result. The first is that by decreasing the particle size, the outside surface area per unit weight increases significantly but the total available surface area, which is comprised mostly of internal area due to the porosity of the particle, changes only slightly. Therefore, the total available surface area may be changing very little as the particle size is decreased. This is illustrated by Table 4-2 which shows the surface area of various size KO_2 particles measured by Kunard and Rodgers (1962) using the standard

TABLE 4-1A

RATE OF REACTION STUDY RESULTS

RUN #	MESH SIZE	TYPE OF RUN	SAMPLE WEIGHT (Gm)	LENGTH OF RUN (Min.)	CO_2 CONC (%)	AIR VELOCITY (Ft/Sec)	WATER CONC $\left(\frac{\text{Grains}}{\text{Lb Dry Air}}\right)$	AIR DRY BULB TEMP. ($^{\circ}F$)
1	-20 + 40	Blank	0.293	--	---	---	----	----
2	-20 + 40	Water	0.280	5	0.5	2.7	48	77
3	-20 + 40	Water	0.251	5	0.5	2.9	51.5	75
4	-20 + 40	Water	0.272	5	0.5	2.9	85	75
5	-20 + 40	Blank	0.180	--	---	---	----	----
6	-60 + 80	Blank	0.245	--	---	---	----	----
7	-60 + 80	Water	0.274	5	0.5	1.6	39	74.5
8	-60 + 80	Water	0.273	5	0.5	1.9	83	74
9	-60 + 80	Water	0.286	5	0.5	1.7	56	72
10	-20 + 40	Water	0.256	5	0.5	2.9	70	73.5
11	-60 + 80	Water	0.263	5	0.5	1.7	67.5	74.5
12	-60 + 80	Water	0.229	5	0.5	1.6	59	77
13	-60 + 80	Water	0.233	5	0.5	1.6	55	76.5
14	-100 + 150	Blank	0.299	--	---	---	----	----
15	-100 + 150	Water	0.297	5	0.5	1.2	50	77
16	-100 + 150	Water	0.315	5	0.5	1.2	61	80
17	-100 + 150	Water	0.345	5	0.5	1.2	70	79
18	-100 + 150	Water	0.369	5	0.5	1.3	82	78
19	-100 + 150	Time	0.263	3	0.5	1.2	82	79
20	-100 + 150	Time	0.237	3	0.5	1.3	62	81
21	-100 + 150	Time	0.264	3	0.5	1.3	69	80
22	-100 + 150	Time	0.212	1	0.5	1.0	61.5	78
23	-100 + 150	Time	0.186	1	0.5	1.2	79.5	81
24	-100 + 150	Time	0.233	15	0.5	1.2	61	81.5
25	-100 + 150	Time	0.221	10	0.5	1.1	62	80
26	-100 + 150	CO_2	0.267	3	1.0	1.1	61	80
27	-100 + 150	CO_2	0.298	10	1.0	1.1	61.5	80
28	-100 + 150	Blank	0.162	--	---	---	----	----
29	----------	Blank	0.158	--	---	---	----	----
30	----------	Blank	0.092	--	---	---	----	----
31	----------	Blank	0.295	--	---	---	----	----
32	----------	Blank	0.362	--	---	---	----	----
33	----------	Blank	0.381	--	---	---	---	----
34	----------	Blank	0.200	--	---	---	----	----
35	-100 + 150	CO_2	0.213	5	1.0	1.2	62	79
36	-100 + 150	CO_2	0.222	5	1.0	1.2	70	79
37	-100 + 150	CO_2	0.240	10	1.0	1.3	70	79
38	-100 + 150	CO_2	0.307	3	1.0	1.2	68.5	77
39	-100 + 150	Blank	0.204	--	---	---	----	----
40	-100 + 150	Blank	0.209	--	---	---	----	----
41	-100 + 150	Blank	0.177	--	---	---	----	----
42	-100 + 150	CO_2	0.196	10	1.0	1.2	67.5	74
43	-100 + 150	CO_2	0.226	15	1.0	1.2	60	75
44	-100 + 150	CO_2	0.171	3	1.0	1.2	61	79.5
45	-100 + 150	CO_2	0.202	15	1.0	1.2	59	76
46	-100 + 150	Time	0.199	5	0.5	1.2	60	80.5

TABLE 4-1B

RATE OF REACTION STUDY RESULTS

RUN #	RELATIVE HUMIDITY (%)	GAS VOL PRODUCED $\left(\frac{cc}{GM}\right)$	% REACTION	REMARKS
1	----	195	----	Equalizing trouble at start - liquid sticking to buret
2	34	16.4	8.3	Equalizing trouble at start - liquid sticking to buret
3	41	87.7	44.5	Magnetic stirrer left on - heated reaction chamber
4	66	156.4	79.4	
5	----	185.9	----	
6	----	194.1	----	
7	30.6	13.4	6.8	
8	65.7	178.3	90.5	
9	45.8	127.6	64.8	Air inlet tube popped off - length of run may be in error
10	55.2	115	58.4	Trouble replacing filter after loading
11	52.7	143.5	72.9	
12	42	115.3	58.6	Gas leak out water buret
13	39.9	137.2	69.7	
14	----	202.4	----	
15	35.6	44.9	22.8	
16	39.1	164.4	83.5	Spilled sample in transfer - some probably lost
17	46.4	115.6	58.7	
18	56.2	159.6	81.0	
19	53	161.5	82.0	
20	37.9	118	59.9	
21	43.6	152.3	77.4	
22	42.8	78.2	39.7	
23	49.1	126	64.3	
24	37	187.7	95.2	
25	39.1	172.3	87.5	
26	39.1	97.9	49.7	
27	39.1	-----	----	Negative gas volume
28	----	14.6	----	Unsintered KO_2 from grinding exp
29	----	113.4	----	Freshly ground sintered KO_2
30	----	75.1	----	Freshly ground unsintered KO_2
31	----	128.5	----	Freshly ground unsintered KO_2
32	----	81.1	----	Freshly ground sintered KO_2 - possible weighing error
33	----	181.5	----	Freshly ground sintered KO_2
34	----	161.6	----	MSA old sample of high density KO_2
35	40.4	148.7	75.6	
36	45.8	188.2	95.6	
37	45.8	-----	----	Negative gas volume
38	49.1	182.9	92.9	
39	----	182.5	92.8	Ground unsintered KO_2
40	----	192.1	----	Low density KO_2
41	----	200.9	----	Low density KO_2
42	54.3	176.4	89.6	
43	46.7	163	82.8	
44	39.9	98.1	49.8	
45	44.8	171.3	87	
46	38.6	155.2	79	

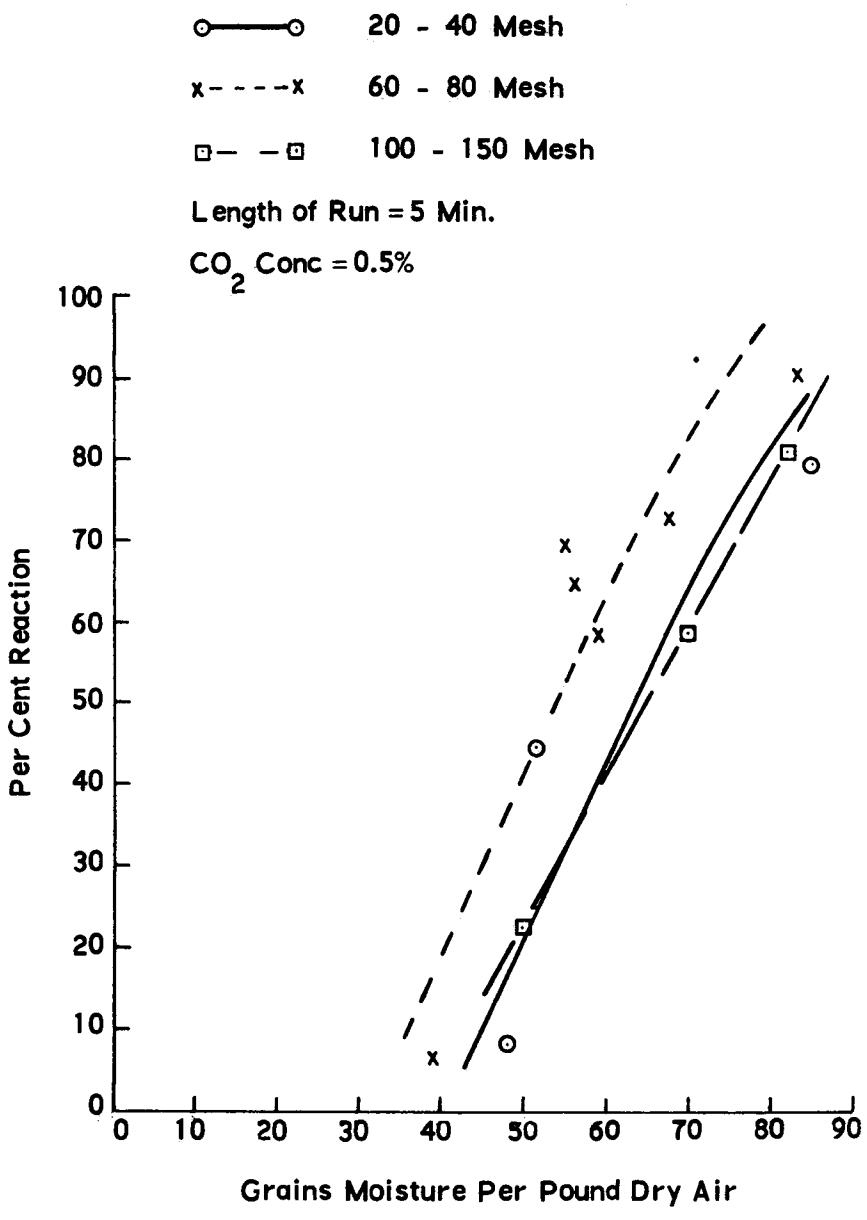


FIGURE 4-2 PER CENT KO_2 REACTION AS FUNCTION OF MOISTURE CONCENTRATION AND PARTICLE SIZE

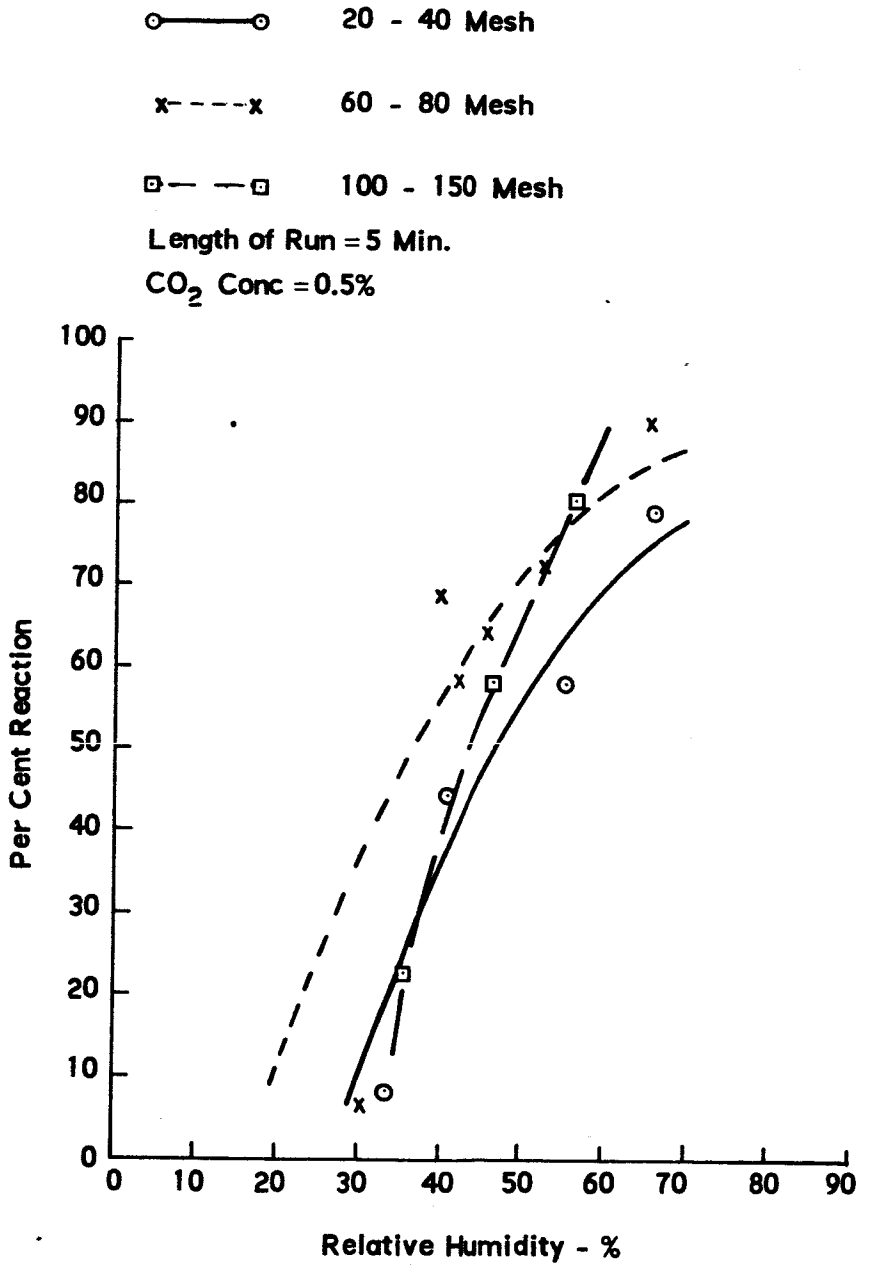


FIGURE 4-3 PER CENT KO_2 REACTION AS FUNCTION OF RELATIVE HUMIDITY AND PARTICLE SIZE

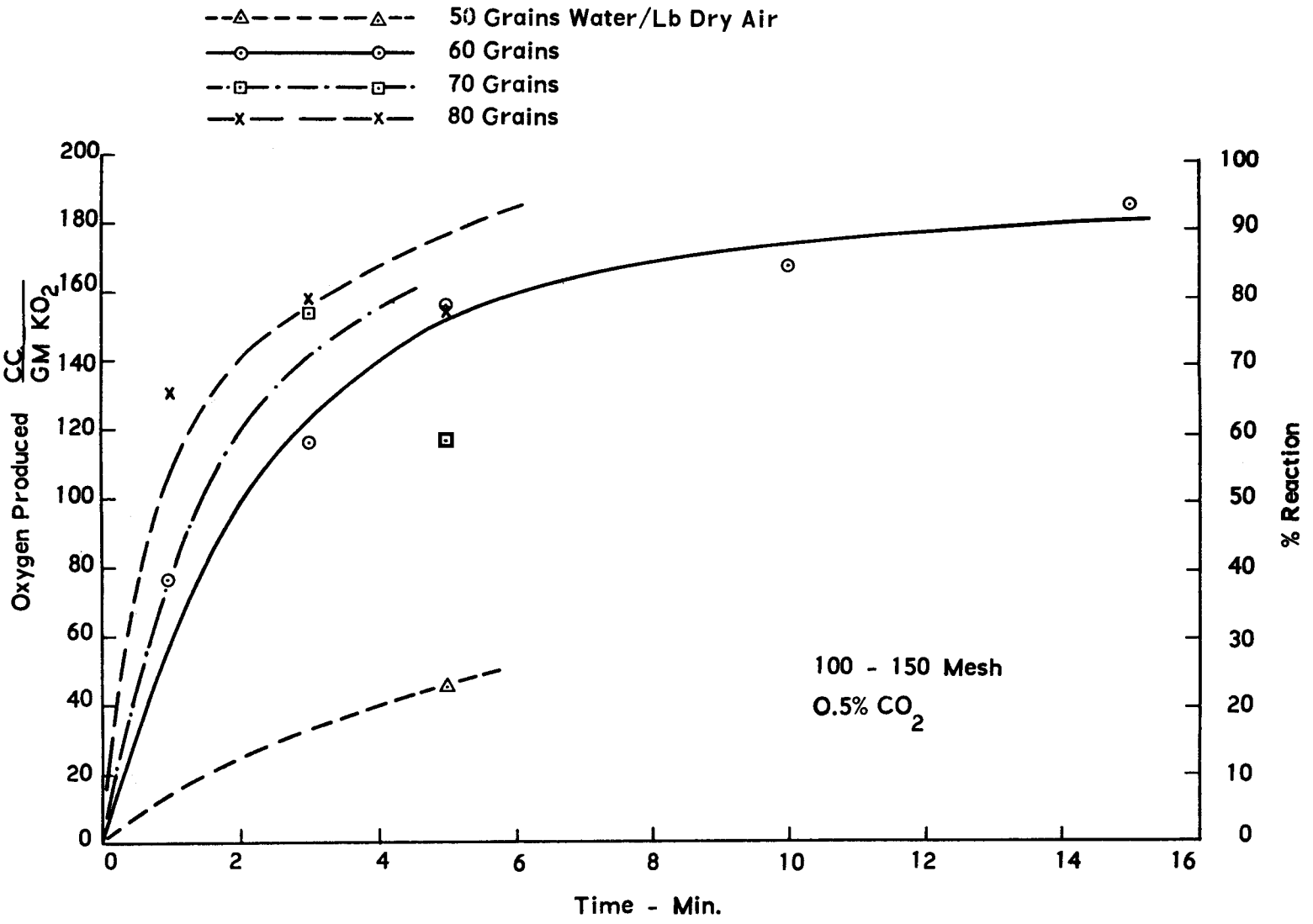


FIGURE 4-4 OXYGEN PRODUCED AS FUNCTION OF TIME AND INLET AIR MOISTURE CONCENTRATION

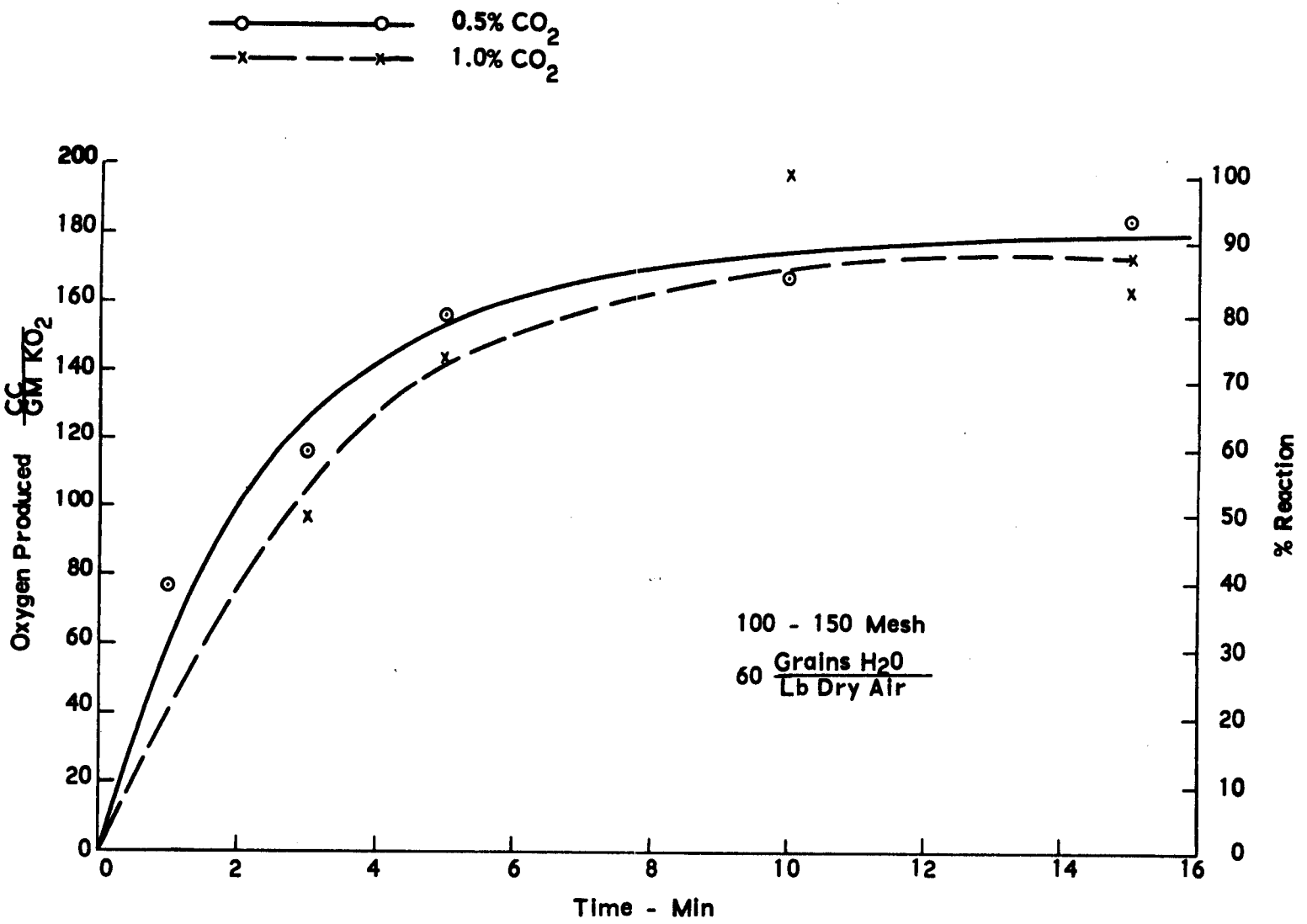


FIGURE 4-5 EFFECT OF CO_2 CONCENTRATION ON RATE OF OXYGEN PRODUCTION

TABLE 4-2

COMPARISON OF MEASURED SURFACE AREA WITH CALCULATED OUTER SURFACE AREA FOR VARIOUS SIZE KO_2 PARTICLES

Particle Size (Mesh)	Measured Surface Area $\left(\frac{m^2}{gm} \times 10^3\right)$	Calculated Outer Surface Area $\left(\frac{m^2}{gm} \times 10^3\right)$
2-4	400	
4-6		0.6
6-8		0.84
8-10	400	1.2
8-10 sintered	< 100	1.2
10-14		1.74
10-14 KO_2 + 1% copper oxychloride catalyst	600	1.74
14-20		2.43
20-28		3.39
"Fluff" (200-270)	400	37.6

Brunnauer - Emmett - Teller nitrogen absorption techniques. As a comparison, the outer surface area of various size particles was calculated assuming the particles were spherical and had a density of about 2.5 gm per cc. This table shows that the outer surface is a small part of the total available surface and, therefore, the actual particle size is not as important as the internal porosity of the material.

The second possible explanation is that the moment the KO_2 particles start to react with the moisture in the air stream, the particles agglomerate, which gives, in effect, larger size particles. The smaller size particles tended to agglomerate more readily than the larger size particles.

Figure 4-4 shows that after an exposure time of two minutes, the percent reaction ranged from 15% to 70% for the different moisture concentrations investigated. The resulting curves indicate that the initial rate of reaction is rapid as a result of it being a surface reaction. Once the reaction becomes diffusion controlled, the rates become slower.

The effect of CO_2 concentration on the rate of oxygen production does not appear to be significant as shown by Figure 4-5. Apparently, the diffusion process for water vapor and oxygen is the same through KOH and its hydrates with small carbonate concentrations as through the same products with higher carbonate concentrations.

After 27 runs with the low-density material had been completed, preparations were made to compare these results with a limited number of runs made with the unsintered, high-density KO_2. The high-density blanks using freshly ground material and previously ground samples from the grinding experiments were all significantly lower than the blanks made on the low-density material (see Table 4-1). No reasonable explanation was evident to account for the lower available oxygen content of the high-density material. However, the supplier guaranteed an over-all purity of 92% on the basis of oxygen evolution on all future high-density KO_2 material. In later shipments, chemical analyses were made by the supplier prior to shipment, and by General Dynamics/Electric Boat upon receipt, to verify the actual oxygen content of the material.

One possible source of error in the experimental work is the determination of the moisture concentration using wet and dry bulb thermometer readings. An error of $1^{o}F$ in the wet bulb temperature changes the moisture concentration by as much as 5 grains water per pound of dry air. The same error in dry bulb readings changes the concentration 1 to 2 grains. The thermometers used were calibrated in $2^{o}F$ increments. Readings were interpolated to the nearest $0.5^{o}F$.

It was concluded that the superoxide particle size in the range of 20 to 150 mesh is not an important variable in the design of the microcontactor for the following reasons:

1. The rate of reaction tests investigated this range of particle sizes and it was found to have little effect on the reaction rate.

2. The grinding experiments discussed in Section Three indicated that the majority of the particles produced with the selected cutting tool were within this range of particle sizes.

It is believed that the resulting particle size is mainly a function of the manufacturing method used in making the high-density block.

SECTION FIVE

MICROCONTACTOR DESIGN

5.1 DESIGN REQUIREMENTS

A one-man capacity unit was selected as the basis for the design of the microcontactor. Since a one-man capacity unit will require a minimum of about 0.3 lb KO_2 per hour, it was felt that as a minimum, this rate of KO_2 consumption should be used in order to work with reasonable quantities of material. The following design parameters were selected:

	Range	Design Point
Temperature	65°F to 80°F	75°F
Relative humidity (at 1 atm)	40% to 60%	50%
O_2 partial pressure	137 to 175 mm Hg	160 mm Hg
Total pressure	0.5 to 1.0 atm	1 atm
CO_2 inlet conc.	0.5 to 1.0%	0.5%
Respiratory Quotient (RQ)	0.6 to 1.1	0.82
CO_2 expired	0.05 to 0.73 lb/hr	0.11 lb/hr
O_2 consumption	0.05 to 0.60 lb/hr	0.10 lb/hr
H_2O loss	0.07 to 0.54 lb/hr	0.18 lb/hr

The microcontactor concept must be capable of operating under a weightless condition.

5.2 MATERIAL BALANCES

5.2.1 Carbon Dioxide Balance

For a one-man capacity, the amount of CO_2 which must be removed from the atmosphere is approximately 0.11 lb/hr. The inlet concentration of the CO_2 and the percent removal as it passes through the KO_2 material are factors which determine the quantity of air which must be forced through the microcontactor. The relationship of these factors is shown in Figure 5-1 for two inlet CO_2 concentrations, 0.5% and 1%.

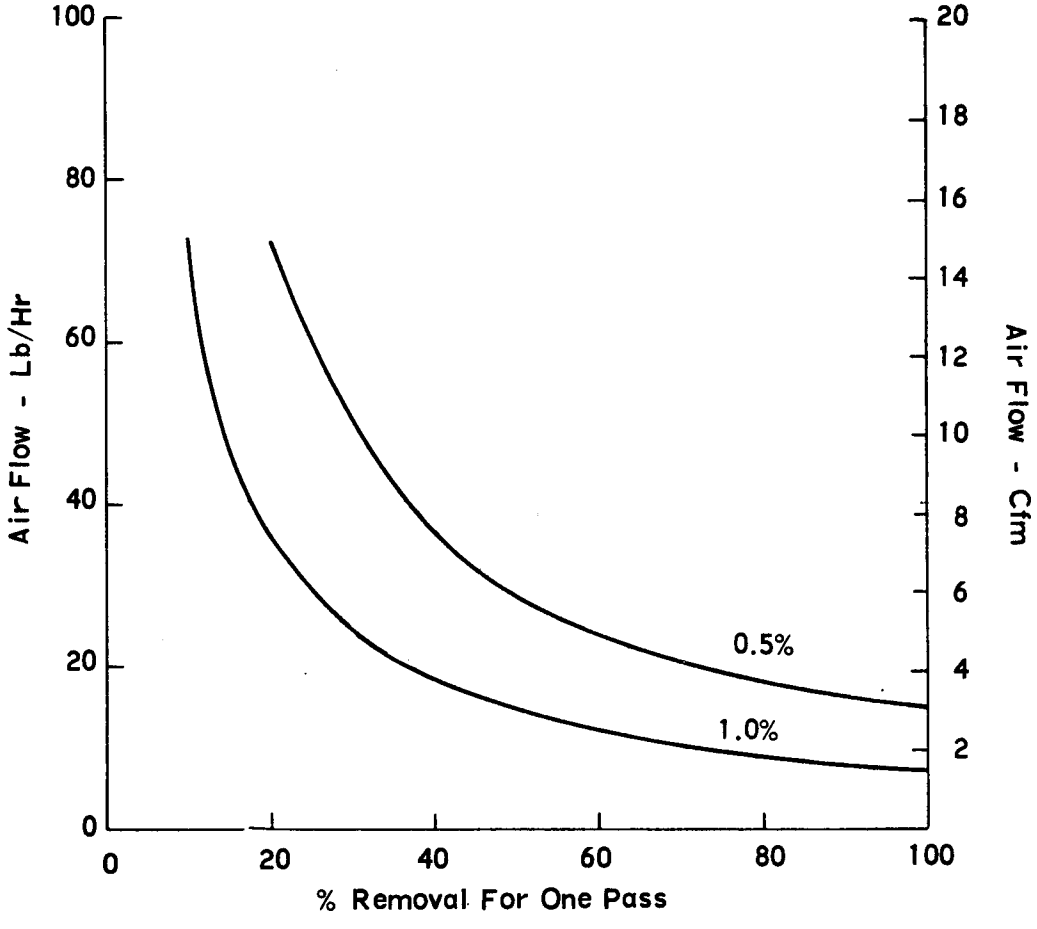


FIGURE 5-1 AIR FLOW REQUIRED TO REMOVE 0.11 LB CO_2/HR WITH INLET CO_2 CONCENTRATIONS OF 0.5 AND 1.0%

Since the 0.5% CO_2 inlet concentration was taken as the design condition, this required higher air flow rates. The percent removal of the carbon dioxide from the air stream is related to the residence time, moisture concentration, and temperature of the air stream.

To remove 0.11 lb/hr of CO_2 requires a minimum of 0.38 lb/hr of the 94% KO_2 material, assuming the reaction goes completely to the carbonate.

As described in paragraph 5.2.2, the oxygen balance would require a minimum of 0.315 lb/hr of the 94% pure KO_2 material. A somewhat larger quantity was required, since the KO_2 reaction was never 100% complete but rather was probably between 80 and 90% complete. This required about 0.4 lb/hr of KO_2. This higher KO_2 rate was sufficient to remove the required quantity of CO_2, especially when a portion of the KOH was converted to $KHCO_3$.

5.2.2 Oxygen Balance

The only oxygen producing KO_2 reaction is:

$$2KO_2 + H_2O \longrightarrow 2\ KOH + 3/2\ O_2$$

To supply the required 0.10 lb O_2/hr, 0.296 lb/hr of pure KO_2 is required. The purity of the available KO_2 is 94%; therefore, 0.315 lb/hr of this material is required. This assumes that all the available oxygen from the KO_2 is released, i.e., there is a complete reaction. Any decrease in oxygen generation efficiency results in a correspondingly higher KO_2 grinding rate. This relationship is shown by Figure 5-2. Since the rate of reaction studies indicated only about 80 to 90% reaction after five to ten minutes, about 0.4 lb KO_2/hr is required.

5.2.3 Water Balance

The system must be capable of removing 0.18 lb/hr of moisture from the atmosphere to maintain the desired environment. Maximum moisture removal requirements can be as high as 0.54 lb/hr depending on the

BASIS: One Man Capacity (0.10 lb O_2/hr)
Available O_2 - 0.34 lb O_2/lb KO_2
KO_2 Purity - 94%

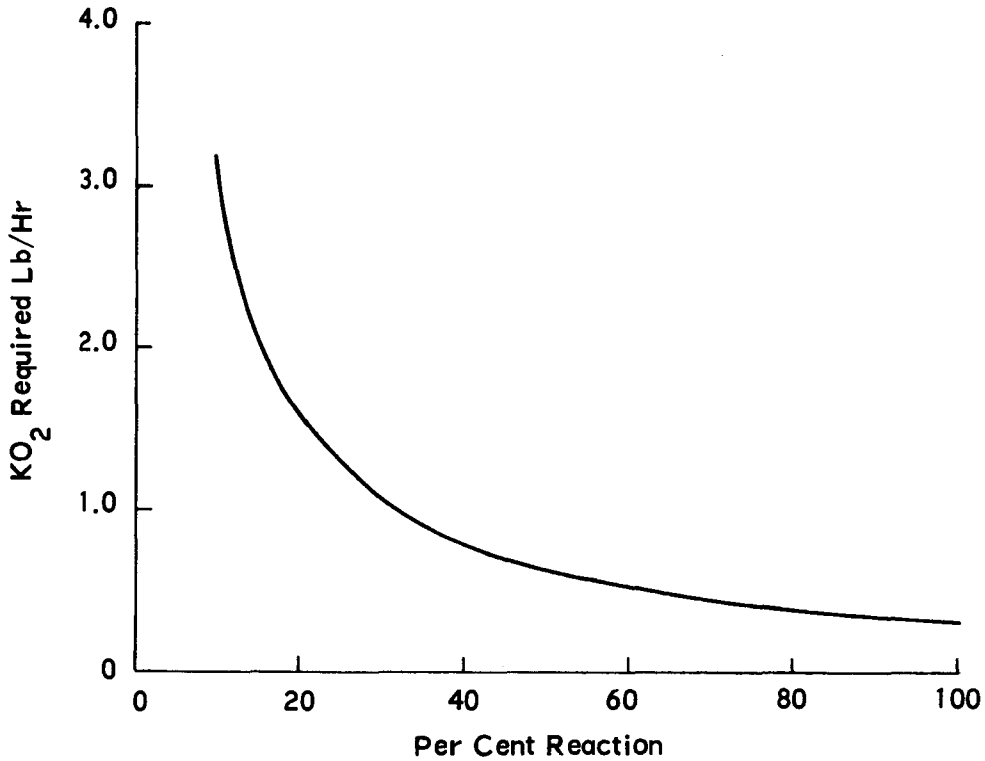


FIGURE 5-2 EFFECT OF KO_2 REACTION EFFICIENCY ON REQUIRED KO_2 GRINDING RATE

activity of the crew members. Within the design parameters, the range of moisture concentration will extend from 37 to 92 grains moisture per pound dry air (40% RH at 65°F to 60% RH at 80°F). The relationship between the inlet moisture concentration, the quantity of water to be removed, and the required air flow is shown in Figure 5-3.

The minimum amount of water required to react with the KO_2 to produce the design figure of 0.10 lb O_2/hr is 0.0375 lb H_2O/hr. The amount of water used in the reaction under actual conditions depends on the amount of hydration which occurs with the reaction products, KOH and K_2CO_3. There undoubtedly is an optimum inlet concentration of moisture, high enough to provide a rapid and fairly reasonable pressure drop through the reaction products. This optimum inlet moisture concentration is not likely to be sufficient to remove completely the moisture introduced into the environment by the crew members. The use of a microcontactor in an integrated life support system would probably require some independent control of the inlet moisture. This could be accomplished along with temperature control by cooling the cabin air in a suitable heat exchanger. During the testing phase of the prototype microcontactor, the inlet moisture concentration will be varied to determine the optimum concentration.

5.3 HIGH-DENSITY KO_2 BLOCK AND GRINDER

For a one-man unit, complete and immediate utilization of the available O_2 from the KO_2 requires about 2.5 lb KO_2 for an 8-hour charge (7.5 lb/day). A 2.5-lb charge of 115 lb/ft^3 density KO_2 would require a volume of 37.5 in.^{3} Table 5-1 shows the resulting heights of 8-hour and 4-hour charges required for the range of charge diameters available from MSA Research Corporation. The maximum height available is about 4 in. Diameters other than those listed or heights greater than 4 in. would require additional tooling costs.

Extrapolating the data from the grinding test results, as shown in Table 3-2, for 7-rpm, unsintered KO_2 gives the grinding rates as shown in Table 5-2. It was assumed that the grinding rate is directly proportional to the grinding area with equal force per length of blade.

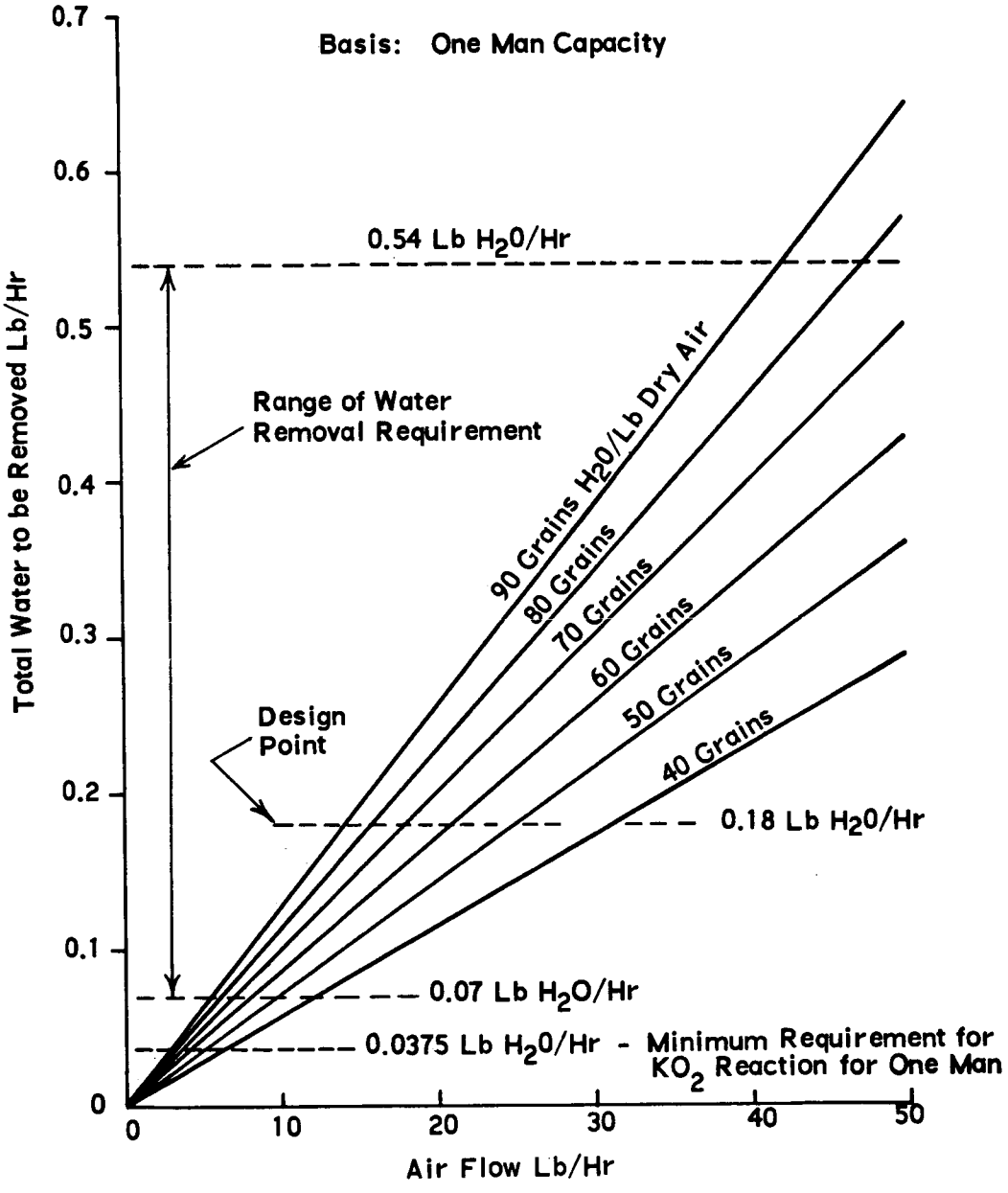


FIGURE 5-3 RELATIONSHIP BETWEEN INLET MOISTURE CONCENTRATIONS, QUANTITY OF WATER TO BE REMOVED AND REQUIRED AIR FLOW

TABLE 5-1

DIMENSIONAL CHARACTERISTICS OF 8-HR AND 4-HR KO_2 CHARGES

Diameter (in.)	Area (in^2)	8-hr Charge Height (in.)	4-hr Charge Height (in.)
1.875	2.77	13.5	6.75
2.0	3.14	12.0	6.0
2.0625	3.35	11.2	5.6
2.625	5.42	6.93	3.47
2.75	5.94	6.32	3.16
3.0	7.08	5.3	2.65
6.0	28.3	1.325	0.662
6.375	32.0	1.172	0.586

TABLE 5-2

EXTRAPOLATED GRINDING RATES 7-RPM UNSINTERED KO_2

Diameter (in.)	Area ($in.^2$)	Area Ratio $\left(\frac{\text{Area}}{2.77\ in.^2}\right)$	Grinding Rate lb/day		
			Force on Blade		
			(1.8 lb/in.)	(3.4 lb/in.)	(4.9 lb/in.)
1.875	2.77	1.0	1.3	2.3	3.7
2.0	3.14	1.13	1.47	2.6	4.18
2.0625	3.35	1.21	1.57	2.78	4.48
2.625	5.42	1.96	2.55	4.5	7.25
2.75	5.94	2.14	2.78	4.92	7.9
3.0	7.08	2.55	3.31	5.86	9.43
6.0	28.3	10.2	13.25	23.5	37.8
6.375	32.0	11.5	15.0	26.5	42.5

A 3-in. diameter KO_2 block with a force of 4.9 lb/in. (of cutting blade face), at 7 rpm gives a grinding rate of about 9.5 lb/day. With this size block and a variable speed grinder, 0-24 rpm, sufficient KO_2 can be produced to support one man. Using four blades with a 1/4-in. hole in the center of the KO_2 block gives a total blade length of 5.5 in. This requires that a total force of 27 lb be exerted on the KO_2 block against the cutting blades. The required starting torque is directly proportional to the total force. From Table 3-3 it can be calculated that the starting torque required for the 3-in. block of KO_2 with a 27-lb force is about 2.0 lb-ft.

5.4 MICROCONTACTOR DESIGN

5.4.1 Microcontactor Requirements

The microcontactor should be capable of providing a total KO_2 particle residence time of 5 to 10 min. with intimate exposure to flowing air in the reaction zone. It must be able to produce about 0.4 lb/hr of fresh KO_2 from a block of the high-density material. From Figure 5-1 it can be seen that an air flow of about 18 lb/hr or 4 cfm will remove the required CO_2 with an inlet concentration of 0.5% and 80% removal efficiency per pass. An air recycle should be provided to permit higher CO_2 removal efficiencies and to provide high-velocity, dry air for removing freshly ground particles from the grinding device with a minimum of sticking.

5.4.2 Microcontactor Concepts

The rate of reaction tests indicated that with a reactor of reasonable size, KO_2 particles suspended in the air stream would not react completely in the short time available. The range of residence times of the KO_2 particles resulting from various reaction zone diameters and heights is shown by Figure 5-4. These residence times are for zero gravity conditions, where the KO_2 particles are swept along by the air stream only and with a total flow of 4 cfm through the microcontactor. Any recycling of the air would result in smaller residence times. Figure 5-4 shows that with a reasonable size reactor, residence

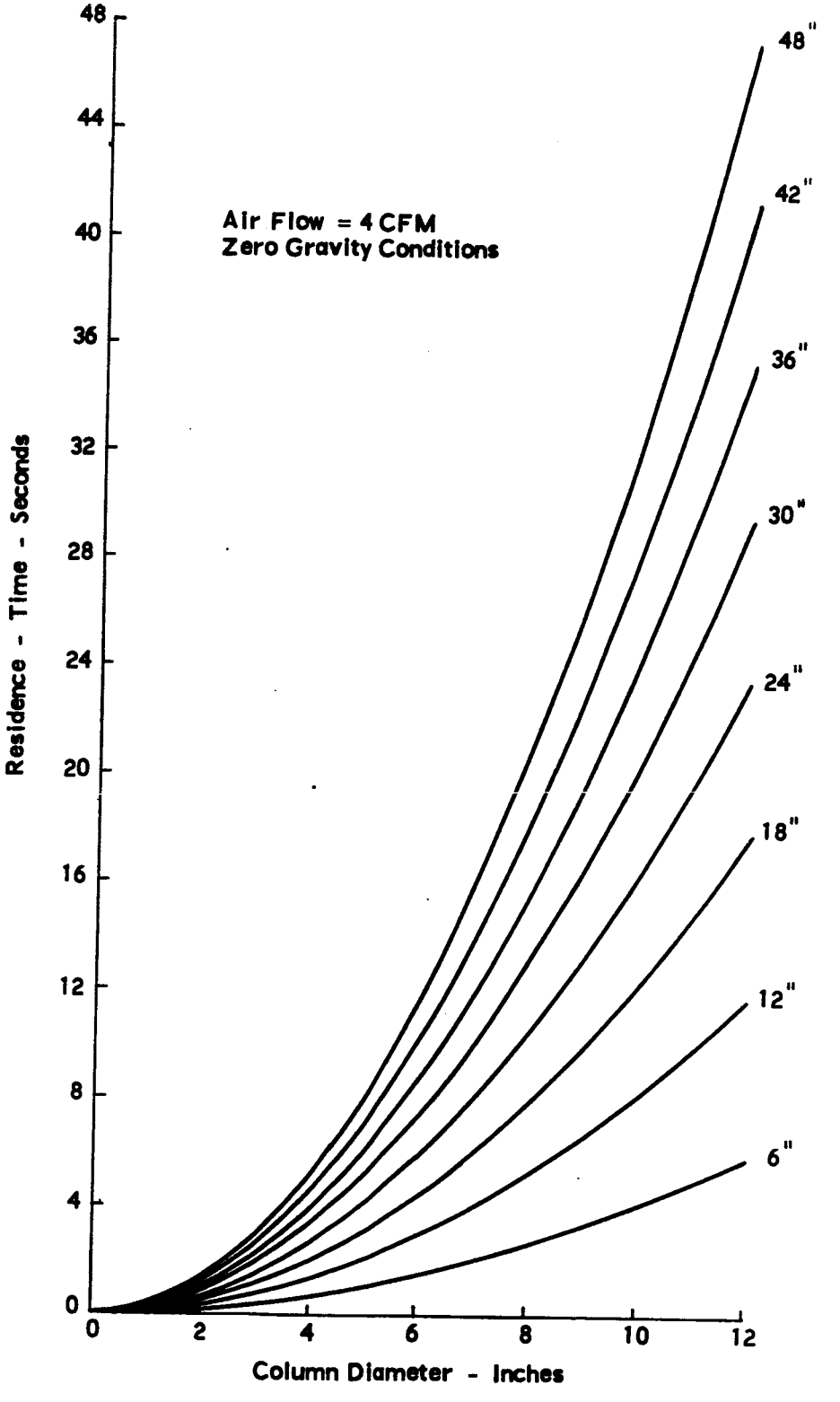


FIGURE 5-4 RESIDENCE TIME FOR VARIOUS COLUMN DIAMETERS AND HEIGHTS

times of more than 20 to 30 seconds are not obtainable. (The results as shown in Figure 4-4 indicate that a residence time of between 5 and 10 min. is required for 80 to 90% reaction). Other approaches were considered in an attempt to design a microcontactor that would produce the required residence time for a nearly complete reaction. These different concepts are discussed briefly below.

1. Fluidization - Fluidization processes are commonly used for gas-solid reactions to assure intimate contact and reasonable pressure drops. This approach would not be feasible for the zero gravity application, since the fluidization concept depends on the presence of gravity.

2. Reaction Zone with Cyclone Separator and Solid Recycle - This method consists of sweeping the air/KO_2 mixture through a reaction zone, and then passing the mixture into a cyclone separator. The KO_2 particles would be recycled into the reactor inlet from the cyclone. This approach would still require excessively large equipment and is considered unfeasible.

3. Filter with Scraper - Another approach considered a reaction zone with a filter at the bottom for collecting the KO_2 and the reaction products.

   When the pressure drop across the filter reached a predetermined value, a scraper would be moved across the filter to push the reacted material into a collection chamber. This method could be used in a batch process for grinding off a fixed quantity of KO_2 and letting it stay on the filter for about 5 min. before scraping it off. This method would overcome the inefficiencies inherent in the continuous scraping of partially reacted material on top of the filter. A disadvantage of this method is that the filter may eventually become plugged with the KO_2 material.

4. Filter Stack with Rotating Scrapers - In an effort to obtain longer KO_2 residence times within the microcontactor, consideration was given to stacking filters in the reaction zone. An opening is provided in each filter, spaced so that all the openings form a spiral flow path down through the reaction zone. A common shaft through the filters would rotate a scraper over each filter in such a way that the KO_2 material would be deposited on each filter and then scraped off and passed through the opening to the next filter. This process appears to have air flow and material distribution problems under zero gravity conditions. Also, it is mechanically complex and does not appear feasible for the small quantity of material to be processed.

5. Reversible-Filter Stack - This method features a series of screens or filters placed in the reaction zone on swivel pins similar to a butterfly-valve arrangement. At some fixed time increment, the filters would be turned over in sequence starting from the bottom. This would result in a self-cleaning action on the filters as the direction of flow through the filter was periodically reversed. A bed depth of about 0.08 in. on each filter would result, with a KO_2 grinding rate of 0.4 lb/hr, using 4-in. diameter filters at a 5-min. hold-up time in the stack for 1 gm/cc bulk density material. A 6-in. diameter filter reduces the bed depth to about 0.035 in. With a recycle ratio of 4 and an inlet flow of 4 cfm, the pressure drop across each 6 in. filter is estimated to be about 2 to 3 in. water. A blower with a 60% efficiency would require about 17 watts for these conditions with two filters in series and 35 watts with four filters in series. The reversible filter stack appears to be the most feasible of the methods investigated.

5.4.3 Solid Products Collection Assembly

In conjunction with the reversible-filter stack described, a solid products collection device is required to remove the solid products from the air stream after these products have been removed from the last filter. One method considered was to have the reaction products impinge at the bottom of the reactor chamber. This would be accomplished by using a nozzle to increase the air velocity after passing the last filter, then introducing a sharp bend to the air exit line immediately downstream of the nozzle. The solid products, due to their high inertia, would be unable to make this sharp turn and would impinge on the bottom of the reaction chamber. A final filter would be installed in the air exit line to remove any stray particles. A questionable aspect of this method is the separation efficiency that could be obtained under zero gravity conditions.

Another method to separate the solid reaction products would be to use a cyclone on the discharge of the microcontactor. Again the air velocity would be increased by funneling it through a small cross-section exit line. The air would then enter the cyclindrical cyclone chamber tangentially and would leave through a central opening. The solid particles, by virtue of their inertia, would tend to move toward the outside cyclone wall. This method of gas-solid separation is often used commercially and the centrifugal separating force may range from five times gravity in very large-diameter, low-resistance cyclones, to 2500 times gravity in very small, high-resistance units. This cyclone approach appears to be the most feasible for the present application.

5.5 PROTOTYPE MICROCONTACTOR DESCRIPTION

A reversible filter microcontactor was designed and fabricated to meet the previously discussed design requirements. The prototype microcontactor consists of a grinding chamber, a filter chamber, and a solid products collection assembly. Each of the above subassemblies was constructed of 1/2-in. thick, clear plexiglass, which allowed visual observations to be made while the unit was operating. Figure 5-5 is a schematic of the KO_2 microcontactor and Figure 5-6 is a photograph of the microcontactor.

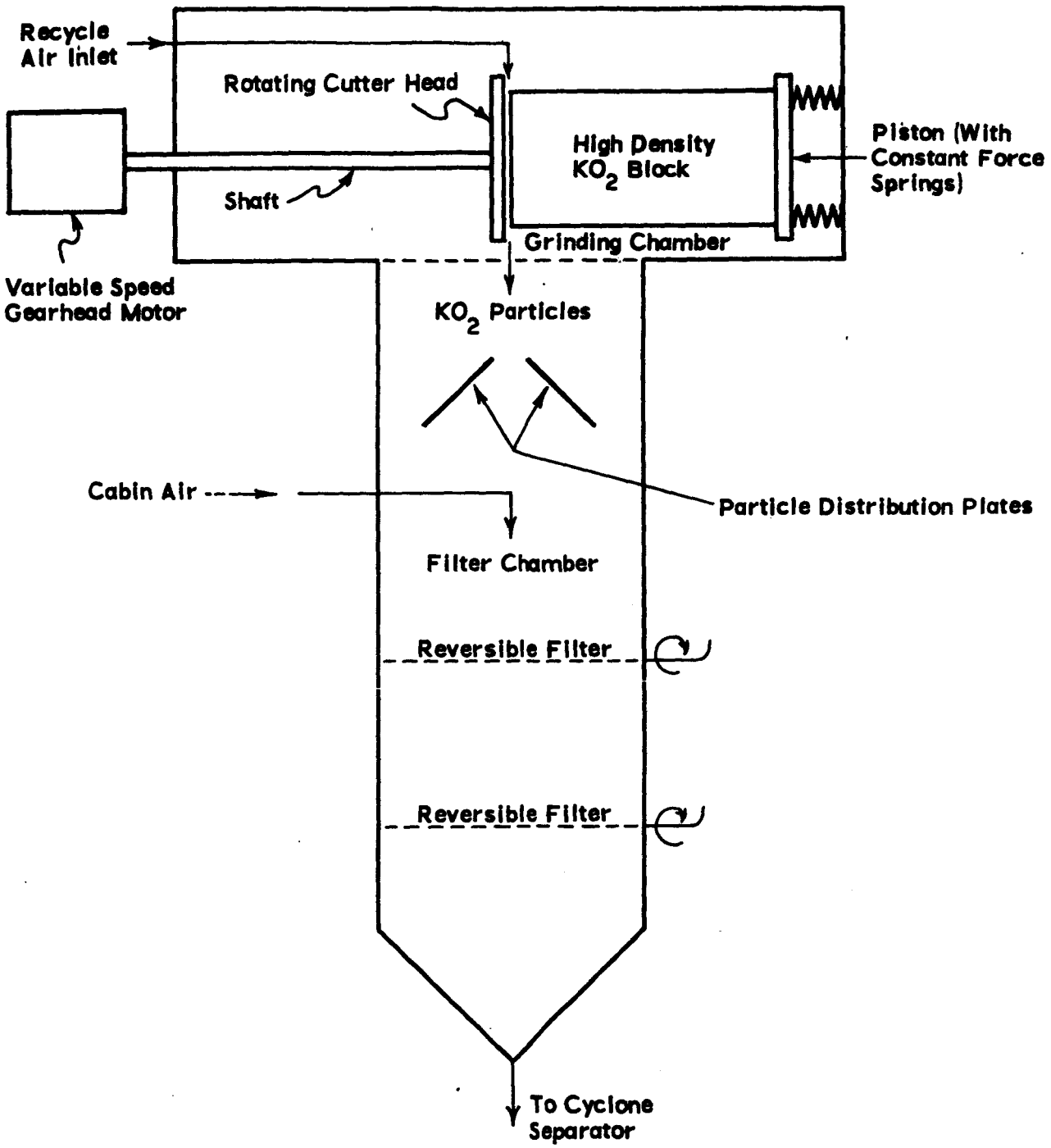


FIGURE 5-5 SCHEMATIC OF KO_2 MICROCONTACTOR

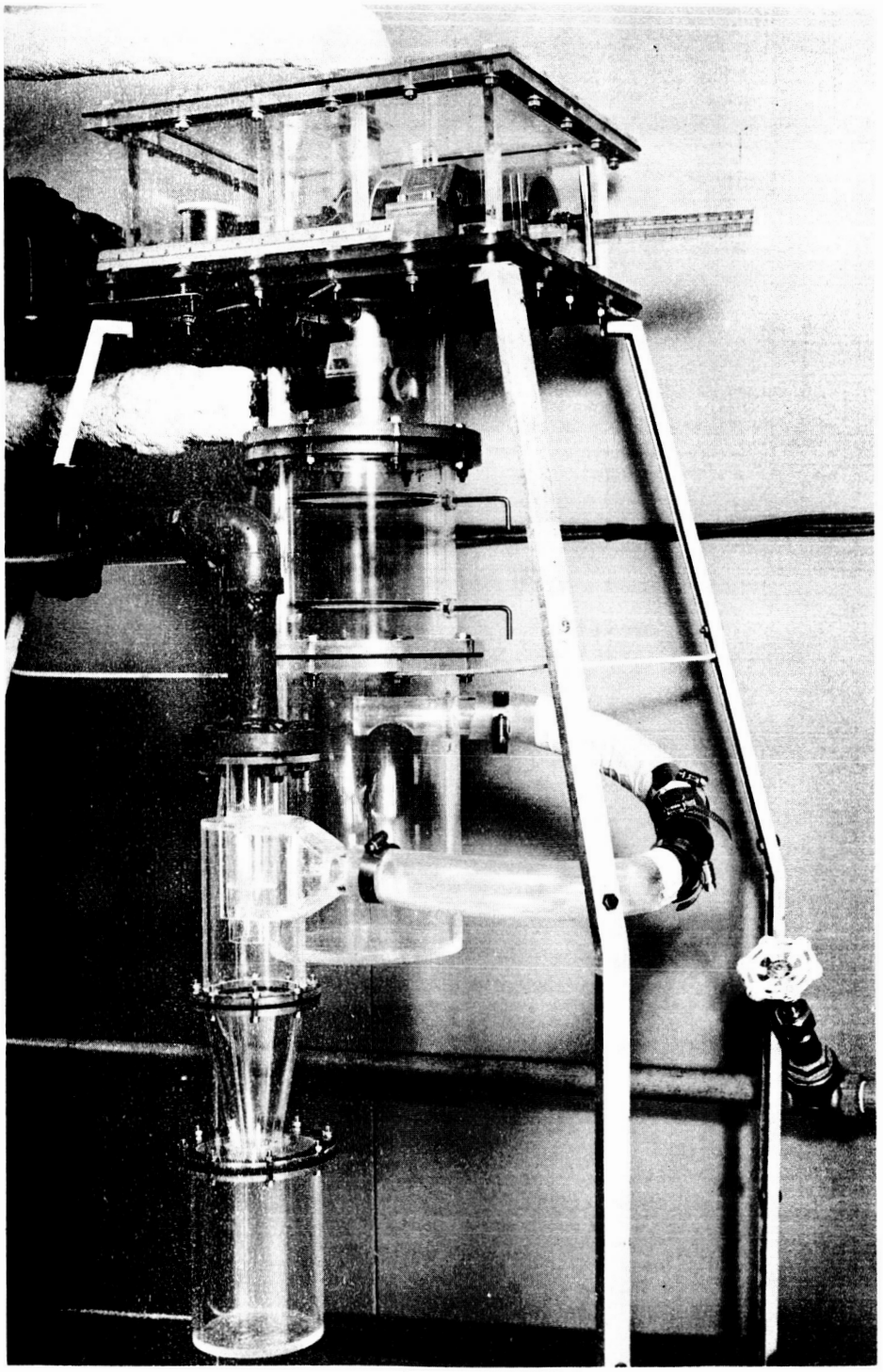


FIGURE 5-6 OVERALL VIEW OF MICROCONTACTOR

The high-density KO_2 blocks were purchased in a cylindrical shape, 6 in. long with a diameter of 3.070 in. ± 0.10 in. A 1/4-in. center-hole ran the length of the block to eliminate the near-zero cutter blade velocity at the center. The KO_2 block was held in the grinding chamber by the cartridge holder fabricated from 316 stainless steel. Two 16.5 lb stainless steel Neg'ator springs acting on a piston maintain the required force between the KO_2 block and the cutter face. The piston is fitted with two pins which engage two holes on the KO_2 block. This prevents the block from rotating with the cutter within its cartridge holder. A handle passing through the side of the grinding chamber is threaded into the piston. The handle is used to retract the piston when loading or unloading the KO_2 block into the cartridge holder. The Neg'ator springs are wound on Teflon spools supported on each side of the shaft. Figures 5-7 and 5-8 are top views of the grinding chamber with the top removed. In Figure 5-8 the cartridge holder cover is removed and the piston is partially retracted.

The cutting tool is driven through a flexible coupling by a d-c shunt motor which is supported on the side of the microcontactor. The 1/15 hp, 115-VDC motor is speed controlled in the range of 0 to 24 rpm and has a torque rating of 88 in.-lb. The shaft passes through a ball bearing mounted in the grinder chamber wall and is supported by a second ball bearing inside the grinding chamber. The end of the shaft is fastened to the 3-3/8 in. diameter stainless steel cutter head which holds the four tungsten carbide cutting blades placed radially and spaced 90° apart. A plexiglass shroud encloses the cutting head and face of the KO_2 block to prevent particle scattering and help channel the freshly ground KO_2 down to the filter chamber. The recycle air inlet is on the cover of the grinding chamber and is positioned directly over the cutting tool when the cover is in place. The grinding chamber is rectangular and is connected to the top of the cylindrical shaped filter chamber.

The filter chamber has a 6-in. ID with a 1/2-in. thick wall and contains two disc filters. The filter material is Teflon FEP mono-filament fabric with 171 x 104 threads to the inch. The Teflon fabric is bonded

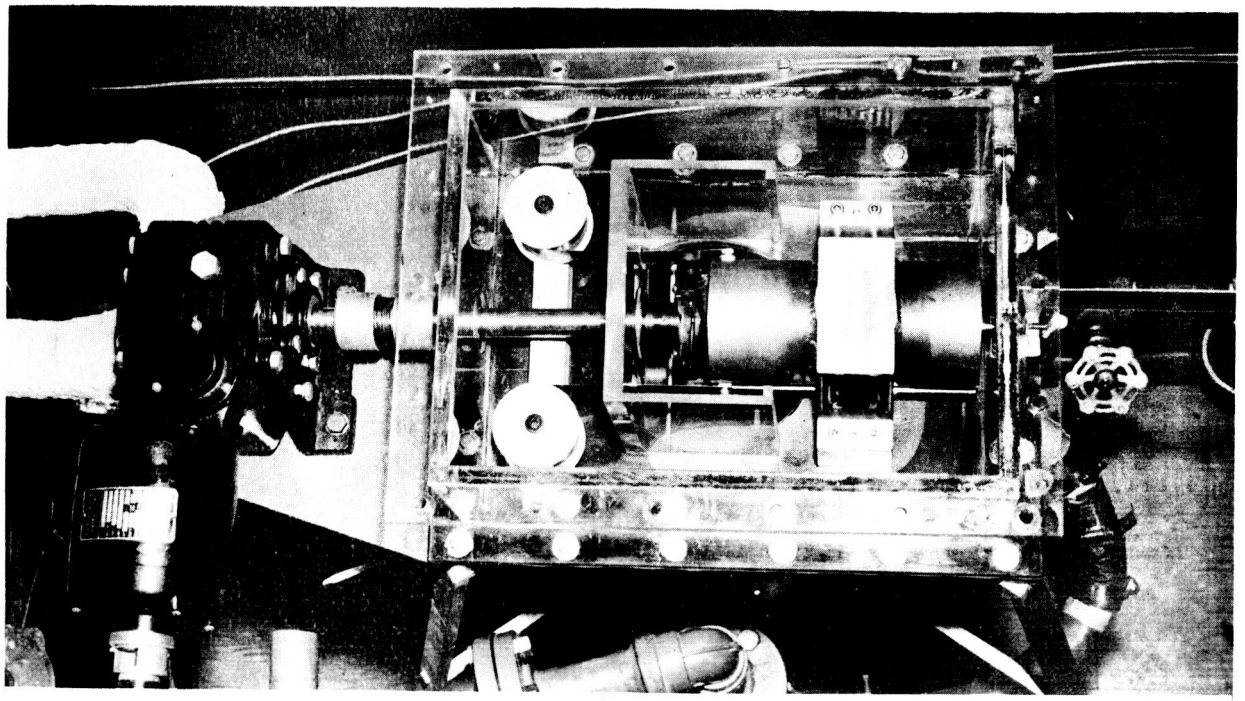


FIGURE 5-7 TOP VIEW OF GRINDING CHAMBER WITH TOP REMOVED

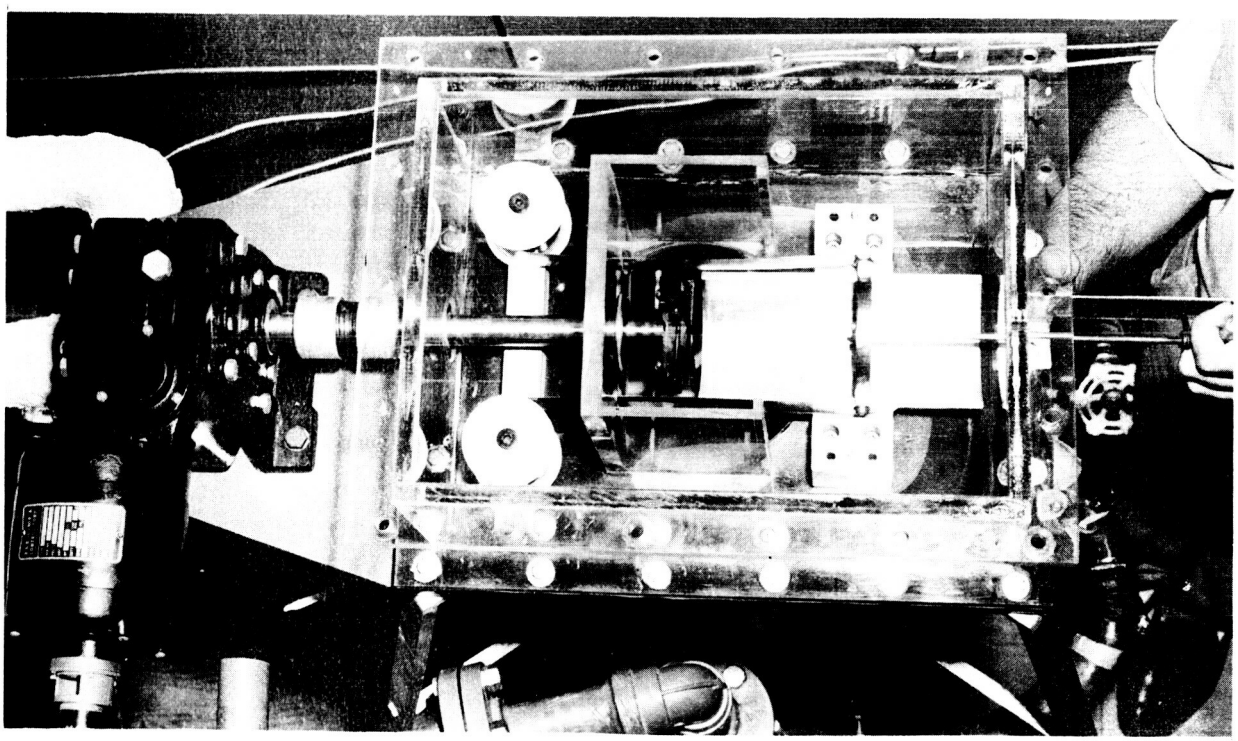


FIGURE 5-8 TOP VIEW OF GRINDING CHAMBER WITH TOP REMOVED, CARTRIDGE HOLDER COVER REMOVED AND PISTON PARTIALLY RETRACTED

between two, thin nickel-copper-aluminum (K-monel) rings making an integral subassembly which is held between two Teflon-coated, stainless steel rings. The stainless steel rings are machined to accept an o-ring at their outer circumference when fastened together. This o-ring serves as a seal to minimize particle blow-by. The complete filter assembly is attached by two screws to the filter handle which penetrates the plexiglass wall through an o-ring seal.

The solid products collection assembly is attached to the filter chamber and consists of a 6-in. diameter, 11-in. deep collection chamber. The air exit line leaves the side of this collection chamber near the top and tangentially enters the cyclone separator. A smaller collection chamber is attached to the bottom of the cyclone separator to collect the fine solid products and facilitate periodic cleaning of the unit.

SECTION SIX

DESCRIPTION OF TEST SYSTEM

The purpose of the test program was to verify the capability of the microcontactor to operate effectively over the range of design conditions. In order to perform the necessary tests it was essential to be able to vary and control the inlet air conditions and to be able to measure inlet and outlet air compositions.

6.1 AUXILIARY TEST APPARATUS

Figure 6-1 shows the process flow sheet for the KO_2 microcontactor test arrangement. A 200 VAC, 3-phase, 400-cps centrifugal fan took ambient air on the suction side and delivered the air under pressure to the test system. For humidifying the air, a steam generator consisting of a copper boiler on a hot plate delivered steam to the inlet air stream as required. A variac was used to control the rate at which the steam was generated.

For dehumidifying, a 35°F chill water source was maintained with a 1/4-ton refrigeration unit with the cooling coil placed in a 35-gallon drum about half full of water. A 4-gpm circulating pump was used to supply the chill water to the dehumidifying coil and return to the chill water source. The finned dehumidifying coil was capable of cooling the inlet air to a 40°F dew point. The inlet air then entered a 1000-watt air heater with a thermostat control.

The carbon dioxide concentration of the inlet air was controlled downstream of the air heater by introducing 100% CO_2 at a constant flow rate with an adjustable flow controller.

The inlet air then passed through a flow meter and valving into the microcontactor. A three-port, two-way ball valve (V-5) was used to direct the inlet air to enter the microcontactor at one of two entrances. One was at the top over the cutter blades (for use if no recycle was used) and the other was through the side just above the top filter (for use if a recycle was used).

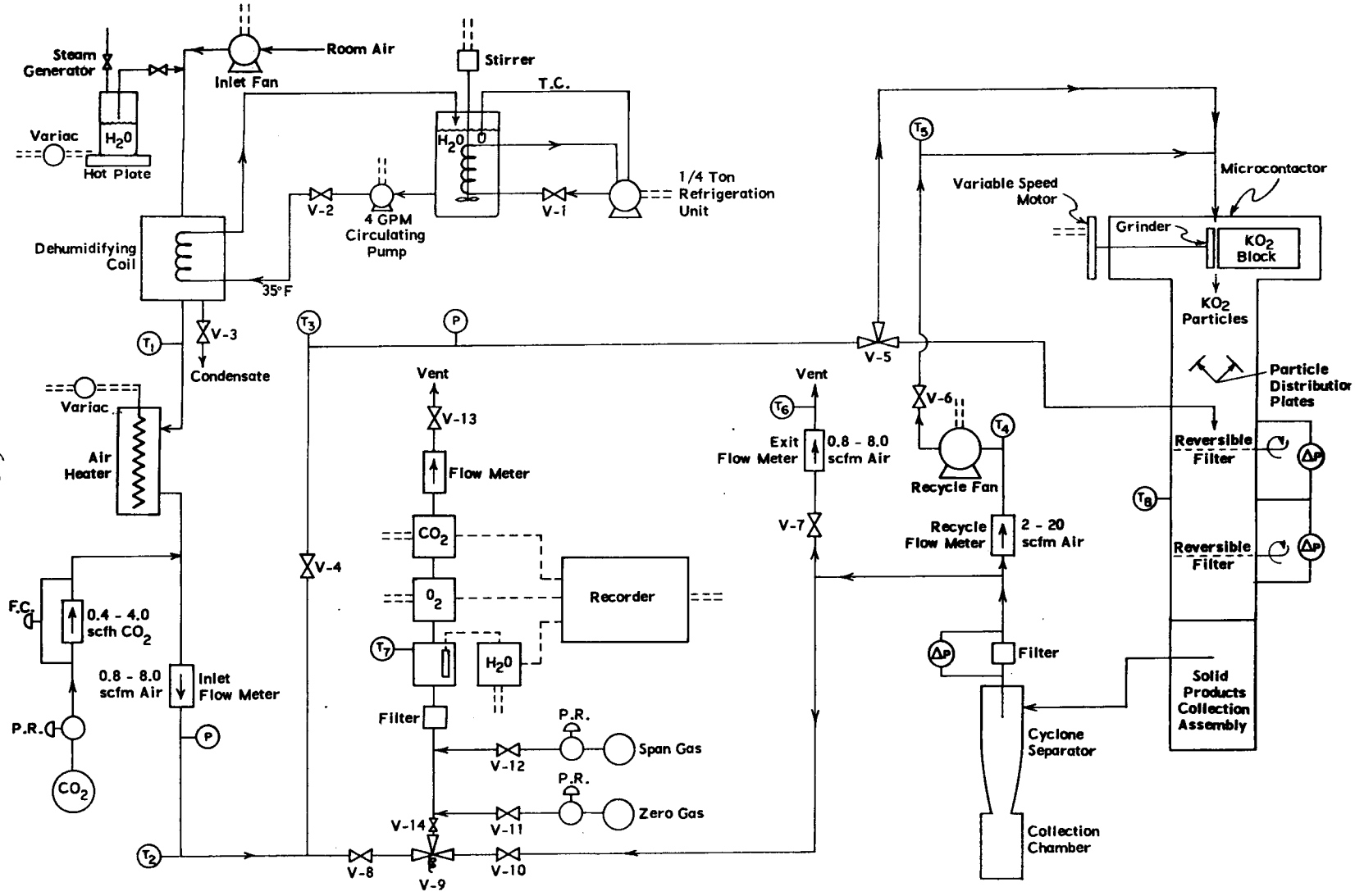


FIGURE 6-1 PROCESS FLOW SHEET KO_2 MICROCONTACTOR TEST ARRANGEMENT

Immediately downstream of the cyclone separator a 400-mesh screen filter was installed to collect any solid product which passed through the cyclone separator. Downstream of the filter, provisions were made to recycle the air, if required, through the recycle fan which was identical to the inlet fan. The exit air passed through a flow meter and was then vented. All air piping was 1-in., nominal, PVC pipe except for the recycle circuit which was 1-1/4 in. PVC. Figure 6-2 is an overall view of the test apparatus and instrumentation.

6.2 INSTRUMENTATION AND CONTROLS

Instrumentation and controls were provided for automatic and manual control of process parameters. In addition, certain indicators and recorders were provided for retrieval of the data for subsequent analysis. Brief descriptions of the instrumentation and control features are given in the following paragraphs.

6.2.1 Power Supply

The test facility required 115-V, 60-cycle, single-phase, a-c power for the operation of the 1/15-hp grinding motor control system. Three-phase, four-wire, 208-V, 400-cycle power is required for the operation of the feed and recycle fans. Both power supplies passed through a small operating panel, where permissive switches, signal lamps, and fuses were provided. Separate 60-cycle power lines go to the auxiliary gas analyzers and the recorder.

6.2.2 Grinding Motor Speed Control

A speed control is provided for adjusting and maintaining the speed of the 1/15-hp, 115-V, d-c motor which drives the KO_2 grinding tool. A 10-turn potentiometer receives a regulated d c voltage and is set for the desired speed in the range of 0 to 24 rpm. The resultant d-c wiper voltage is compared to a feedback voltage from a motor driven tachometer. The difference is amplified in a magnetic amplifier which, in turn, drives a full-wave, rectified, SCR controller connected to the motor.

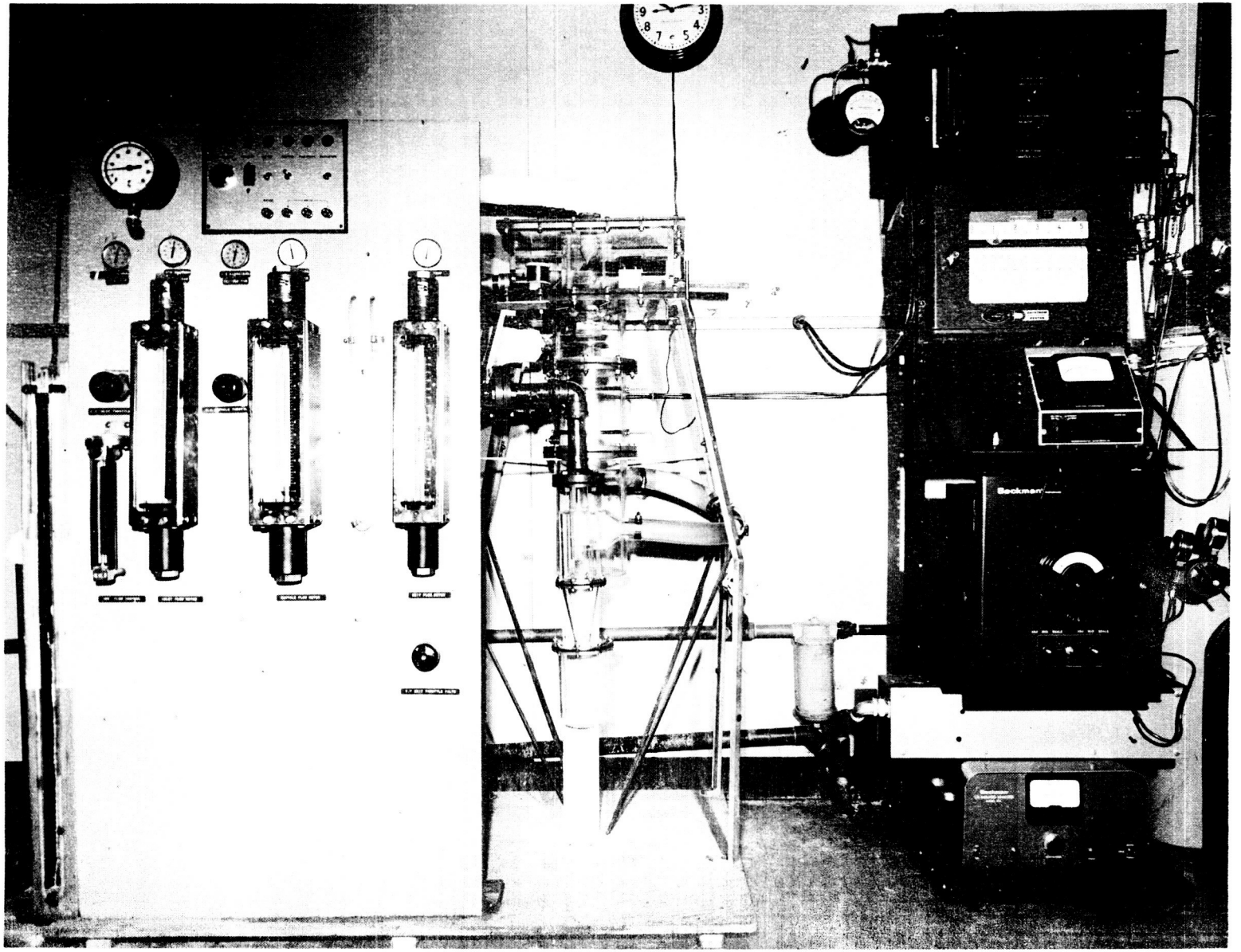


FIGURE 6-2 OVERALL VIEW OF TEST APPARATUS

6.2.3 Temperature

Bimetallic temperature sensing dial indicators were installed in the flow lines to determine the temperature distributions. A glass, mercury-filled thermometer was used to measure the air temperature in the filter chamber between the two filters.

6.2.4 Pressure

A bourdon tube pressure gage and water manometers were installed at various points to determine system pressure and pressure differentials.

6.3 GAS ANALYSIS

Provisions were made for sampling the air stream before and after entering the microcontactor. A solenoid valve (V-9) was used to select the sampling point.

6.3.1 Water (Humidity) Analyzer

An electric hygrometer indicator was used to indicate relative humidity. The sensing element and thermometer were placed in a chamber in the gas sampling line with a cable connection to the indicator. The sensing element has a calibration accuracy of ± 1.5% RH.

6.3.2 Oxygen Analyzer

A Beckman Model F3 paramagnetic oxygen analyzer was used to measure the inlet and outlet oxygen concentration. The range of the instrument was modified to 20 to 25% for this application. The analyzer accuracy is ±1.0% of full scale.

6.3.3 Carbon Dioxide Analyzer

A Beckman Model 15A L/B Infrared Analyzer was used to measure the inlet and outlet concentrations of carbon dioxide. The range of the instrument was 0 to 2%. The analyzer has a sensitivity of 0.5% of full scale and an accuracy of ±1.0% of full scale.

6.3.4 Recorder

The output from the three gas analyzers was fed into a Daystrom Weston recorder with a chart speed of 1 foot per hour. The recorder printed about every 15 seconds.

SECTION SEVEN

PRELIMINARY TESTS

7.1 GENERAL

Various static and dynamic preliminary tests were performed on the complete test system prior to any operation with the high-density KO_2 blocks.

The complete system was pressure tested at 33 in. H_2O. All detectable leaks were repaired and a static pressure change of 1 in. H_2O over a 15-min. period was obtained before test runs were initiated.

The response times of the gas analyzers to gas concentration changes in the filter chamber were determined at an air flow of 4 cfm and a gas sample flow of 0.5 scfh from the outlet sample line. The response times were determined by introducing pure O_2, CO_2, and steam through a pressure tap on the filter chamber. The following response times were obtained:

	Meter Start-to-Move	Meter Balanced
O_2	60 (sec.)	136.3 (sec.)
CO_2	40 (sec.)	66.3 (sec.)
H_2O	62 (sec.)	200.8 (sec.)

Preliminary runs were made using an epoxy/talc block in place of the high-density KO_2 block to check out the grinder and the mechanical performance of the system. It was necessary to shim some of the cutter blades to obtain essentially uniform cutting by all blades and to prevent one blade from cutting too deeply and binding the cutting tool on the block. After adjusting the cutting blades, the grinder operated very effectively with the epoxy/talc product being removed like strips of crepe paper.

7.2 GRINDING RATE AS A FUNCTION OF MOTOR SPEED

The grinder speed as a function of the input setting was determined under zero load conditions. Figure 7-1 shows these results and indicates good speed control from 0 to 24 rpm. It also shows that after removing part of the cutter head, as described later, the speed of the cutter increased at a given setting due to the smaller mass.

Five and 10-minute runs were made at various grinder speeds; the amount of KO_2 ground was determined by weighing the block, in its container, before and after each run. The length of the block was also measured before and after each run. Table 7-1 summarizes the results of these runs and Figure 7-2 illustrates grinding rate as a function of grinder speed. The last column of Table 7-1 indicates a variation of density within each block of KO_2. The average measured motor speed as shown in Table 7-1 agrees very closely with the speeds obtained under zero load at the same potentiometer setting as shown in Figure 7-1.

7.3 GROUND PRODUCT SIZE

Samples of the KO_2 ground product of block no. 4 were taken at various grinding speeds during the grinding rate studies. A screen analysis was made on the four samples take. The results are shown in Table 7-2.

Figure 7-3 shows a high-density KO_2 block and a sample of the grinder product.

7.4 INDENTATION HARDNESS TEST

An indentation hardness test was made on six high-density KO_2 blocks according to ASTM specification D 1706. The tests were performed in a dry box using a Shore Type D Durometer. Five durometer readings were made on each end of the KO_2 blocks. The Type D durometer is calibrated to read 100 when pressed on a piece of flat plate glass. Other reference values for this hardness test are values of 80 to 95 for hard rubber samples. The metal screw cap from the KO_2 block container produced a reading of 72. The KO_2 block hardness test results are shown in Table 7-3.

TABLE 7-1

GRINDING RATES

Block No.	Motor Speed Setting	Ave Measured Motor Speed (RPM)	Length of Run (Min)	Weight KO_2 Ground (Grams)	Length KO_2 Ground (inch)	Ave Grinding Rate (Gm/min)	Ave Density (lb/ft^3)
2	50	0.64	10	12	0.068	1.2	90.8
2	100	2.8	5	25	0.134	5.0	96.2
2	100	2.8	5	24	0.137	4.8	90.3
2	150	5.0	5	41	0.240	8.2	88.1
2	150	5.0	5	47	0.279	9.4	86.8
4	100	3.0	10	16	---	1.6	---
4	100	2.9	10	23	---	2.3	---
4	100	2.9	10	21	---	2.1	---
4	200	7.3	10	51	---	5.1	---
4	200	7.4	10	49	---	4.9	---
4	200	7.5	10	39	0.203	3.9	99.0
4	200	7.5	10	32	0.172	3.2	95.8
4	200	7.5	10	30	0.172	3.0	89.8
4	300	11.6	10	47	0.250	4.7	96.8
4	300	11.8	10	37	0.172	3.7	111.0
4	400	16.0	10	39	0.203	3.9	99.0
5	150	4.9	15	62	0.345	4.1	92.6
5	150	4.9	10	47	0.220	4.7	110.0
5	150	4.9	10	44	0.230	4.4	98.6
9	100	2.8	5	14	0.065	2.8	111.2
9	100	2.8	5	15	0.058	3.0	133.3
9	115	3.5	5	22	0.117	4.4	97.2
9	125	3.8	5	27	0.110	5.4	126.7

TABLE 7-2

SUMMARY OF SCREEN ANALYSIS
(Size Distribution in Percent by Weight of Product)

Mesh Size	2.9 (RPM)	7.4 (RPM)	11.7 (RPM)	16.0 (RPM)
-20 +40	1.9	4.0	1.5	1.2
-40 +60	5.4	8.9	4.0	2.1
-60 +80	11.6	11.7	4.5	4.8
-80 +100	15.0	7.1	2.9	6.3
-100 +150	51.9	28.6	15.7	15.8
-150 +200	11.5	20.0	31.2	30.4
-200	2.7	19.7	40.2	39.4

TABLE 7-3

INDENTATION HARDNESS TEST RESULTS
(Average of Five Readings)

Block No.	Front Face	Back Face
2	69	73
4	75	74
5	76	74
9	72	65
10	78	72
11	75	75

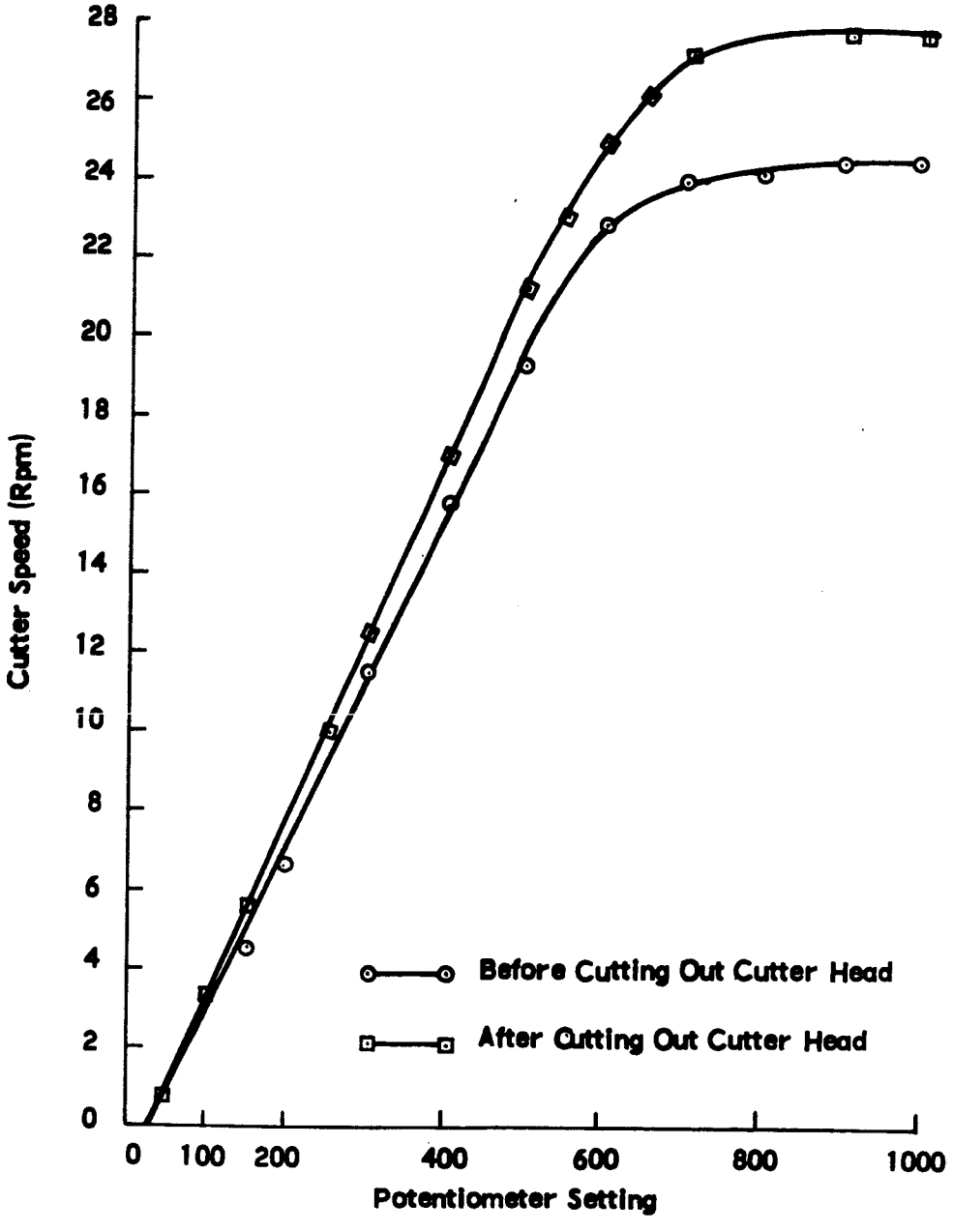


FIGURE 7-1 CUTTER SPEED AS FUNCTION OF POTENTIOMETER SETTING

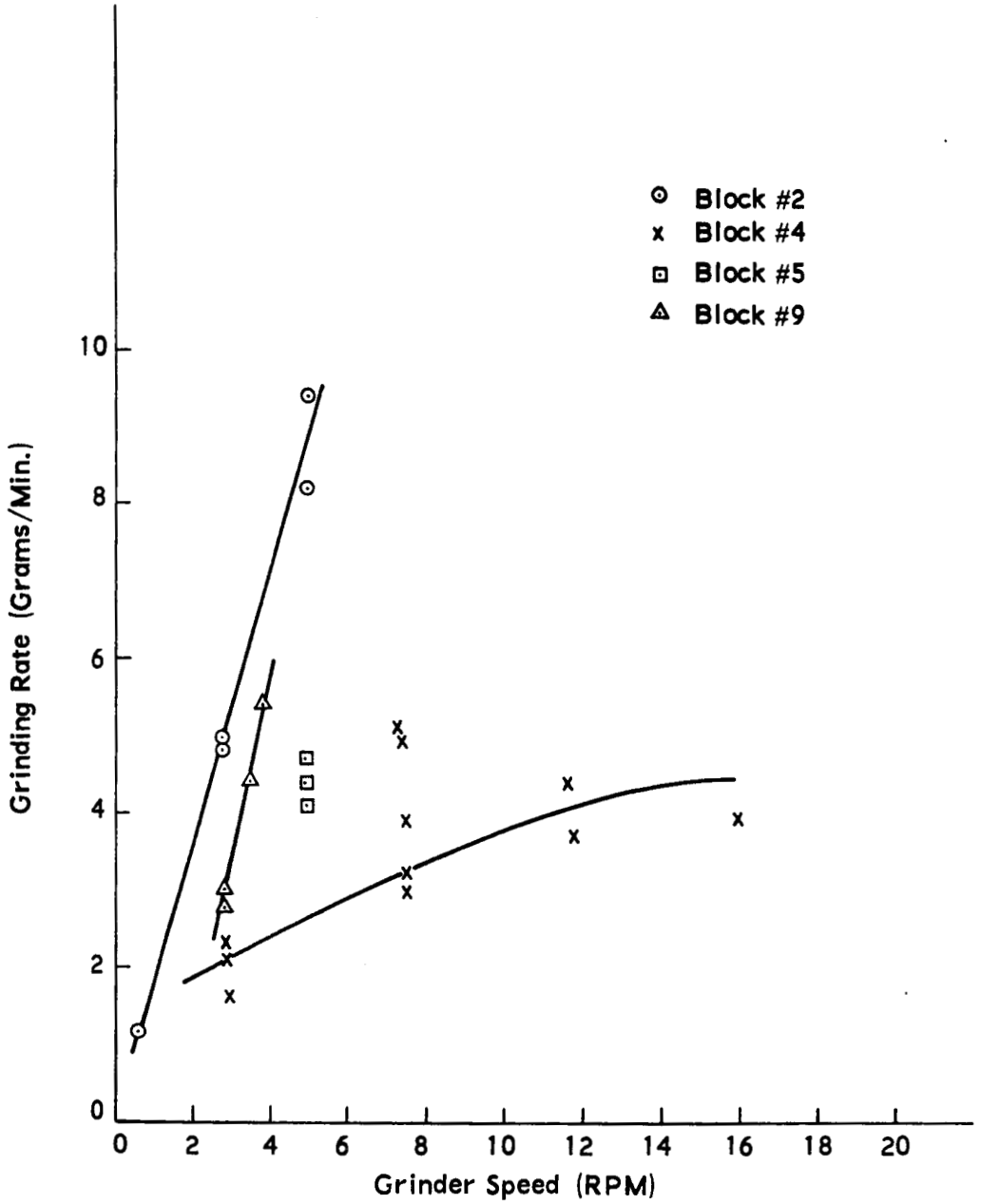


FIGURE 7-2 KO_2 GRINDING RATE AS FUNCTION OF GRINDER SPEED

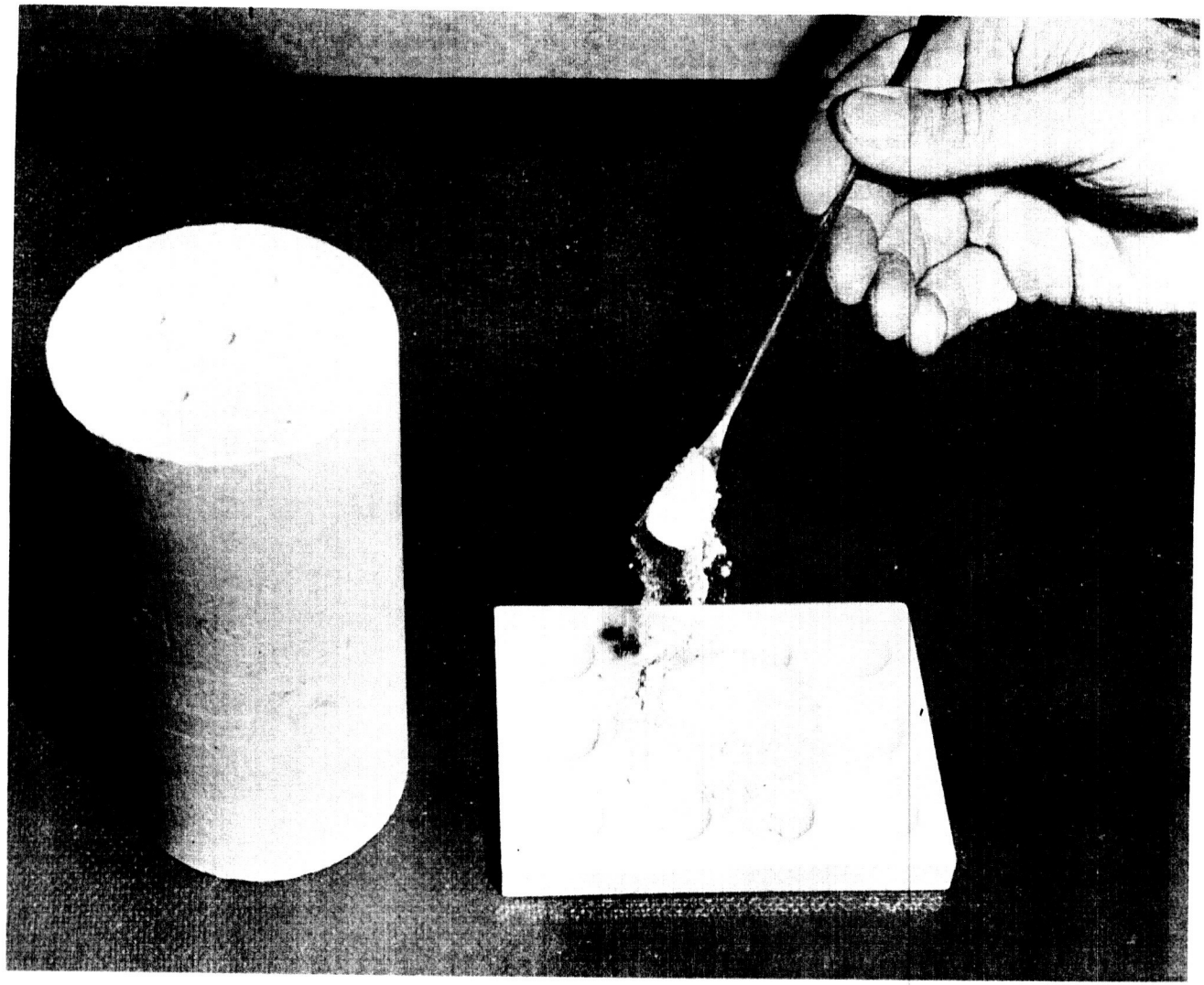


FIGURE 7-3 HIGH-DENSITY KO_2 BLOCK AND SAMPLE OF GRINDING PRODUCT. (END OF BLOCK SHOWS 1/4 CENTER HOLE, 2 PISTON PIN HOLES, AND A SAMPLING HOLE.)

7.5 PRELIMINARY KO_2 RUNS

Several preliminary runs were made with the complete microcontactor system and the high-density KO_2 blocks. These preliminary runs resulted in the following modifications being made:

1. A fan-shaped nozzle was placed on the end of the air inlet pipe, directly over the cutter head, this increased the inlet air velocity forcing the freshly ground KO_2 particles off of the cutter head and cutting blades.

2. The transition piece between the bottom of the microcontactor and the cyclone separator was enlarged to 1-1/4 in. ID in an attempt to eliminate a plugging problem. The solid products still tended to plug in this new transition piece. The solid product collection assembly was then modified to include a 6-in. ID collection chamber for the large size, solid, reaction products just below the filter chamber. The air exit line extended from the side of this collection chamber near the top and continued into the cyclone separator. This modification eliminated the plugging problem.

3. The two distribution plates in the filter chamber were removed since the KO_2 particles were sticking to these plates and piling up even though the plates had been coated with Teflon.

4. Pie-shaped sections of the cutter head were cut away between the four cutting blades in order to facilitate the removal of the freshly ground KO_2 by the air stream.

5. A binding problem in the KO_2 cartridge holder was reduced by reworking and smoothing the finish of all bearing surface of the cartridge holder and the piston. The two Neg'ator springs were also placed in better alignment with the piston.

7.6 COATING OF KO_2

A preliminary investigation of various coatings was performed in order to determine the feasibility of coating the high-density KO_2 blocks with a thin, water-impermeable film to reduce or eliminate any reaction of the KO_2 block which may cause the block to become wedged in the cartridge holder. The following coatings were tested:

1. Phenolic varnish (Mil-V-1174A Formula #80)
2. Tygon corrosion resistant coating (TP-210 Blue)
3. "Krylon" spray paint
4. Melted Polyethylene (DuPont Low Melt AC Pellets)
5. Devron Epoxy (Formula #215)
6. Epoxy (Epi Reg 50A Resin + DETA 100/6)
7. Epoxy (Epi Reg 50A Resin + EpiCure 855 100/30)
8. Teflon Spray (Rulon Spray Lubricant)
9. "Ram" mold release agent (GS-3)

Small test pellets of KO_2 were made with a pellet maker in the dry box. The various coatings were brushed or sprayed onto the pellets. In the case of the polyethylene, the pellet was dipped in a beaker of melted polyethylene. After allowing the coating to dry, the coated pellets, along with a control pellet, were removed from the dry box and exposed to ambient conditions. The only satisfactory coatings were the phenolic varnish and the polyethylene. The disadvantage of the polyethelene is the difficulty of applying it to the KO_2. It must be in a liquid state to apply but it solidifies very quickly, resulting in an uneven coating.

7.7 DISCUSSION OF PRELIMINARY TEST RESULTS

7.7.1 Grinding Rate as a Function of Motor Speed

Results of the preliminary grinding rate tests as shown on Table 7-1 indicated a variation of density within each block of KO_2 tested. The blocks were manufactured by loading the 15-in. mold only twice and then compressing to the desired 6-in. length. This resulted in a considerable pressure drop over the full length of the block while

it was in the mold, which would produce various densities of material over the length of the block.

A modification of the molding procedure could possibly eliminate or minimize the variation in density. If a smaller load of starting KO_2 material were placed in the mold, e.g., about 100 grams, and that amounts were compressed to the maximum density, then a second load of 100 grams would be loaded in the mold and this amount would be compressed to the maximum density. Small loads would be added continually until the full loading of about 1200 grams had been compressed. This procedure would result in a more uniform density over the full length of the block. It would also probably result in a higher density for the whole block, since the pressure drop would be minimal with each small loading.

7.7.2 Grinder Product Size

The summary of the screening analysis (Table 7-2) shows that as the grinding speed is increased, a higher percentage of the product is reduced to fines (-200 mesh). At the higher speeds, more of the grinding energy is expended in particle size reduction rather than in just the cutting action. The distribution with the slow speed (2.9 rpm) is rather symmetrical, with the majority of the product material being between 100 and 150 mesh. The results of 7.4 rpm are very similar to those obtained during the laboratory grinder tests under similar conditions of speed and force on blades (Table 3-3, 7-rpm, 16-lb force). The distributions at 11.7 and 16.0 rpm are nearly identical and 85% or more of the product is less than 100 mesh. The limited grinding rate results show no increase in grinding rate between these two speeds. The KO_2 block may have started to become bound in the cartridge holder at this time.

SECTION EIGHT

TEST PROCEDURE

In preparation for each test run, inlet moisture and carbon dioxide concentrations were adjusted to the desired values. The inlet CO_2 concentration was adjusted easily by using the CO_2 flow controller. Adjusting the inlet moisture required more time and effort, since such factors as ambient humidity, chill water temperature, and amount of steam being added all had an effect on the final inlet moisture concentration.

The KO_2 block which was to be used was weighed (to the nearest gram) in its cannister on a Toledo scale. After checking the calibration of the three gas analyzers as well as checking that the inlet conditions were properly adjusted, the top of the microcontactor was removed and the KO_2 block placed in the cartridge holder. After replacing the top, the grinder was started. Each run continued for about one hour. Temperatures, flow rates, and pressures were recorded every 5 minutes. A continuous sample of the downstream gas was run to the gas analyzers. Once or twice during each run a gas analyzer sample was taken from the upstream side of the microcontactor for about 5 minutes.

Two filters were used on all runs, with the bottom filter being turned every 10 minutes and the top filter being turned every 5 minutes.

At the end of each run, the grinder and the fan were shut off, and the top of the microcontactor removed. The remaining portion of the KO_2 block was removed, replaced in the cannister, and weighed again to the nearest gram.

SECTION NINE

MICROCONTACTOR TEST RESULTS

9.1 GENERAL

Tests were run mainly to demonstrate 1) that the microcontactor, as conceived, would operate effectively under various inlet conditions of moisture and carbon dioxide and, 2) to demonstrate that the unit was capable of matching an R.Q. of about 0.82. Figures 9-1 through 9-7 show the gas analysis results for each run. The times during which the inlet and outlet samples were taken for gas analysis are shown on each curve. Table 9-1 summarizes the results of the runs and Table 9-2 shows the maximum air temperature and the maximum pressure drop across each of the filters encountered during each run.

Seven runs were made with the complete microcontactor system. The inlet flow for all the runs was 4.0 scfm and the inlet air temperature was about 80°F. It was originally intended that all seven runs have a constant grinding rate, but due to the varying densities within the KO_2 blocks, it was impossible to control the grinding rate too closely.

Coefficients of correlation were determined for the various parameters shown in Table 9-1. Columns 1 to 4 of this table were considered input parameters, while the remaining columns were taken as output parameters. The coefficients calculated for all seven runs resulted in only one good correlation; that being between the average KO_2 grinding rate and the oxygen production rate. Runs 2 and 6 were then discarded as possibly being biased and the coefficients of correlation were determined for the remaining five runs. No explanations were apparent relating to why runs 2 and 6 should be biased but the resulting coefficients indicated, in addition to the previous good correlation, good correlations between the average KO_2 grinding rate and the R.Q. and between the inlet CO_2 concentration and the R.Q. The three "good" correlations are shown in Figures 9-8 to 9-10.

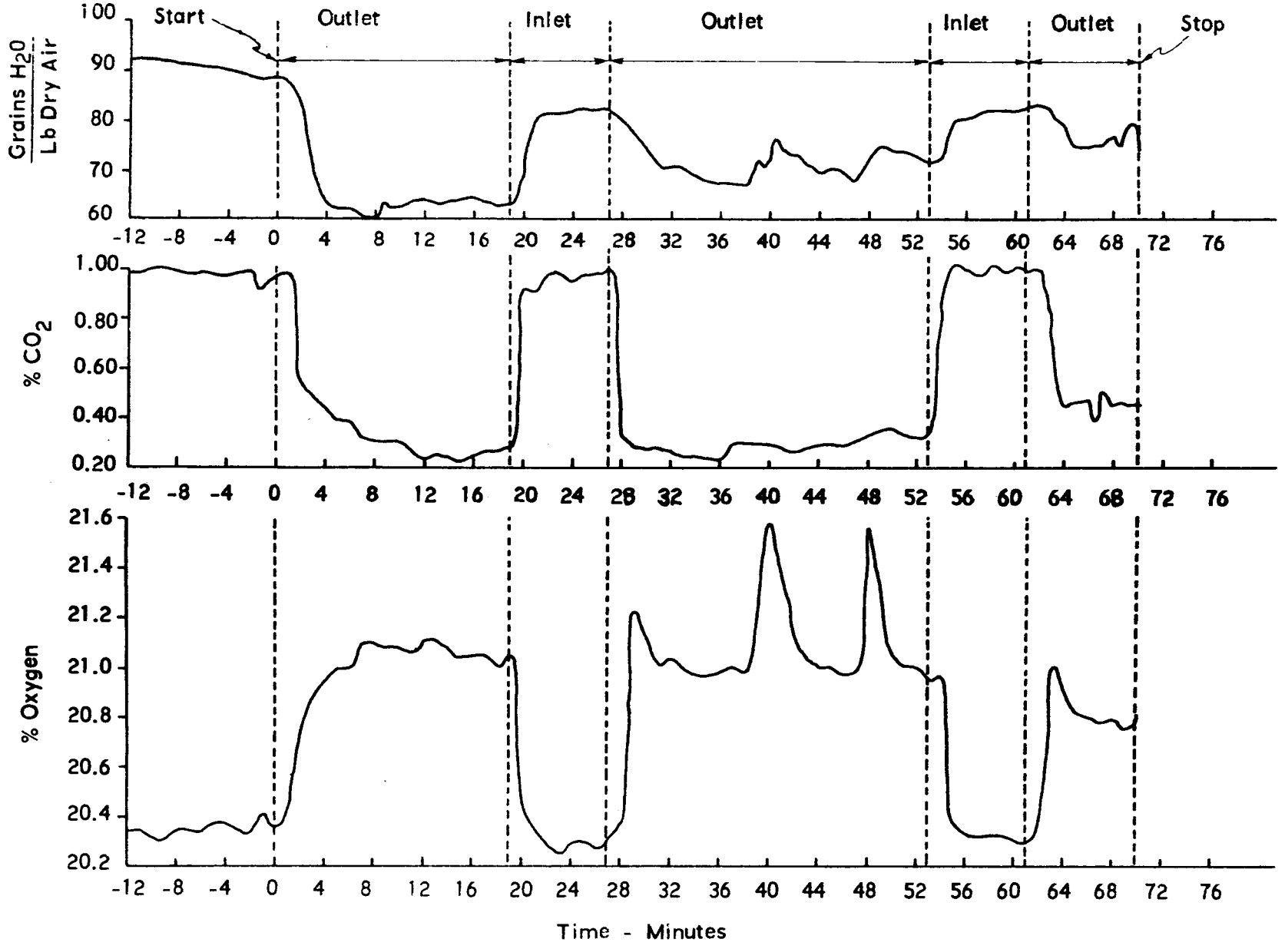


FIGURE 9-1 GAS ANALYSIS DURING RUN #1

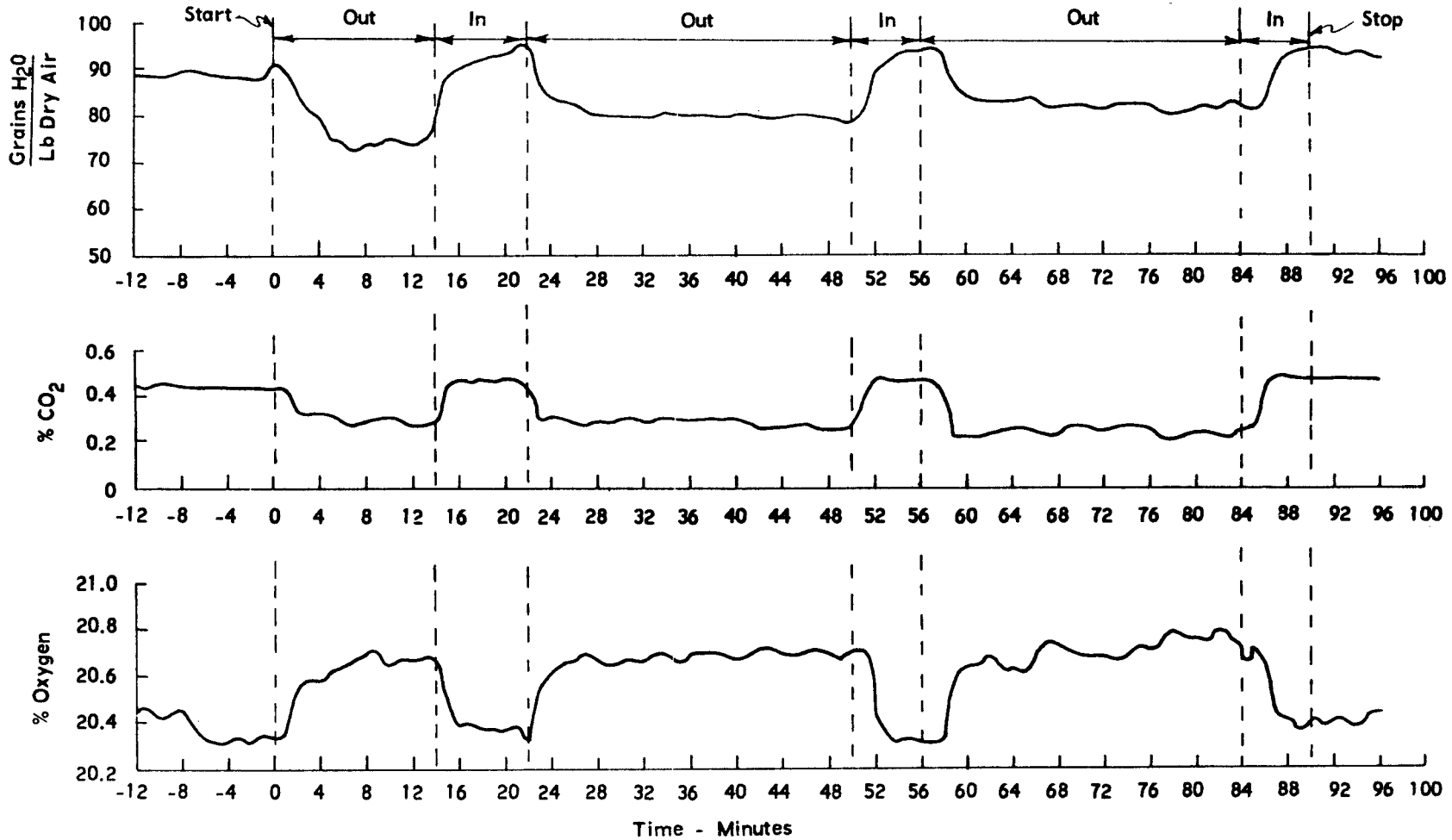


FIGURE 9-2 GAS ANALYSIS DURING RUN #2

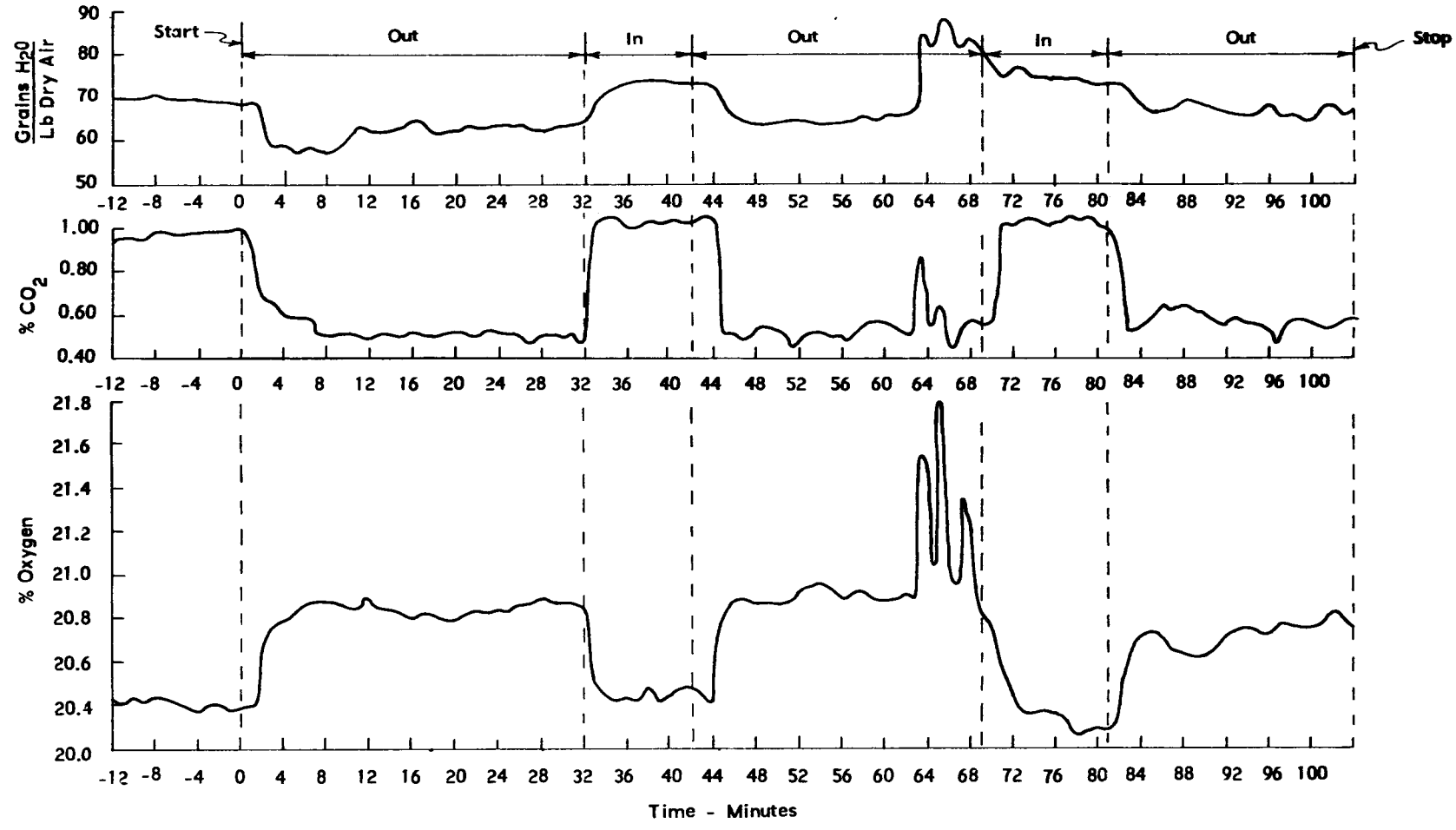


FIGURE 9-3 GAS ANALYSIS DURING RUN #3

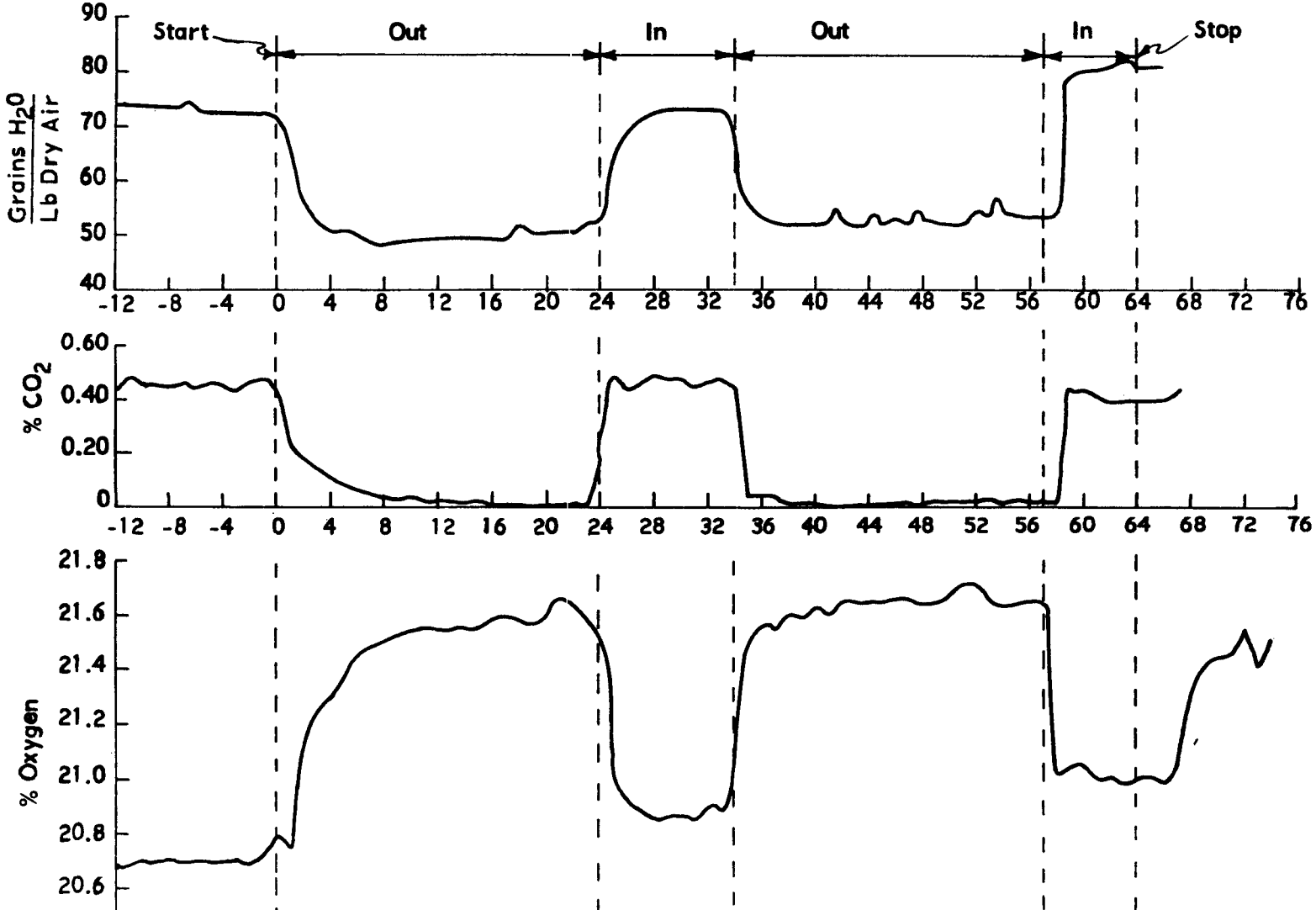


FIGURE 9-4 GAS ANALYSIS DURING RUN #4

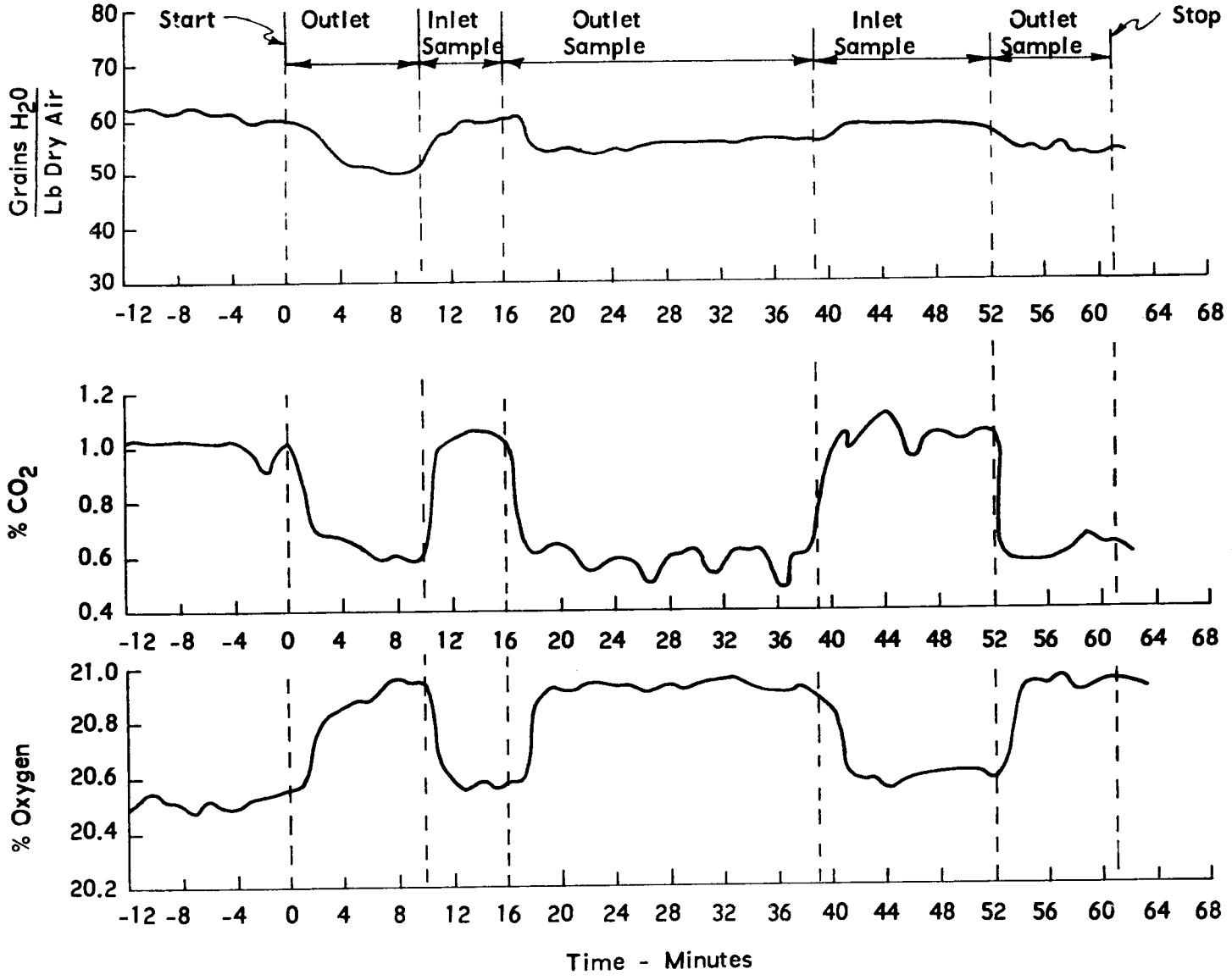


FIGURE 9-5 GAS ANALYSIS DURING RUN #5

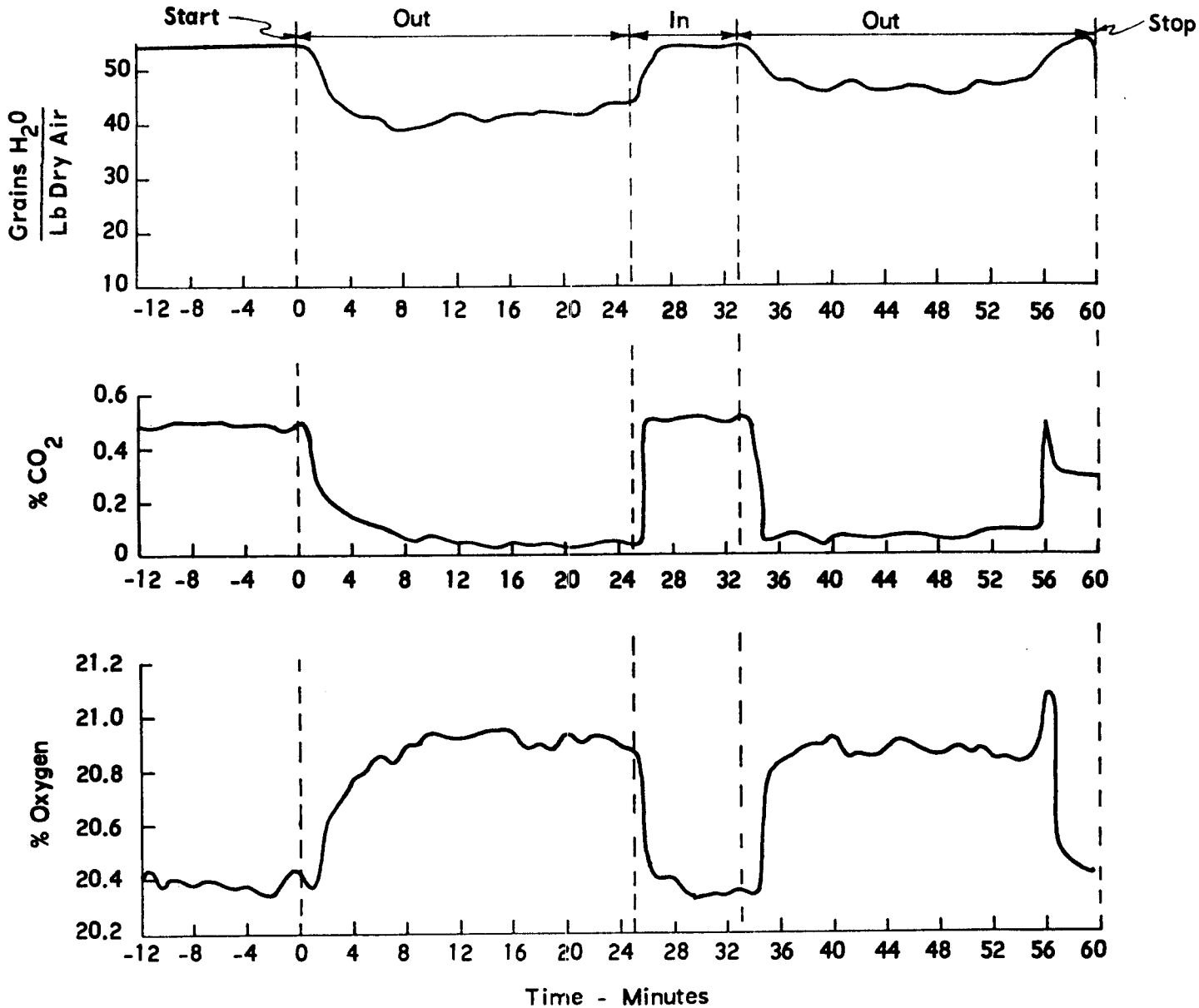


FIGURE 9-6 GAS ANALYSIS DURING RUN #6

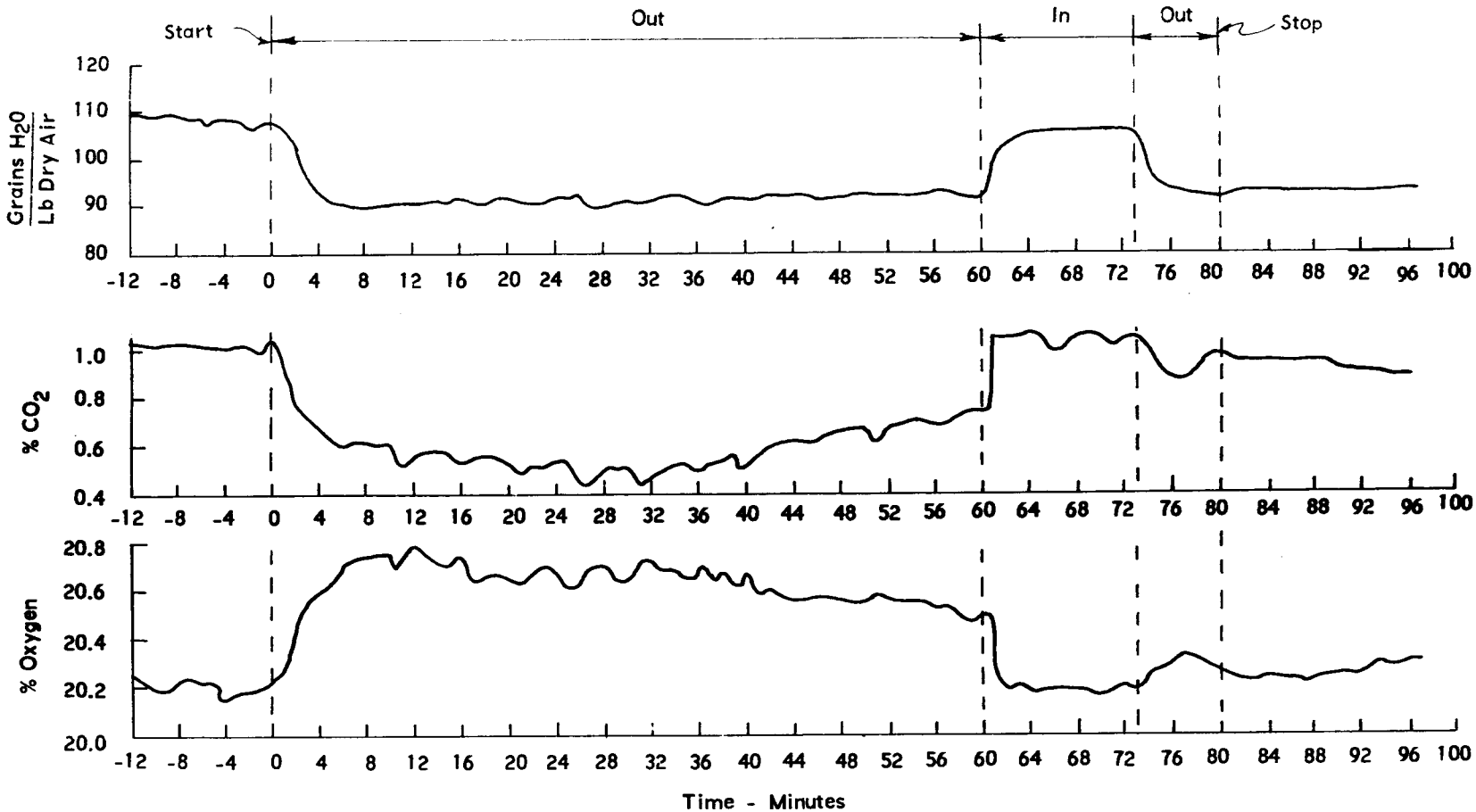


FIGURE 9-7 GAS ANALYSIS DURING RUN #7

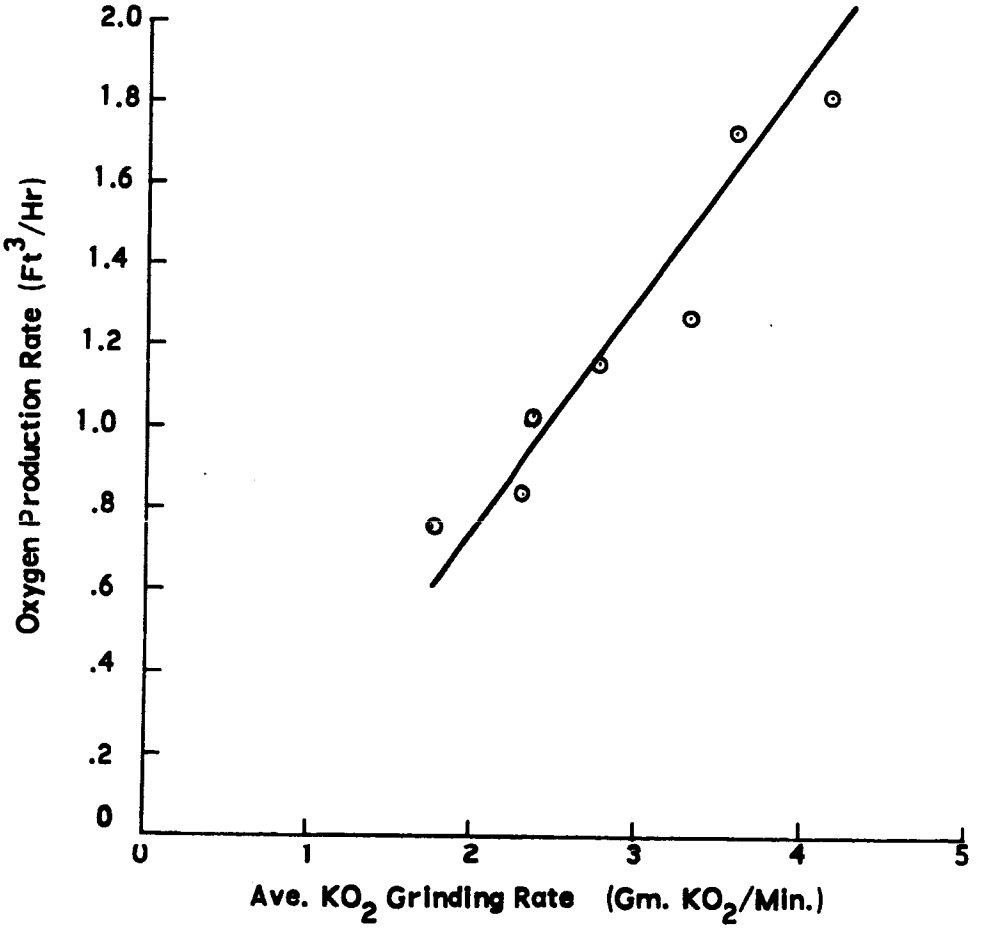


FIGURE 9-8 THE EFFECT OF KO_2 GRINDING RATE ON THE OXYGEN PRODUCTION RATE (Data from Table 9-1)

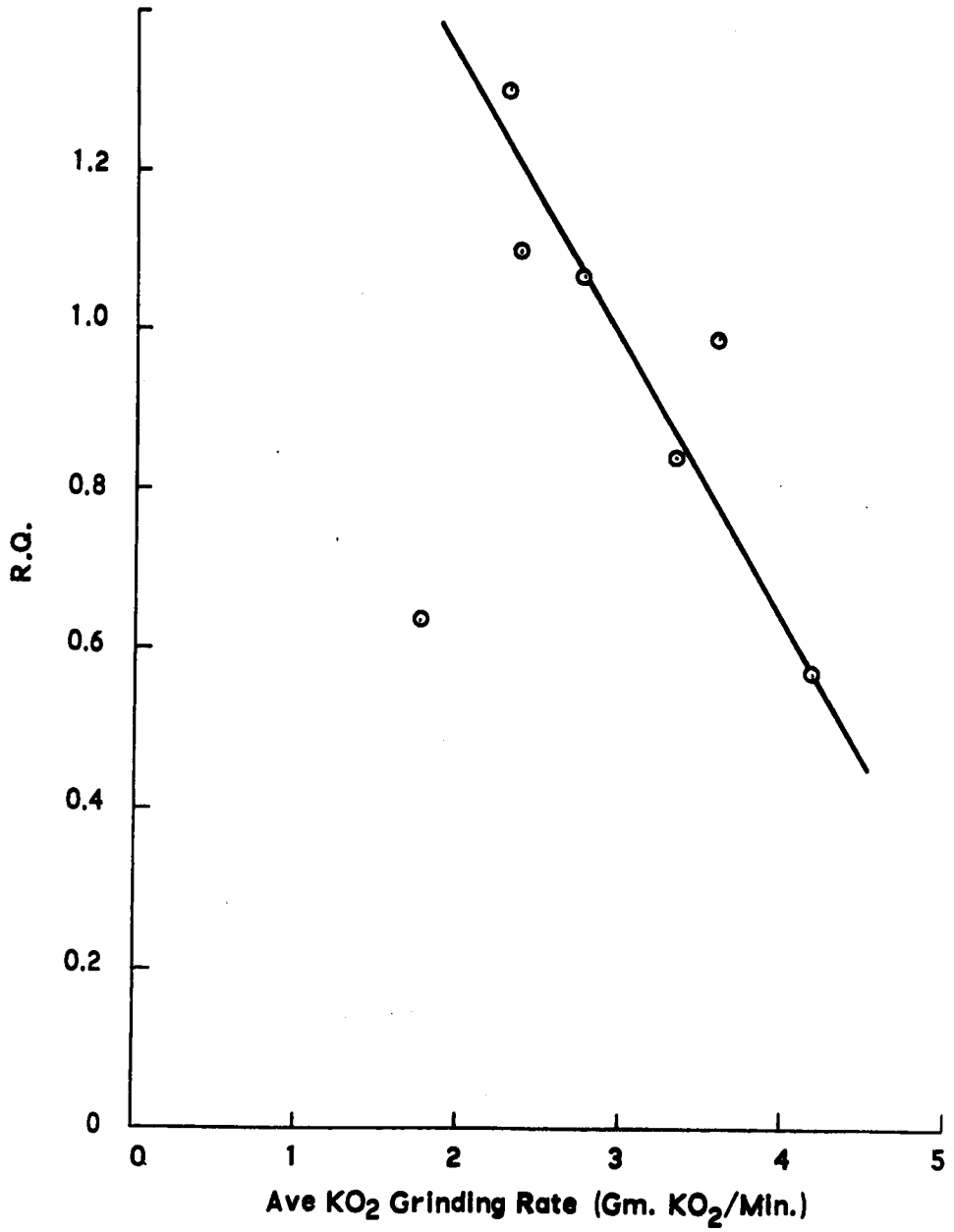


FIGURE 9-9 EFFECT OF KO_2 GRINDING RATE ON R.Q. (Data from Table 9-1)

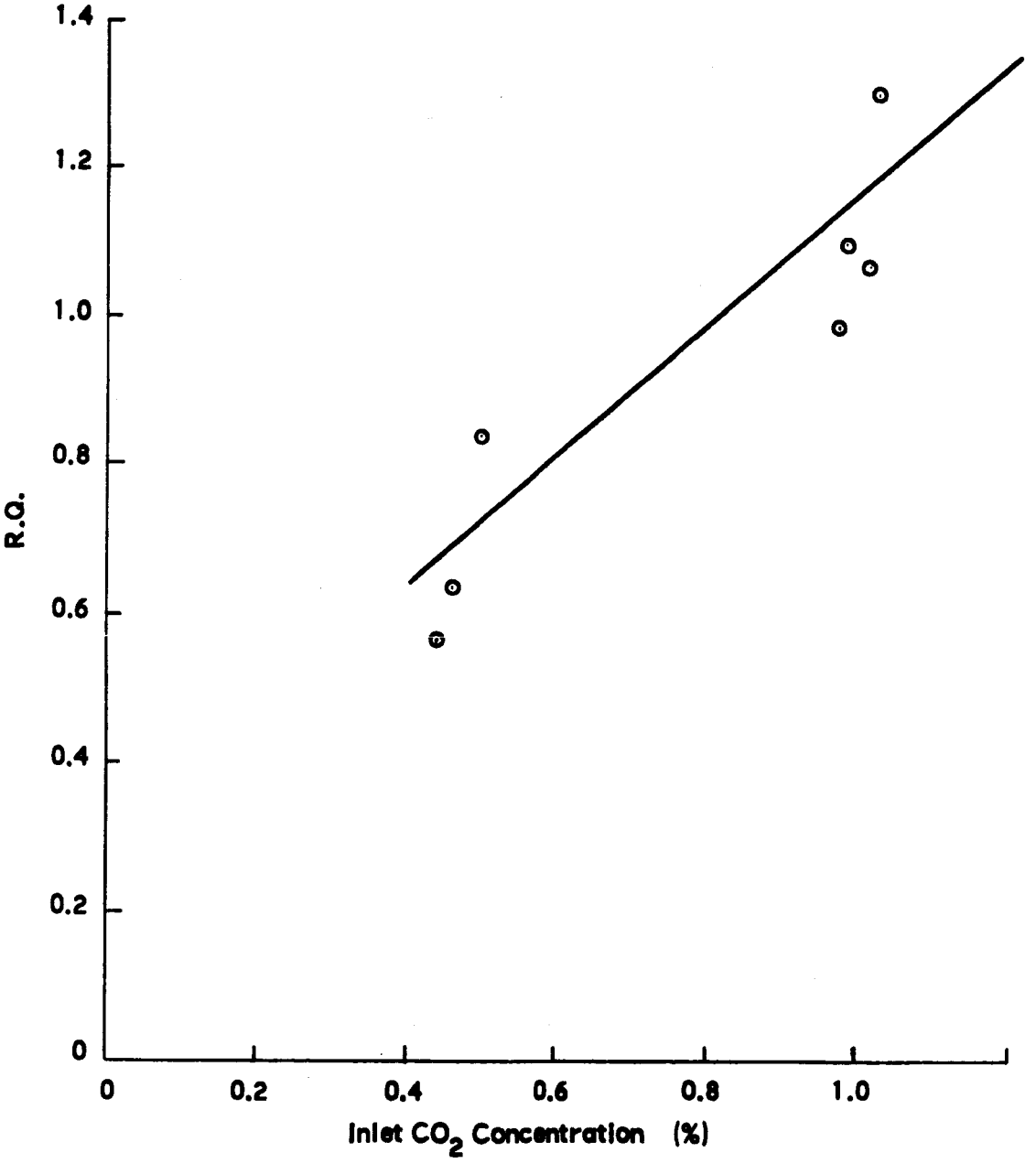


FIGURE 9-10 EFFECT OF INLET CO_2 CONCENTRATION ON R.Q.
(Data from Table 9-1)

TABLE 9-1

SUMMARY OF EXPERIMENTAL RESULTS

Run#	Block #	Inlet Moisture Conc. (Grains H_2O / lb dry air)	Inlet CO_2 Conc. (%)	Ave KO_2 Grinding Rate (g/min.)	Length of run at Equilibrium (min.)	Eff of KO_2 Utilization (%)	Total O_2 Prod (ft^3)	O_2 Prod Rate (ft^3/hr)	Total CO_2 Removal (ft^3)	CO_2 Removal Rate (ft^3/hr)	R.Q. (CO_2/O_2)
1	9	82	0.98	3.6*	45	105*	1.30	1.73	1.29	1.72	0.99
2	10	92	0.46	1.77	82	99.3	1.04	0.76	0.67	0.49	0.64
3	11	73	0.99	2.38	54	100	0.97	1.08	1.06	1.18	1.1
4	11	72	0.44	4.18	49	96.2	1.49	1.82	0.86	1.05	0.57
5	9	60	1.03	2.31	53	79.4	0.74	0.84	0.96	1.09	1.3
6	11	54	0.50	3.35	45	84.3	0.96	1.28	0.81	1.08	0.84
7	1	108	1.02	2.76*	28	92.7*	0.54	1.16	0.58	1.24	1.07
		①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩

* Adjusted to account for decreasing grinding rate near end of run.

TABLE 9-2

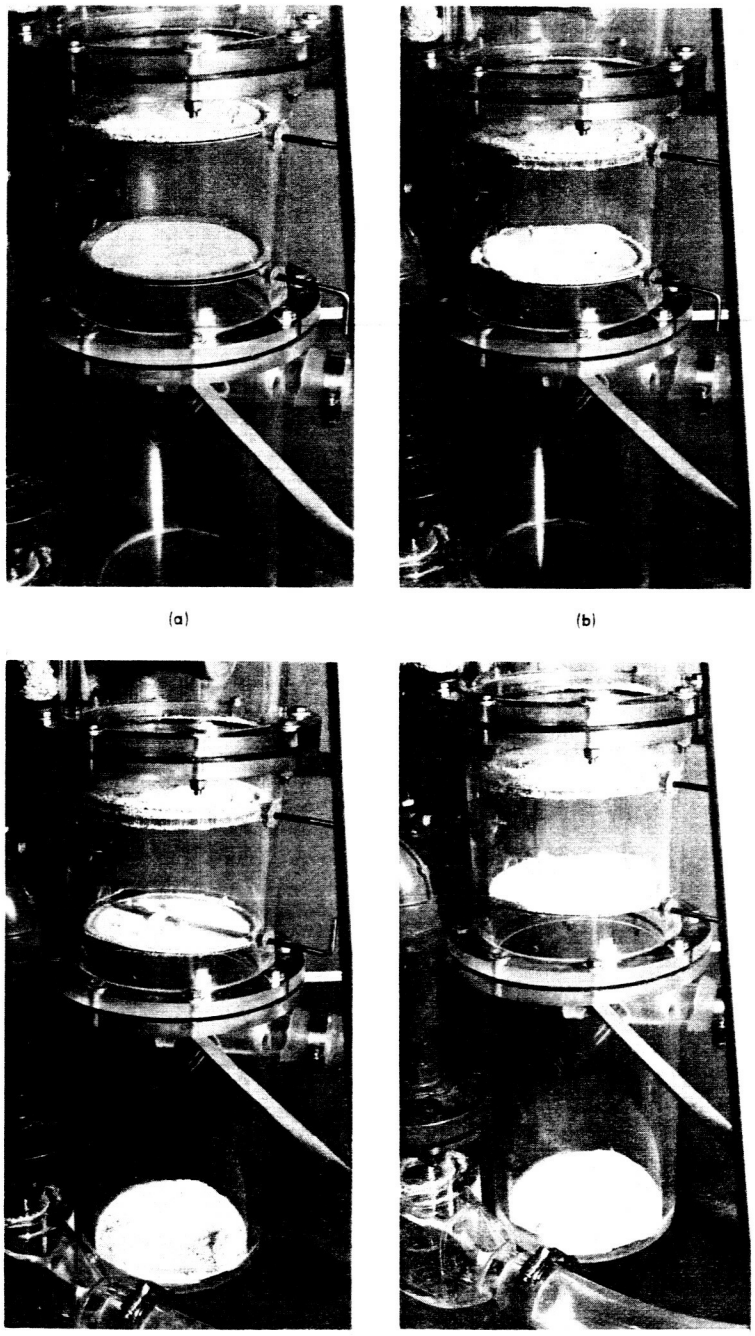
TEMPERATURE AND PRESSURE RESULTS

Run #	Max. Air Temp. in Filter Chamber (^{o}F)	Max. Filter Pressure Drop (in H_2O)	
		Top Filter	Bottom Filter
1	109	---	---
2	104	---	---
3	99	---	---
4	114	5.1	2.0
5	88	---	---
6	99	2.6	2.0
7	106	3.9	1.7

Coefficients of correlation vary from -1.000 to 1.000. The closer the coefficient is to 1.0 or to -1.0, the better the correlation between the two variables. The negative coefficient merely indicates a negative correlation, i.e., when one variable increases, the other decreases. A coefficient with an absolute value of 0.98 or higher is considered very good, and the two variables appear to be highly correlated. Coefficients with an absolute value between 0.90 and 0.98 are considered good and in these cases there are potential indications of good correlation. Due to the small number of samples, more runs should be made to verify the correlation. Coefficients less than 0.90 are considered only fair, and they indicate little or no correlation in predicting one factor from another. Again, in these runs the number of samples were limited and possibly these parameters with low coefficients of correlation should be investigated further.

A series of photographs were made of the filter chamber and the collection chamber during a run (not one of the seven test runs). Figure 9-11 shows the two chambers during the typical run. Photo (a) shows the filter chamber after 5 minutes of operation just prior to turning the top filter. A portion of the KO_2 particles have piled up near the front-center of the top filter, but otherwise, the filter is fairly uniformly covered with KO_2. A few KO_2 particles have bypassed the top filter and are caught on the bottom filter. Some of the material on the top filter is white, which indicates carbonate formation.

Photo (b) shows the filters after about 15 minutes. The top filter has been turned twice and both filters were about to be turned. There appears to be better particle distribution on the top filter. The bottom filter shows how the reaction products come off the top filter in small agglomerates or chunks. The large chunk on the left is from the pile of material in photo (a). The material on the bottom filter is all white except for the large chunk which shows some yellow areas.



(a) (b)

(c) (d)

FIGURE 9-11 FILTER CHAMBER AND COLLECTION CHAMBER DURING TYPICAL RUN

About 10 minutes passed between photos (b) and (c). Both filters were turned together in the interim and the top filter turned once alone. Photo (c) was taken just prior to turning both filters. The large chunk in the collection chamber still shows areas of yellow, indicating unreacted KO_2.

Photo (d) shows the filters and collection chamber after a total operating time of about 35 minutes.

It should be noted that the formation of a higher pile under the cutter blade was caused by the force of gravity acting on the KO_2 particles. Under weightless conditions, the particle would not "fall" but would be carried by the air flow, therefore, a more uniform distribution on the top filter would be obtained.

9.2 DISCUSSION OF MICROCONTACTOR TEST RESULTS

The curves, Figures 9-1 to 9-7, show the gas analysis of oxygen, carbon dioxide, and moisture for the seven test runs. In all cases, the time axis starts prior to the loading of the KO_2 and the start of the grinding to show the established inlet conditions. It took 5 to 6 minutes to load the KO_2 cylinder into the grinding chamber. Some of the curves show breaks in them during this time when the top of the microcontactor was removed and flow rates were upset. All the runs show a rapid change in the air composition at zero time at which time the grinder motor was started. Allowing for the response times of the gas analyzers as shown in Section seven, the curves indicate essentially an instantaneous reaction of the KO_2 particles with the inlet air. In all the runs, the gas analysis curves show that steady-state or equilibrium conditions are established after about only 8 minutes of operation.

To discuss the individual curves and test runs; during run no. 1, two large oxygen peak concentrations occurred at 40 and 48 minutes. There were peak moisture concentrations at the same time which indicate there may have been slugs of water entering the system due to entrainment in the dehumidifying coil. These peak concentrations were

disregarded when calculating average gas concentrations. The grinding rate began to slow down after about 50 minutes of operation due to the KO_2 block binding in the cartridge holder. Figure 9-1 shows that the exit CO_2 begins to increase about this time and the oxygen begins to decrease. The average KO_2 grinding rate and the efficiency of KO_2 utilization was adjusted to account for the decreased grinding rate at the end of the run.

Run no. 2 was made with the smallest average KO_2 grinding rate (1.77 g/min.) of any of the runs. This small grinding rate limited the oxygen production and also the CO_2 removal. Essentially, all of the available oxygen from the KO_2 was released.

Peak concentrations of all three gases occurred between 62 and 68 minutes during run no. 3. Again, the explanation is that there may have been entrainment in the dehumidifying coil which caused a few slugs of water to enter the microcontactor. These peak concentrations were also disregarded when calculating the average gas concentrations.

The CO_2 curve of Figure 9-3 indicates some oscillations between 45 to 62 minutes. The valley of the curves coincide with the time at which the filters were reversed (taking into account the response time of the analyzer). Any time a filter is reversed there is a sudden rise in the air flow rate. With the inlet CO_2 being fed into the air stream at a fixed flow rate, this increase in air flow results in a decrease of CO_2 concentration.

Run no. 4 resulted in the highest oxygen production rate of any of the test runs and it also had the highest average KO_2 grinding rate.

Oscillations of the CO_2 curve are also apparent in Figure 9-5 for run No. 5. Again, as in Figure 9-3, the valleys of the curve coincide with the times at which the filters were turned.

The gas analysis of run no. 6 (Figure 9-6) indicates a peak gas concentration for oxygen and CO_2 at 56 minutes. These peak values were discarded when determining average gas compositions.

Figure 9-7 for run no. 7 shows oscillations of the CO_2 curve and also the oxygen curve. The valleys of the CO_2 curve coincide with the times at which the filters were turned, while the valleys of the oxygen curve do not have as regular a pattern. The grinding rate began to slow down after about 40 minutes of operation due to the KO_2 block binding in the cartridge holder. The oxygen and CO_2 curves of Figure 9-7 reflect this decrease in grinding rate. At the 40 minute mark, the oxygen concentration starts to drop off and the CO_2 concentration starts to increase.

The temperature of the filter chamber, as measured between the two filters, would increase rapidly at the start of the run to the maximum values as shown in Table 9-2 and would remain at about these values as long as the grinding rate did not start to decrease. In runs 1 and 7, the grinding rate did start decreasing during the run as discussed previously and the filter chamber temperature started to decrease at the same time.

It was originally planned to make all the test runs at a constant grinding rate to determine the effects of the other variables, such as inlet moisture and carbon dioxide concentration. Due to the non-homogeneity of the KO_2 blocks, the control of the grinding rate was severely limited. Therefore, the average grinding rate became one of the variables evaluated. As a result, the correlation between the grinding rate and the oxygen production rate produced the best coefficient of correlation. Figure 9-8 shows that the range of grinding rates studied produced sufficient oxygen for about 1 to 1.5 men. The present microcontactor design is capable of grinding, as a minimum, at least twice the range of the grinding rates studied, and possibly as much as three of four times the rates studied. Therefore, the present microcontactor design would be capable of supporting from 3 to 6 men.

The negative correlation between the average grinding rate and the resulting R.Q., as shown in Figure 9-9, is an effect of the higher grinding rates producing more oxygen. Since the R.Q. is inversely proportional to the oxygen production rate, the R.Q. decreases with increasing oxygen production.

The effect of the inlet CO_2 concentration on the resulting R.Q. is shown by Figure 9-10. With the higher inlet CO_2 concentration, more CO_2 is removed by the reaction products. The R.Q. is directly proportional to the CO_2 removal rate; therefore, the higher inlet concentration results in higher R.Q.'s.

One would expect a correlation between inlet moisture and rate of oxygen production. The coefficients of correlation for these two variables did not indicate a good correlation. This discrepancy is probably due to the fact that the average KO_2 grinding rate was different for each of the seven runs and the effect of this uncontrollable variable was predominant over any effect produced by the various inlet moisture concentrations. To determine the effect of inlet moisture concentration, the KO_2 grinding rate must be more exactly controlled.

The inlet moisture concentrations covered a wide range from 54 to 108 grains H_2O/lb dry air. At 80°F, these values correspond to relative humidifies from about 35 to 70%. At the higher moisture concentrations the KO_2 and the reaction products tended to be stickier and therefore more difficult to remove from the filters. The two runs during which the grinding rate slowed down were both at high inlet moisture concentrations (82 and 108 grains H_2O/lb dry air). At the high moisture levels there is probably more reaction on the surface of the KO_2 block in the cartridge holder, which causes the block to bind. Coating the block with a thin, water-impermeable film would reduce or eliminate any reaction of the KO_2 block. Moisture concentrations less than 75 grains H_2O/lb dry air presented no binding problem.

SECTION TEN

SUMMARY

A summary of the work performed under NASA contract NAsw-551 and the results obtained is given below:

1. A small laboratory grinder device was designed and fabricated in order to determine the grinding characteristics of samples of high density (115 lb/ft^3) KO_2 both sintered and unsintered. The test results showed that both types of KO_2 could be ground; the unsintered material produced a majority of particles less than 100 mesh while the sintered material tended to crumble resulting in most of the particles being greater than 80 mesh.

2. Semi-quantitative tests were made to determine the reaction rates of finely divided KO_2 as a function of time, moisture concentration, particle size, and carbon dioxide concentration. For any given length of run the amount of reaction increased with an increasing moisture concentration as was expected, but there was no apparent effect produced by the different particle sizes in the range tested (20 to 150 mesh). The test results show that with 100 to 150 mesh KO_2 particles and an exposure time of two minutes, the per cent reaction ranged from 15% to 70% over the range of moisture concentrations investigated (50 to 80 grains water/lb dry air). The resulting curves indicate that the initial rate of reaction is rapid as a result of it being a surface reaction. Once the reaction becomes diffusion controlled, the rates become slower. The effect of CO_2 concentration (0.5% and 1.0%) on the rate of oxygen production did not appear to be significant.

3. The rate of reaction tests indicated that with a reasonable size reactor, the KO_2 particles, if simply suspended in the air stream, would not react completely in the short time available. Other approaches were considered in order to design a microcontactor that would produce the required residence time for a nearly complete

reaction. The most feasible of the methods investigated was a reversible-filter microcontactor which featured a series of filters placed in the reaction zone on swivel pins similar to a butterfly-valve arrangement. At a fixed time increment, the filters would be turned over in sequence starting from the bottom (see Figure 5-5).

4. A reversible-filter microcontactor with a one-man capacity as a basis was designed and fabricated. The prototype microcontactor consisted of a grinding chamber, a filter chamber, and a solid products collection assembly.

5. A test program was performed to verify the capability of the microcontactor to operate effectively over the range of design conditions. Preliminary grinding tests with the microcontactor indicated a variation of density within each block of KO_2 tested. This variation was a result of the method used in manufacturing the high density KO_2 blocks and was of sufficient magnitude to make close control of the grinding rate impossible.

6. Seven runs were made with the complete microcontactor system. The inlet air flow for all the runs was 4.0 scfm and the inlet air temperature was about 80°F. The efficiency of KO_2 utilization, i.e., the percent of the theoretical available oxygen from the KO_2 which was released during the run, ranged from 80 to 100% during the seven test runs. The test results showed that the microcontactor concept is capable of producing the range of R.Q. (0.6 to 1.1) that is comparable to that required by man. The results also showed that there was no over-production of oxygen as is commonly encountered when using cannisters of the superoxide. The microcontactor approach resulted in steady-state conditions (with respect to gas composition) being obtained after only about 8 minutes of operation.

   A good correlation was obtained between the average KO_2 grinding rates and the oxygen production rates in the seven test runs. Oxygen production rates between 0.8 and 1.8 ft^3/hr were obtained

with KO_2 grinding rates in the range of 1.7 to 4.2 gm KO_2/min. The test results indicated a negative correlation between the average KO_2 grinding rates and the resulting R.Q.'s. This is an effect of the higher grinding rates producing more oxygen. The tests also indicated that higher inlet CO_2 concentrations result in higher R.Q.'s.

SECTION ELEVEN

CONCLUSIONS AND RECOMMENDATIONS

The results of the work performed show that the microcontactor concept is feasible as a method of revitalizing a sealed atmosphere. The high-density KO_2 can be ground into small particles which rapidly react with the flowing air stream. The test results show that by grinding the required quantity of KO_2 and exposing it to the air stream, there is no over-production of oxygen as is commonly encountered when using cannisters of the superoxide. The microcontactor approach results in steady-state conditions being obtained after only a few minutes of operation. The test results also show that the microcontactor concept is capable of producing the range of R.Q. (0.6 to 1.1) that is comparable to that required by man. It would seem likely that by setting the inlet conditions of moisture and grinding rate of the KO_2, any R.Q. within the above range could be produced.

Although the microcontactor concept is feasible, it cannot be said that the present filter chamber and solid collection chamber assembly are the optimum design to perform the required functions. The grinding chamber design, with some additional modifications and refinements, has shown promise of operating very effectively.

The function of the reversible filters is to provide a retention time of sufficient duration for the KO_2 particles in the flowing air stream to completely react before passing into the solid product collection assembly. The most serious problem with the filters was that the KO_2 reaction products would partially stick to the Teflon monofilament filter material even after the filter had been turned and the air direction was reversed. The anticipated "self-cleaning action" never materialized. The continuing sticking of the material with time resulted in an increasing pressure drop across the system. Other means for providing the required retention time must be investigated.

The solid product collection assembly is affected by the performance of the method used to provide the required retention time. In the case of the present design, the reversible filters resulted in a reaction product consisting of many agglomerates and chunks of material. The entrance to the cyclone separator was easily plugged by these large agglomerations. If an approach for providing the retention time could also prevent any agglomeration, then a cyclone separator would operate effectively.

The test program, although limited, had as its primary objectives verification of the microcontactor concept; illustrating that by supplying freshly ground KO_2, the system would replenish oxygen and remove CO_2; and indicating the range of R.Q. that could be maintained. No attempt was made to investigate tne effects of all the independent variables, or their interactions.

Three variables were held constant during the test program, 1) inlet air temperature, 2) air flow rate, and 3) retention time. The latter is a function of the number of filters and the frequency of reversing the filters. These variables have all yet to be investigated in order to determine the optimum operating conditions for producing any R.Q. in the desired range. The effect of temperature could prove to be quite significant, since the amount of bicarbonate formation is temperature dependent. Flow rate and retention time could be equally as important.

As a consequence of the favorable results obtained from the experimental work completed to date, it is recommended that additional work be performed to do the following:

1. Make the necessary revisions and/or modifications to obtain a constant and predictable grinding rate at any rpm setting within the grinder range. This would probably entail a modification to the manufacturing method for the KO_2 cylinders in order to obtain homogeneous density throughout the block.

2. Set up a statistical, experimental design to determine the effects and interactions of the following independent variables:

   a) KO_2 grinding rate

   b) inlet moisture concentration

   c) inlet CO_2 concentration

   d) inlet air temperature

   e) air flow

   f) retention time

3. Study and investigate other methods of providing the necessary retention time for the KO_2 particles in the flowing air stream, along with comparable methods of solid product collection.

4. Determine the grinding characteristics of high-density NaO_2. If NaO_2 appears feasible, perhaps item 2 above should also be performed on NaO_2.

5. Make necessary modifications to the present system to optimize the unit.

6. Perform extended runs of 8 and 24 hours.

7. Integrate the system with a primate and/or man for an extended run.

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