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STUDY OF OXIDES FOR $O_2 - N_2$ SEPARATION

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Section 1

INTRODUCTION

This progress report summarizes work done on Contract AF 33(657)-10797 during the sixth quarter (15 April - 15 July, 1964). The primary objective of this program is the development of reactant particles suitable for application in a high temperature $O_2 - N_2$ separator device.

The fifth quarterly progress report (April 30, 1964) of the present contract described the results obtained with different types of particles in the Rotary Disc Separator, and the approaches being followed to develop faster reacting particles which will be able to withstand the RDS operation. It was clear from the reaction rate data obtained that reaction rate increases from 2 to 3 times faster are all that is necessary to meet target requirements. It was quite apparent also that the surface areas of the pure CoO reactant particles tended to decrease to 0.2 to 0.5 m^2 /gram regardless of the manufacturing method employed, while target surface areas are of the order of 1.0 m^2 /gram for pure CoO. On the basis of these results, the work performed during the present quarter was directed towards:

- a) Gaining a deeper insight into the morphology of micro-porous particles in general, and the developed reactant particles in particular.
- b) Increasing the understanding of the sintering behavior of cobalt oxide and beryllium oxide powders and agglomerates.
- c) From (a) and (b), arrive at and test approaches which could yield more suitable particle structures than the ones presently obtained.

The results obtained from steps (a) and (b) are quite interesting, although by no means unexpected. The application of the findings (step c), has not been brought to completion yet, and it is unknown whether or not some of the new approaches will be successful. The RDS testing of reactant particles is the most important means

for their evaluation, and unfortunately the number of test runs which can be performed is small compared with the number of reactant materials which would be desirable to submit to RDS testing.

Section 2

STUDIES OF THE MORPHOLOGY OF MICROPOROUS PARTICLES

2.1 General Background

It is very difficult to observe visually the structure of microporous particles such as the ones being manufactured (0.1 to 2μ prime particle size) because of several reasons:

- 1) The limit of resolution of light microscopy is reached at this point, since the particle size is of the order of magnitude of the wavelength of light.
- 2) The depth of field for either an optical or an electron microscope at the necessary magnification is just about zero, making it impossible to look at a particle surface with any perspective. If flat samples are prepared by polishing techniques, the sample surface is altered (particles pull out from the surface during the polishing stage).

An example of the best results that can be obtained with careful polishing of a sample and subsequent observation in an optical metallograph can be seen in Figures 13 and 14, Fifth Quarterly Progress Report, Contract AF 33(657)-10797. Such results are hopelessly inadequate as far as gaining insight into the actual structure of the porous particles.

It has been possible to observe two sets of cobalt oxide particles and two sets of cobalt on beryllia particles in an electron beam scanning device, which has a resolution of better than 0.5μ , and permits the observation of three dimensional samples with an extraordinary depth of field.

2.2 General Description of the Equipment

Very briefly described, the Micro-Scan "sees" with a beam of electrons,

accelerated by an applied voltage, and focused with magnetic lenses onto the sample. Magnetic deflection coils guide the beam across the surface of the sample in a scanning sequence. When the primary electrons strike the surface they cause the material to emit low-energy secondary electrons in quantities determined by the nature of the material and the angle of incidence. A portion of these secondary electrons is collected by a scintillator, whose light output is detected by a photomultiplier. The signal from the photomultiplier is converted to a voltage, amplified, and used to control the brilliance of a display cathode ray tube whose electron beam is moved in synchronism with the primary beam on the specimen. The display cathode ray tube thus presents an image, in terms of secondary electron emission, of the surface topography of the specimen.

2.3 Results

Two sets of pure cobalt oxide particles and two sets of BeO supported cobalt oxide particles were submitted to microscan inspection. The specimen particles were coated with a gold film approximately 500-1000 $\text{\AA}$ thick, in order to render the specimen surface equipotential, as the basic materials under study are not good conductors.

Figures 1 and 2 show the appearance of the cleaved surface of an extruded-paste cobalt-oxide particle, sintered for about 2 hours at 1800 $^{\circ}\text{F}$. The prime particles have sintered to each other to quite an extent, as evidenced by the smooth, rounded appearance of the particles and their coalescence into larger agglomerates in some areas. The average particle diameter (1-2 μ) as observed in the above pictures gives a good check to the surface area measured for this material (0.843 m^2/gram).

Figures 3 and 4 show the appearance of the cleaved surface of a cobalt oxide particle obtained by pressing the prime powder into tablets, subsequently sintering at 1800 $^{\circ}\text{F}$ for a total of about 2 hours. The results are remarkably similar to the ones shown in Figures 1 and 2 for extruded particles. Here again there is evidence of pronounced sintering, and the average particle diameter of

1 to 2μ agrees well with the measured B.E.T. surface area of $1.2 \text{ m}^2/\text{gram}$. The particles have sintered to each other to the extent that they form a series of continuous chains or bridges, which may account for the high strengths achieved with these sintered particles.

Figures 5 and 6 represent the structure of sintered BeO spherical agglomerates (obtained by powder tumbling techniques) impregnated with several coats of $\text{Co}(\text{NO}_3)_2$, subsequently decomposed by firing in air at 1600°F . There is no significant difference between the outer surface of the particles and their cleaved cross section, indicating that the cobalt oxide coating does not obstruct appreciably the pores on the particle surface. The prime particle (and pore size) is small enough to reach the limit of resolution of the instrument, but it can be estimated to be of the order of 0.2 to 0.5μ , which is in accordance with the measured B.E.T. surface area of $7.1 \text{ m}^2/\text{gram}$.

Figure 7 shows a cleaved cross section of an extruded 50% by wt. BeO - 50% by wt. Co_3O_4 particle, sintered at 1800°F for 2 hours. Its structure is fairly similar to the coated particle of Figures 5 and 6, although there is a suggestion of more severe sintering having taken place. The average prime particle size, again of the order of 0.5μ , is in agreement with the measured B.E.T. surface area of $6.8 \text{ m}^2/\text{gram}$. After running in RDS 34, the particles sintered down to $3.5 \text{ m}^2/\text{gram}$, confirming the visual observation that a material with this high Co_3O_4 content is quite susceptible to sintering.

Section 3

STUDY OF SINTERING OF COBALT OXIDE POWDERS

The extent to which both compacted and uncompact cobalt oxide powders sinter (loose surface area) motivated a study directed towards establishing the magnitude of a stable surface area which could be achieved by proper control of such variables as initial particle size distribution, shape, sintering conditions, etc. Loose powders, even of submicron particle size, can be readily observed by means of transmission electron micrographs, and with regular optical microscopes if of larger particle sizes. Figure 8 shows a typical cobalt oxide starting material. It consists of either micron-sized highly porous particles, or of micron-sized agglomerates of submicron particles. A great variation in pore size (particle size) distribution is evident. It is not surprising, then, that upon heating to 1800°F for 1 hour, the cobalt oxide particles appear as they do in Figure 9. The cobalt oxide has sintered into larger, smooth agglomerates which should be quite stable from then on. The presence of such a fine prime particle size enables the powder to coalesce upon sintering into agglomerates of a diameter of even several micron.

Ball milling of a coarse powder to increase its surface area is not very effective, since this process produces a very wide particle size distribution, with a large amount of fines, which account for most of the surface area after milling, and which sinter markedly as soon as exposed to high temperatures. A coarse cobalt oxide material of $1.8 \text{ m}^2/\text{gram}$ surface area was ball milled to $3.37 \text{ m}^2/\text{gram}$, at which point it appeared as shown in the electron micrograph of Figure 10. The preponderance of fines is self-evident. After sintering for 1 hour at 2000°F , the powder was cooled and held in air at 1400°F for 1 hour, and finally cooled at room temperature. The surface area after this treatment had dropped down to $0.40 \text{ m}^2/\text{gram}$.

It is clear from the above described experiences that it would be desirable to start with an oxide powder of uniform particle size, which will sinter

to a moderate extent during the firing stage following particle compaction. One promising approach appeared to be starting with a cobalt bearing substance of the desired and uniform particle size, which upon calcination would yield cobalt oxide particles of similar size and shape. Cobalt oxalate yields cobalto-cobaltic oxide when fired in air at temperatures of the order of 1000°F . The particle size of cobalt oxalate can be readily controlled by varying the precipitation conditions of the salt from the nitrate solution. The most convenient variables are cobalt and oxalic acid concentration, and precipitation temperature. The product oxalate crystals are fine needles, at least 10 diameters long. Figures 11, 12, and 13 illustrate how smaller crystal sizes are obtained by increasing the reagent concentrations at a given precipitation temperature. The surface area of the cobalt oxalate shown in Figure 13 is $6.42 \text{ m}^2/\text{gram}$. The temperature at which the oxalate is decomposed to yield the oxide is quite important, since the additional effect of the oxalate igniting and producing hot spots during the decomposition stage exists. This effect has been observed in one case where cobalt oxalates being decomposed at 1100°F glowed red hot inside the furnace, and resulted in cobalt oxides of only $0.5 - 0.65 \text{ m}^2/\text{gram}$ surface area, whereas cobalt oxalates decomposed at 900°F did not ignite, and resulted in oxide surface areas of $13.2 \text{ m}^2/\text{gram}$.

Figures 14, 15, and 16 show cobalt oxide powders obtained by different decomposition techniques. The oxides still retain the needle-like shape of the parent oxalate.

Section 4

PARTICLE DEVELOPMENT PROGRAM

4.1 Unsupported Cobalt-Oxide Reactant Particles

Unsupported reactant particles of cobalt oxide have been manufactured from prime particles and/or agglomerates by three basic methods: one, compacting by rolling cobalt oxide powder into spheres; two, pressing the powder into tablets; and three, extruding a paste of cobalt oxide and a liquid. Each of these basic methods provides a unique means for bringing prime particles and/or agglomerates into intimate contact with each other prior to the particle sintering process.

4.1.1 Extruded Particles

Extruding a paste of cobalt oxide powder and a liquid (usually water) makes use of surface tension to effect intimate contact between prime particles and/or agglomerates. In an effort to vary the amount of interparticle contact (and therefore reactant particle porosity and surface area), the surface tension of the liquid used for making the cobalt-oxide paste was changed.

Two methods were employed to change surface tension: one, extruding the paste at an elevated temperature; and two, mixing methanol with the water prior to extruding. Reactant particles made by each of these methods were then tested in the Bomb Rate Tester. Results from these tests are shown in Figure 17 for paste extruded hot and BRT No. 575 for methanol and water. No significant increase in reaction rate was observed.

4.1.2 Fillers

The use of various filler materials has been proposed for manufacturing cobalt-oxide reactant particles with the proper pore geometry and distribution. The prime particles and/or agglomerates of cobalt oxide, together with the appropriate filler material, are mixed thoroughly. Sintering of the reactant particles takes place at a temperature which will not destroy the integrity of the filler material. After

sintering is complete, the filler material is removed by suitable means and interconnected pores of the proper size are left in the reactant particles.

Two different types of filler material were used to evaluate the above outlined procedure: cellulose fibers and cobalt sulfate. The cellulose had fiber diameters of 10-20 μ and fibril diameters of 0.1 to 0.3 μ . Small amounts of cellulose were mixed with water and cobalt oxide; the resulting paste was extruded, dried, sintered (the cellulose was removed as carbon dioxide and water vapor) and sized to form reactant particles. These particles were run in the Bomb Rate Tester. Bomb Rate performance is shown in Figure 18. Results indicate no significant increase in reaction rate.

Based on the particle performance in Bomb Rate Test No. 566, it seemed appropriate to raise the cellulose concentration in order to obtain a greater reaction rate. Therefore, larger amounts of cellulose fibers were ball milled with cobalt oxide and water. The resulting paste was dried, sintered and sized. This material was reacted in the Bomb Rate Tester; results are shown in Figure 19 (particle oxidation). Inspection of these results indicated that the carbon formed from cellulose decomposition in an oxygen depleted atmosphere had reduced the cobaltic oxide to elemental cobalt. This observation was further corroborated by the magnetic character of the reactant particles after the Bomb Rate tests. The particles were removed from the bomb and oxidized fully to cobaltic oxide in an oxygen rich atmosphere. The reactant particles were then re-evaluated in Bomb Rate Test No. 573 shown in Figure 20. The oxidation rate of these particles is not exceptional.

The other filler material, cobalt sulfate, was mixed thoroughly with cobalt oxide; the resulting mixture was pressed into tablets. Sintering was carried out at 1400°F for 2 hours (in air) and finally at 1800°F in air for one hour. During the final sintering stage, cobalt sulfate was decomposed to cobalt oxide and sulfur dioxide. The reactant material was sized and evaluated in Bomb Rate Test No. 574, the results of which are shown in Figure 21. The oxidation rate is not atypical.

4.1.3 Selective Reduction and Leaching

Regeneration of a cobalt-oxide reactant particle takes place initially along pore walls and particle surfaces. In order to increase reactant particle porosity and surface area, it was decided to exploit the above stated fact by allowing reactant particles to regenerate partially, and then selectively leaching the lower oxide formed by regeneration.

Tablet type cobaltic oxide particles were prepared and sintered in the conventional manner, placed in a Vycor vial, and reduced under vacuum (at 1800°F) until the pressure of oxygen over the particles corresponded to 15% (by weight). The cobaltous oxide formed on pore walls and surfaces of the particles were then leached in a hydrochloric acid solution; the particles were washed, dried, and prepared for a Bomb Rate Test. Figure 20 shows the test results. No improvement in oxidation rate was noted. Further studies are planned using selective reduction and leaching in a stepwise manner. This will be accomplished by reducing 1-3% leaching, and sintering between each step until the total desired per cent reduced is achieved.

4.1.4 Use of Controlled Size and Shape Cobalt Oxide Powders

Cobalt oxide powder manufactured by controlled precipitation and decomposition of cobalt oxalate are being tested as starting materials for both extruded paste and pressed tablet cobalt oxide particles. This investigation has just be initiated, and therefore results are not yet available. However, the whole approach is highly promising, since a uniform particle size starting material should yield an agglomerate devoid of large sintered zones with very low surface areas.

4.2 Supported Cobalt Oxide Reactant Particles

All of the work performed on the development of supported cobalt oxide reactant particles was based on the use of beryllium oxide as the particle support material. Since there is some degree of interaction between the BeO support and the cobalt oxide reactant, the trend has been towards increasing the cobalt oxide

loadings, and using the BeO in the structure to act as a barrier to prevent pronounced sintering of the reactant oxide.

4.2.1 Cobalt Oxide Coated BeO Particles

Beryllium oxide support particles of considerable strength which retain the necessary surface area can be obtained by sintering the powder agglomerates at higher temperatures than the ones previously used. For example, particles obtained by extruding, drying and sintering a 50% by weight water BeO power paste yielded the following results:

Sintering Temperature °F	Sintering Time hours	Surface Area m ² /gram
2000	2.0	12.8
2200	2.0	11.5

A sample of beryllia particles, sintered at 2000°F for 2 hours, with a 0.63 void fraction was impregnated with 5 watts of 4 M Co (NO₃)₂, drying and decomposing each coat at 1400°F. The resulting particle bulk density increased from 0.56 g/cc to 1.09 g/cc, indicating that the resulting CoO loading was of the order of 48.5%. These particles were run in the Bomb Rate Tester, and the results of the oxidation tests at 1600°F and 1750°F can be seen in Figure 22. There is an appreciable reaction rate at 1750°F and 50 psia, contrary with the experience with lower loading CoO on BeO particles, which in many cases did not react at all. The same type particles were run in Rotary Disc Separator Tests 28 and 33, as shown in Figure 23 and 24. The particles do react under cyclic conditions, but the reaction rate is slow, slower than for some pure cobalt oxide particles tested to date.

4.2.2 Cobalt Oxide - BeO Mixtures

The addition of varying amounts of beryllium oxide powder to the reactant oxide was considered for the purpose of reducing the extent to which the pure reactant oxide sinters at operating temperatures. Up to the present time, two main methods have been employed for the consolidation of the intimate mixtures

of cobalt oxide - beryllia powders (usually obtained by ball milling the powders together):

- a) Rolling the powder into spherical particles
- b) Extruding the particles as a water paste.

In both cases it is difficult to assess which fraction of the total surface area corresponds to the cobalt oxide.

4.2.2.1 Rolled Particles

Figure 25 shows the BRT performance of particles containing 10% by weight BeO. The reactant material appears to "open up" or activate after the first two runs. Figure 26 presents similar results obtained with a reactant material containing 25% by weight beryllia. The observed reaction rates are actually slower. Figure 27 shows results obtained with a 50% by weight BeO material. As in all cases, the regeneration rates are quite fast compared to the oxidation rates, and are promptly limited by the adiabatic cooldown of the particles throughout the test. Finally, Figure 28 presents results obtained with particles identical to the ones of Figure 27 (50% by weight BeO) except for the particle size, which is 250 to 350 μ diameter instead of 710 - 1000 μ . The fact that the results are almost identical despite a change in particle size of almost a factor of three strongly indicates that diffusional effects (in a pure O₂ atmosphere more properly called bulk flow) are negligible.

4.2.2.2 Extruded Particles

One batch of particles was prepared by extruding a water paste of a 50% by weight mixture of Co₃O₄-BeO (ball milled together for 2.0 hours) and sintering the particles for 2.0 hours at 1900°F. The Bomb Rate Tester results are shown in Figure 29. The surface area of the material decreases progressively with each run, but the oxidation rate is not appreciably affected. The pore volume of the starting material is 0.158 cc/gram corresponding to a porosity of 38.5%. The performance of the particles on Rotary Disc Separator testing is of the same magnitude, as evidenced by the data from R.D.S. Test No. 36 shown in Figure 30.

This is surprising since cyclic reaction rates have always been faster than bomb rate test reaction rates. Since the particle size was rather large (over 1000 μ) and the porosity is not particularly high, there is a possibility that diffusion control may be important during air operation (that is, to say, in the R. D. S. test). This question will be tested by running similar particles of smaller particle size.

Another set of extruded particles, identical to the ones described above, was loaded with additional cobalt oxide by impregnating with two coats of 4 M $\text{Co}(\text{NO}_3)_2$, decomposing the salt after each impregnation by firing in air at 1400°F. The resulting pore volume, 0.133 cc/gram, and approximate porosity of 33% are small enough to suggest that the diffusion problem should be even more acute during air (R. D. S.) operation. This was definitely the case, as it can be seen from the following sequence of tests. The Bomb Rate test results (Figure 31) indicate that the particles do deactivate with use, but the observed reaction rates are the same or slightly higher than for the uncoated extruded particles. The Rotary Disc Separator test shown in Figure 32, however, is slow, suggesting that diffusion may be a problem here as well. Testing of smaller particles will establish whether or not this explanation is correct.

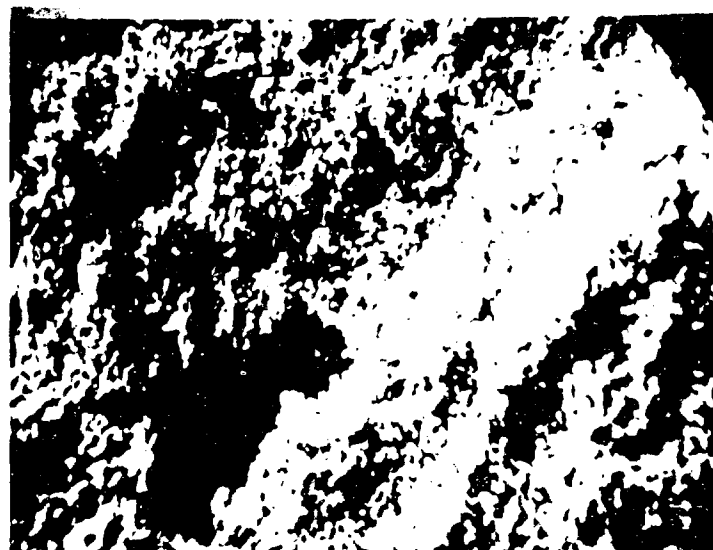


Fig. 1 Extruded Co_3O_4 Particle, Cleaved Cross Section
From RDS 30 Control, .25X

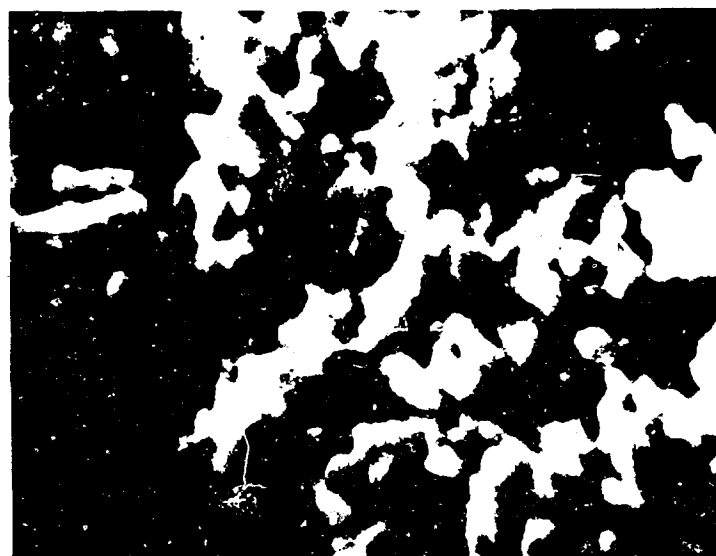


Fig. 2 Extruded Co_3O_4 Particle, Cleaved Cross Section
From RDS 30 Control, 5000X

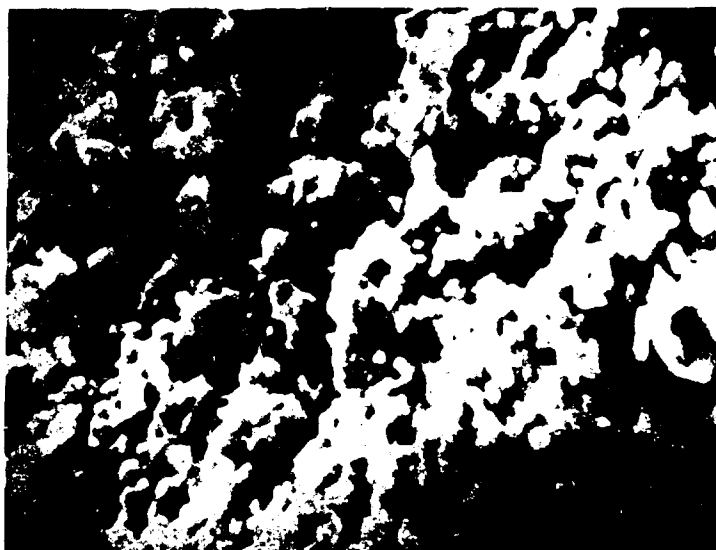


Fig. 3 Pressed Tablets, Co_3O_4 , Cleaved Cross Section
From RDS 20 Control, 2250X



Fig. 4 Pressed Tablets, Co_3O_4 , Cleaved Cross Section
From RDS 20 Control, 7250X

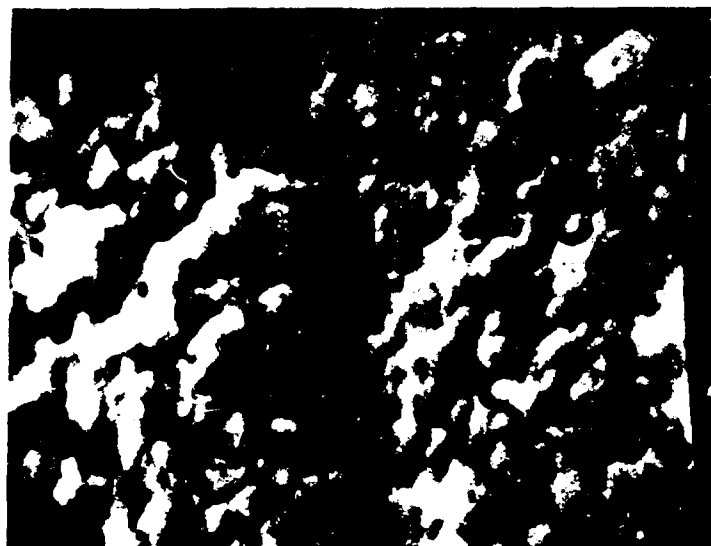


Fig. 5 Sintered BeO Spheres Impregnated with Co_3O_4
Before RDS 28, Outer Surface, 5000X



Fig. 6 Sintered BeO Spheres Impregnated with Co_3O_4
Before RDS 28, Cleaved Surface, 5000X

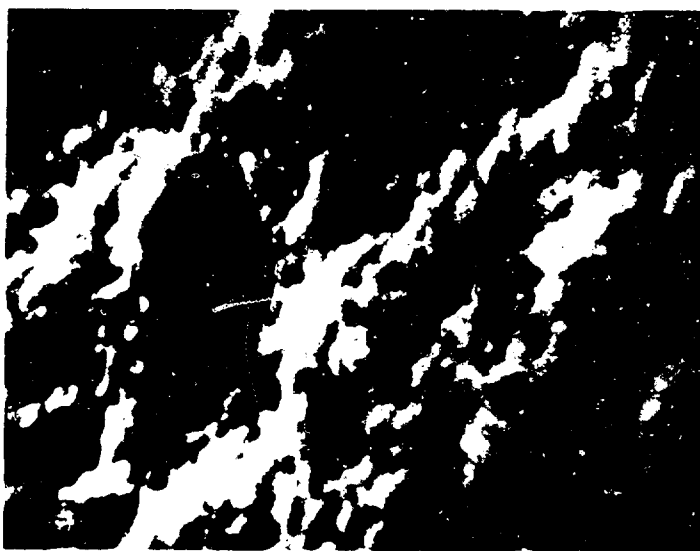


Fig. 7 Extruded Co_3O_4 - BeO Particle, Cleaved Surface
From RDS 34 Control, 5000X



Fig. 8 J. T. Baker Thermistor Grade Cobalt Oxide Powder
8.5 m²/gram, Unsintered
15,000X

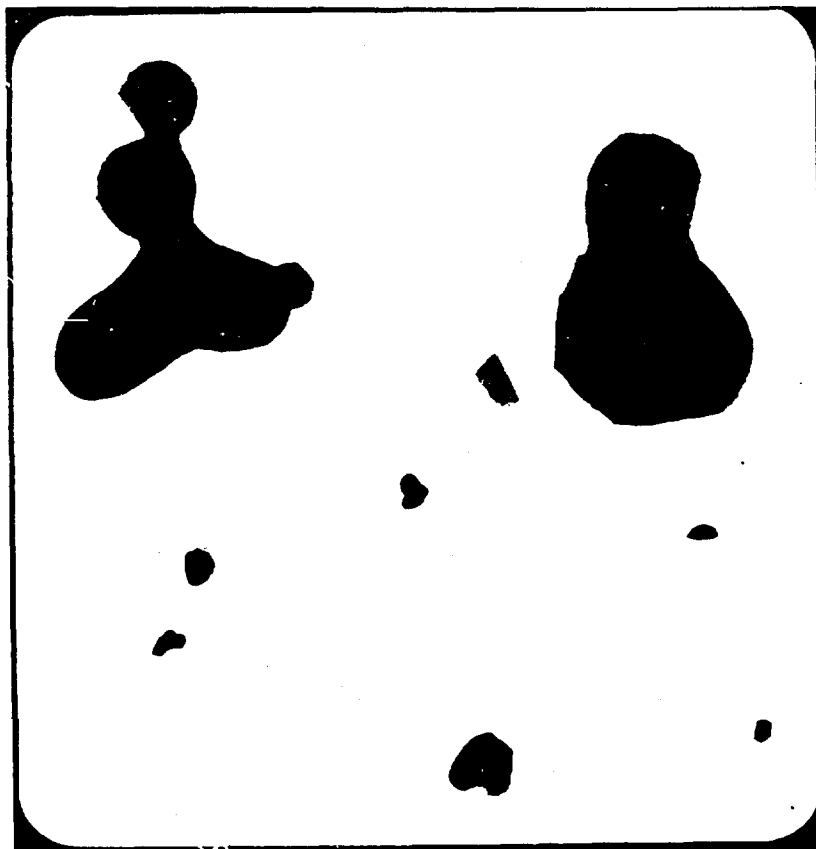


Fig. 9 J. T. Baker Thermistor Grade Cobalt Oxide Powder
Sintered for 1 hour at 1800°F, 15,000X



Fig. 10 Ball Milled Co_3O_4 Powder
2.83 m^2/gram Surface Area, 15,000X

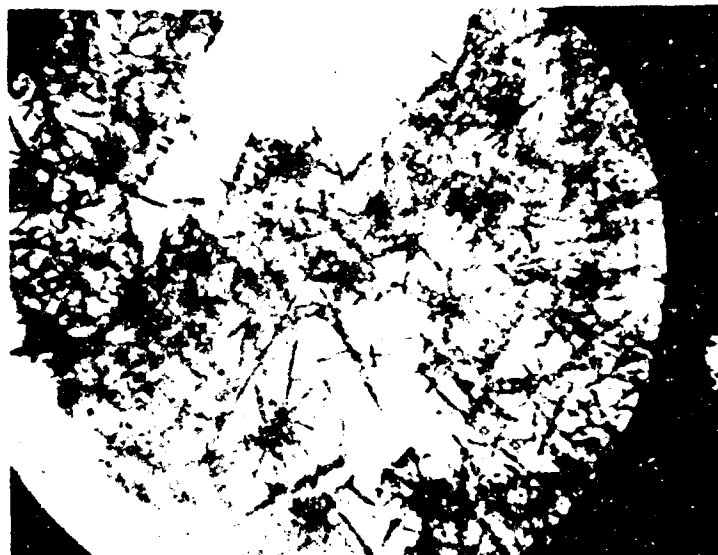


Fig. 11 Cobalt Oxalate Crystals from Precipitation of 0.04 M
Solution of $\text{Co}(\text{NO}_3)_2$ in 2M Oxalic Acid, 280X

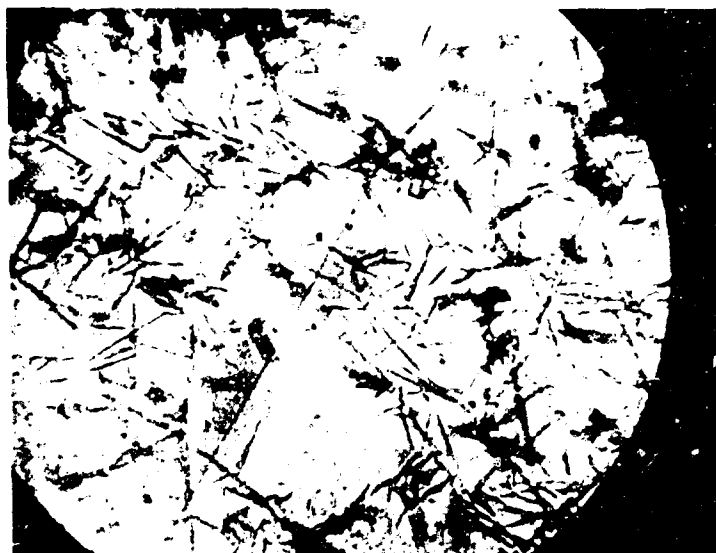


Fig. 12 Cobalt Oxalate Crystals from Precipitation of 0.2 M
Solution of $\text{Co}(\text{NO}_3)_2$ in 2M Oxalic Acid, 280X

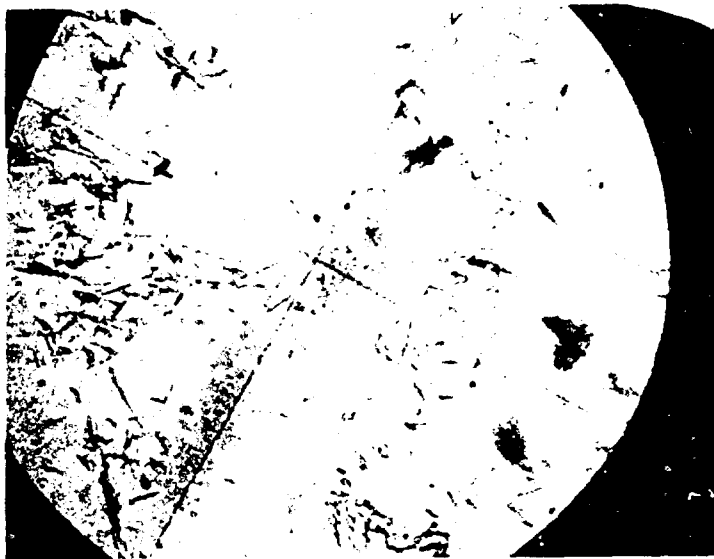


Fig. 13 Cobalt Oxalate Crystals from Precipitation of 1 M
Solution of $\text{Co}(\text{NO}_3)_2$ in 2 M Oxalic Acid, 280X

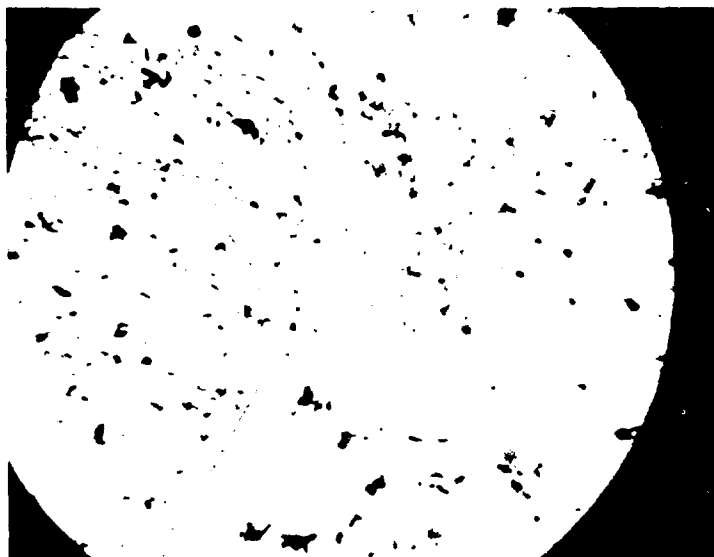


Fig. 14 Cobalt Oxalate from Fig. 13, Fired in Air
1.0 hours at 1800°F

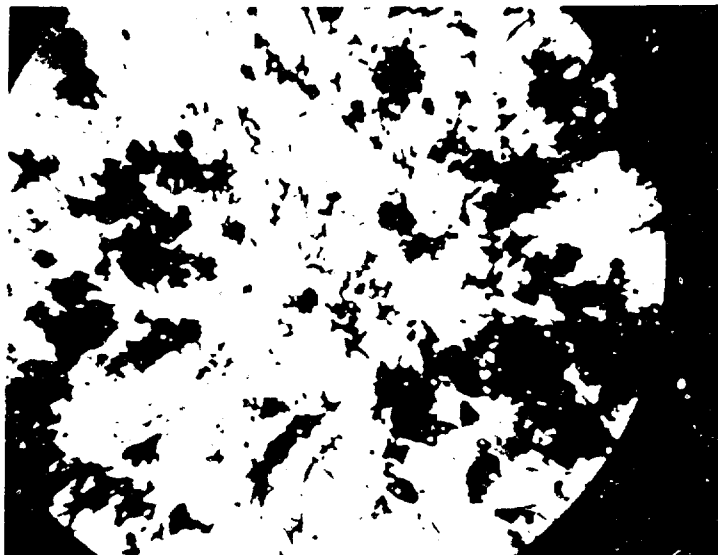


Fig. 15 Cobalt Oxalate From Fig. 13, Fired in Air
1.0 hours at 1100°F, 280X

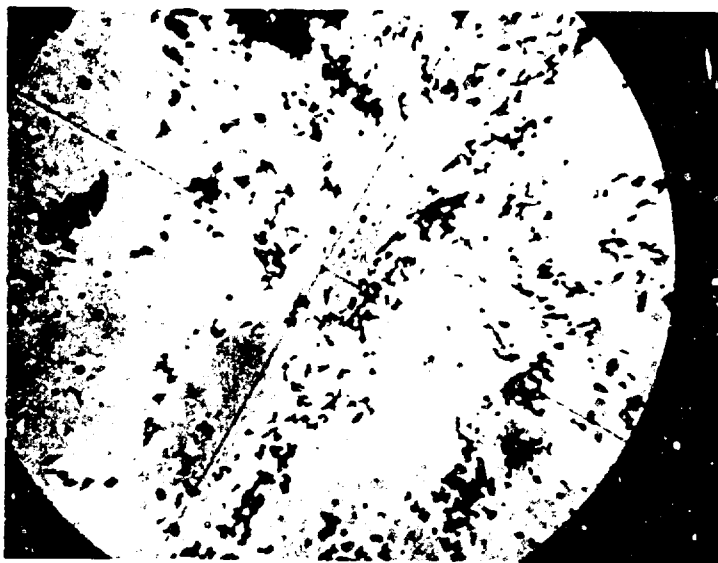


Fig. 16 Cobalt Oxalate From Fig. 13, Fired in Air
1.0 hours at 1100°F, 1.0 hours at 1800°F.
280X

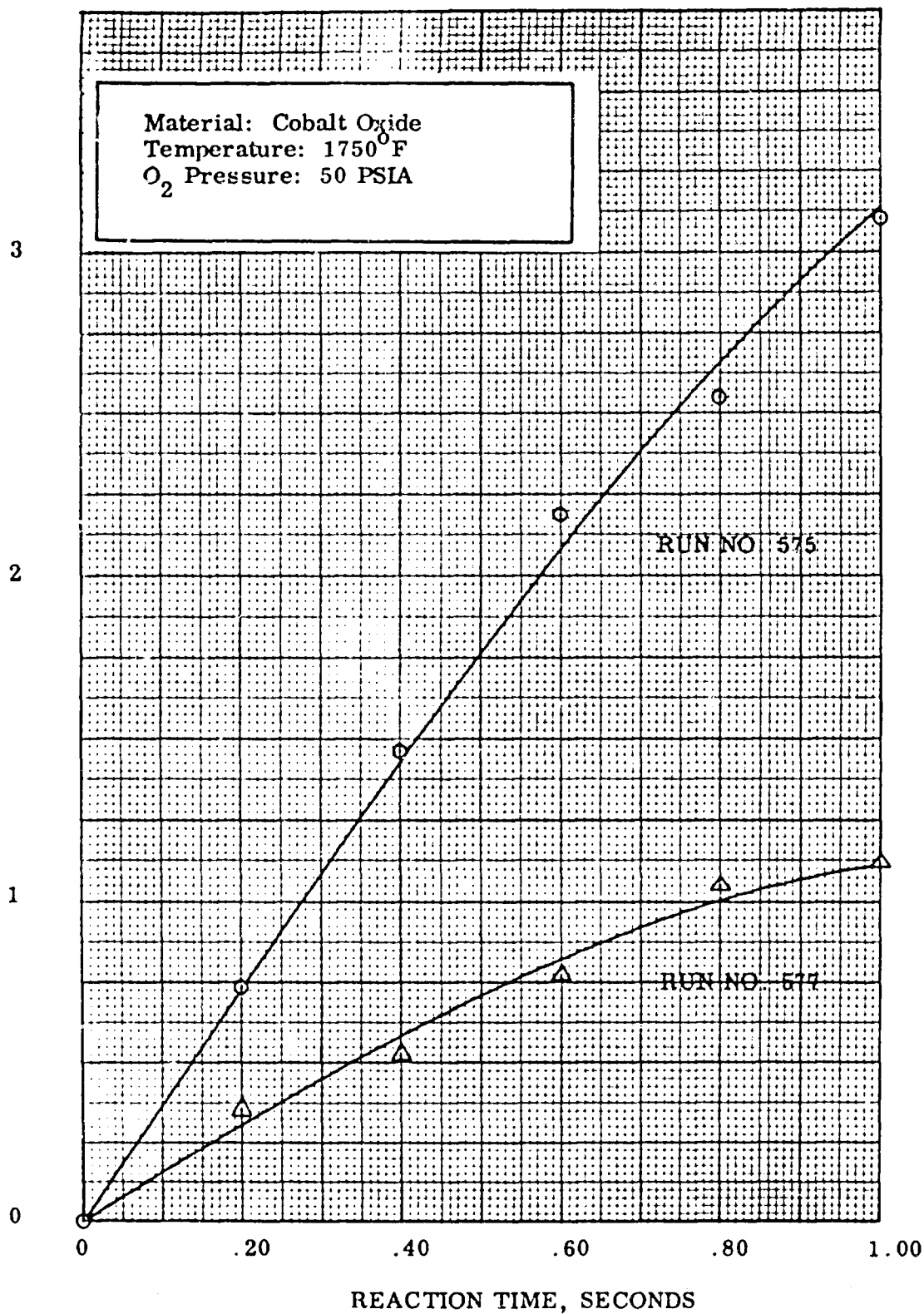


FIGURE 17

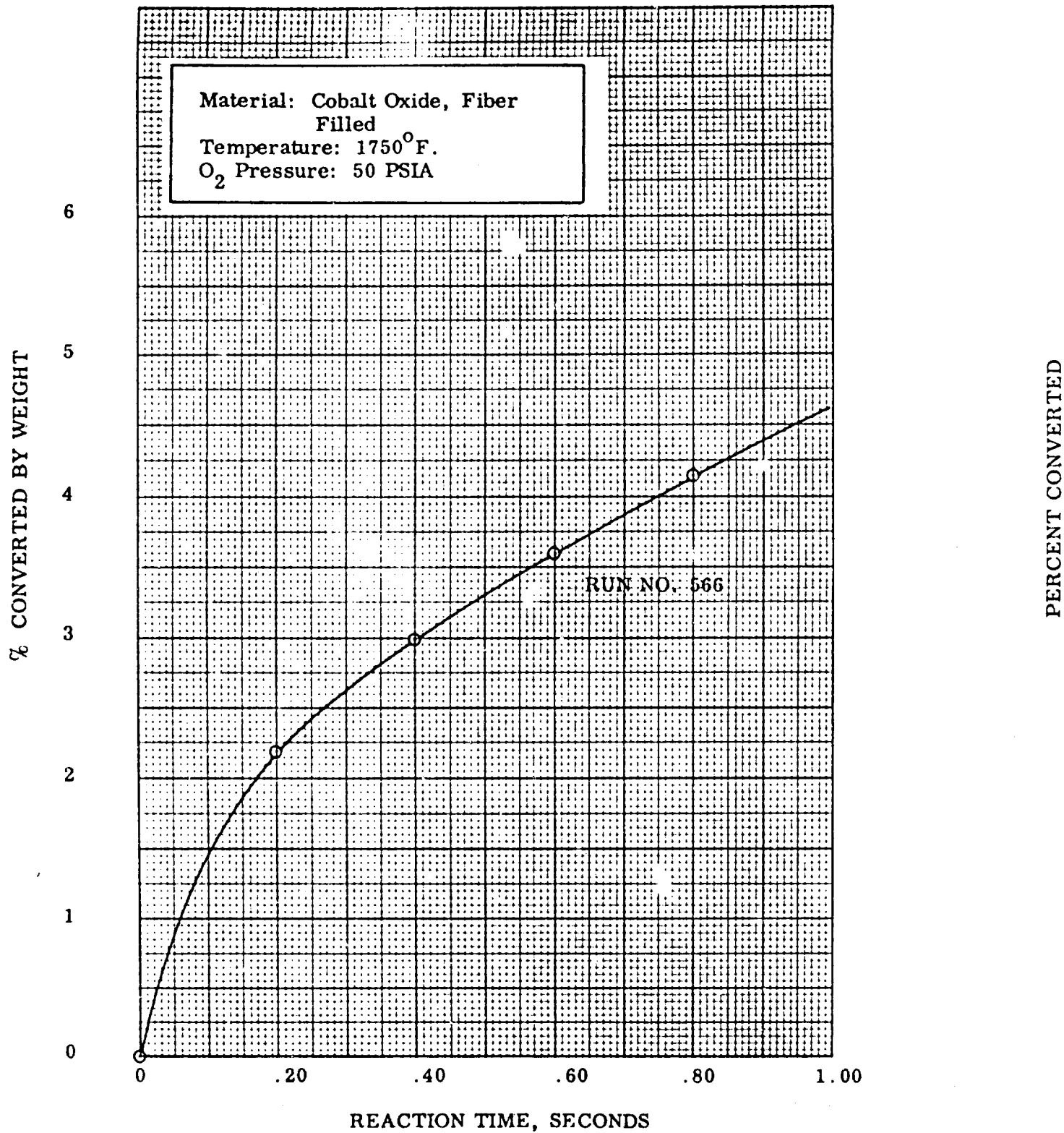


FIGURE 18

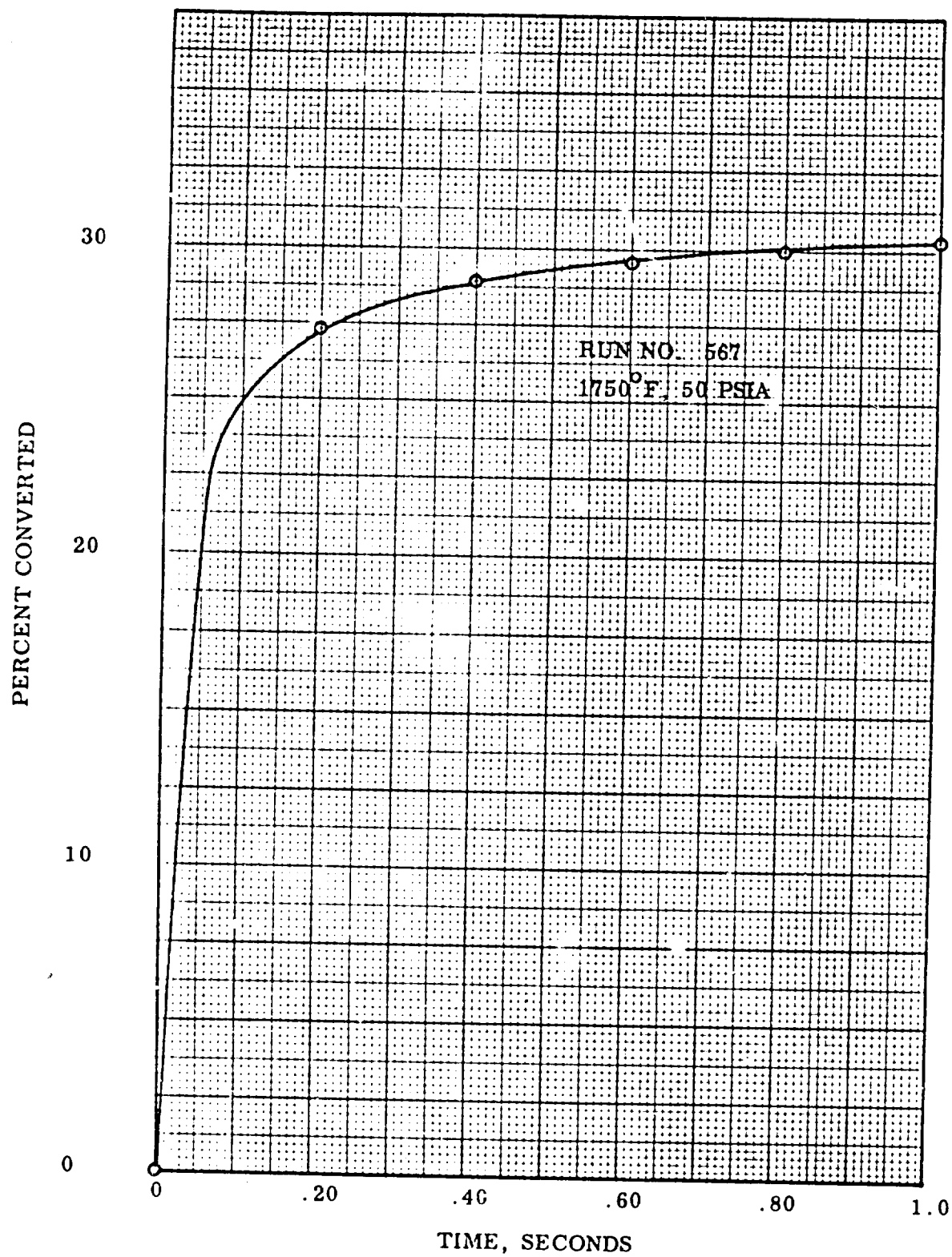


FIGURE 19

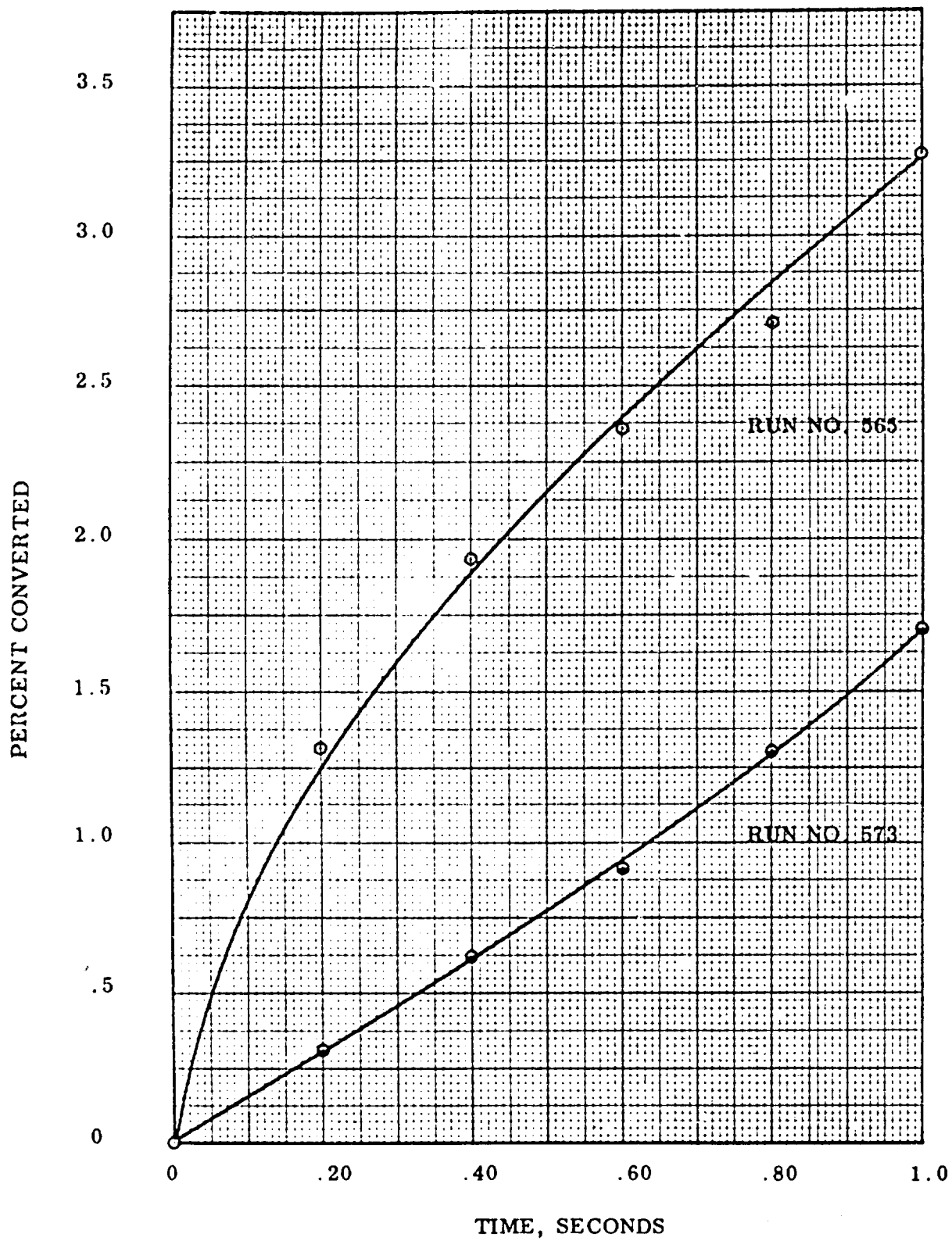


FIGURE 20

% CONVEI.TED BY WEIGHT

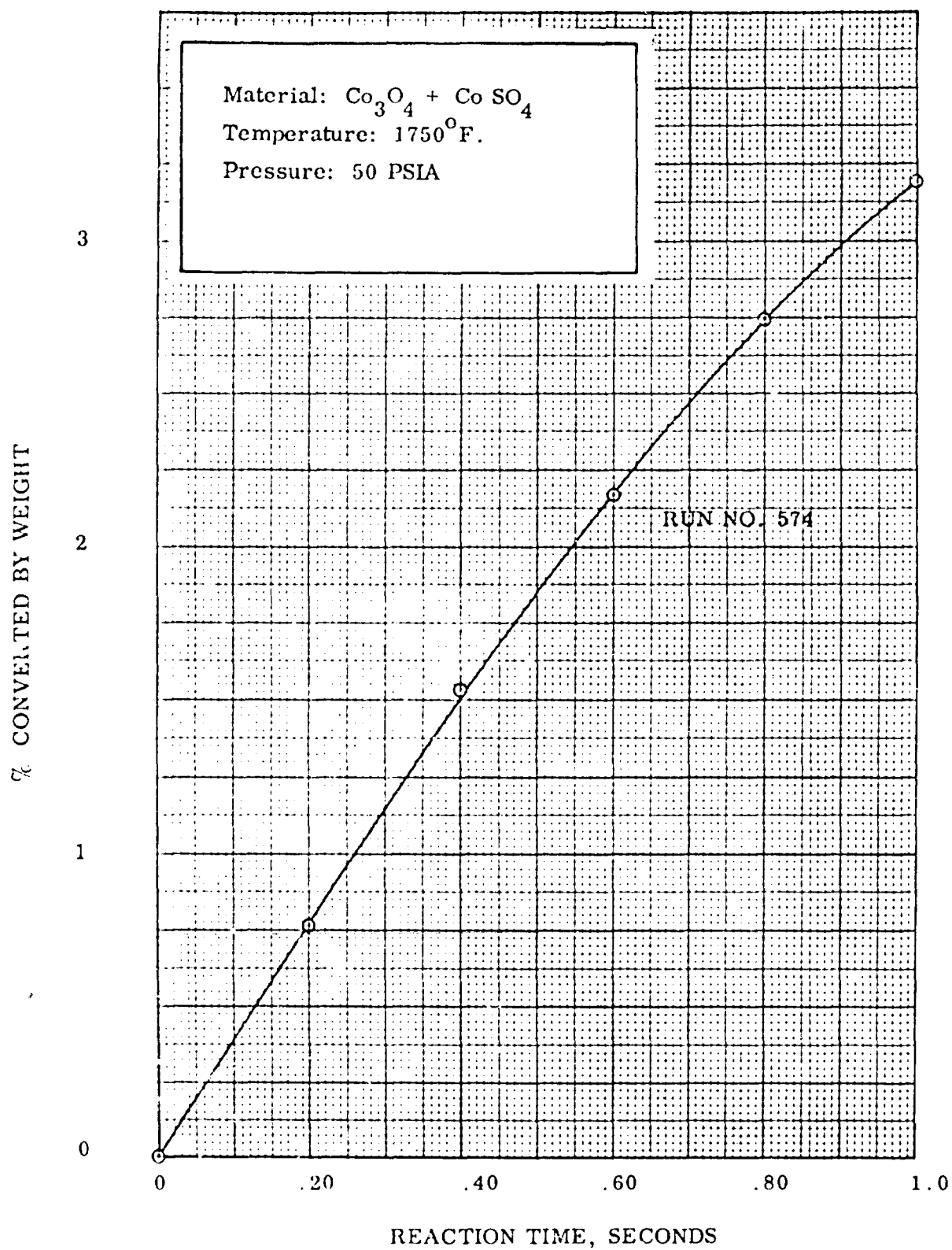


FIGURE 21

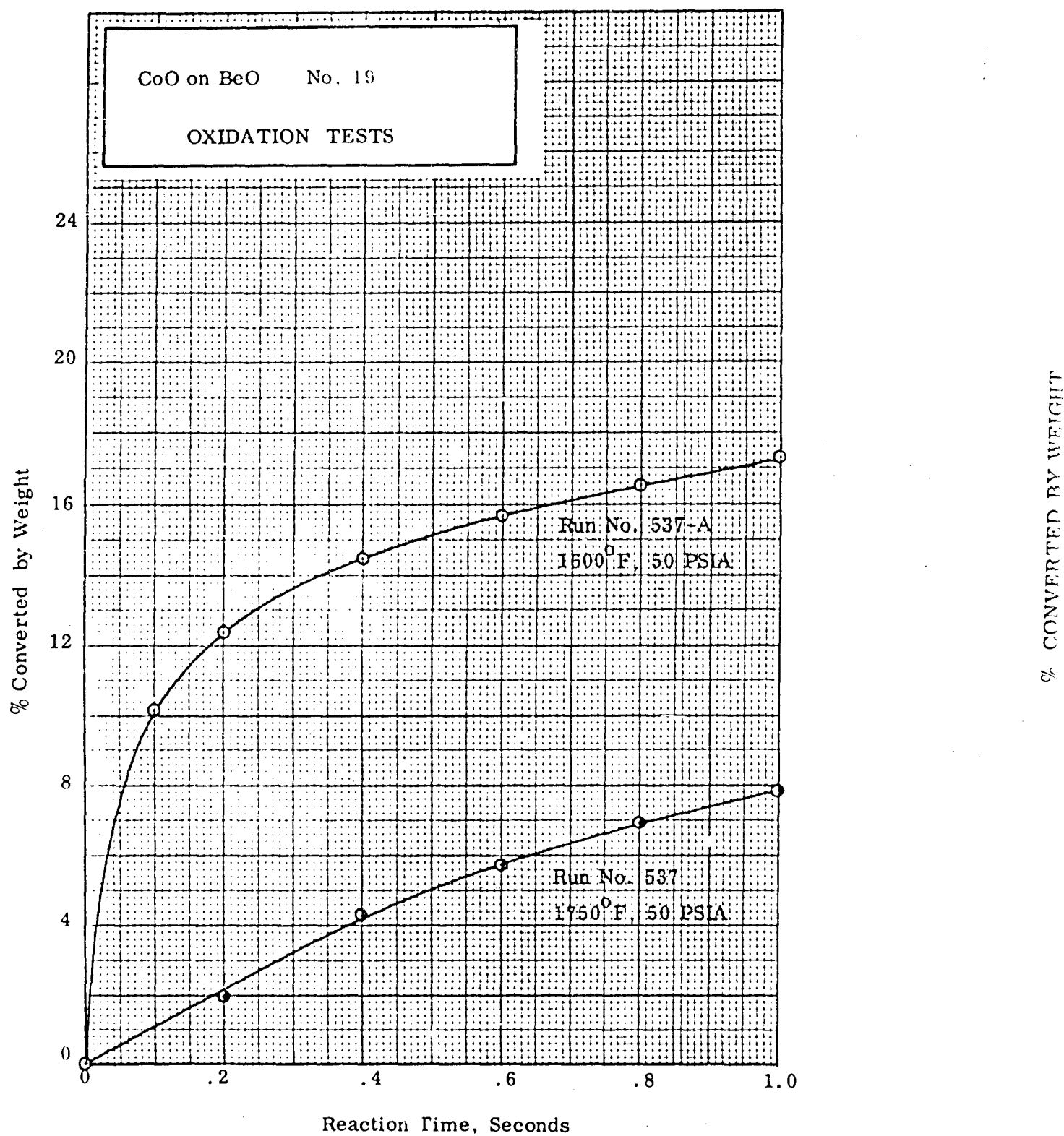


FIGURE 22

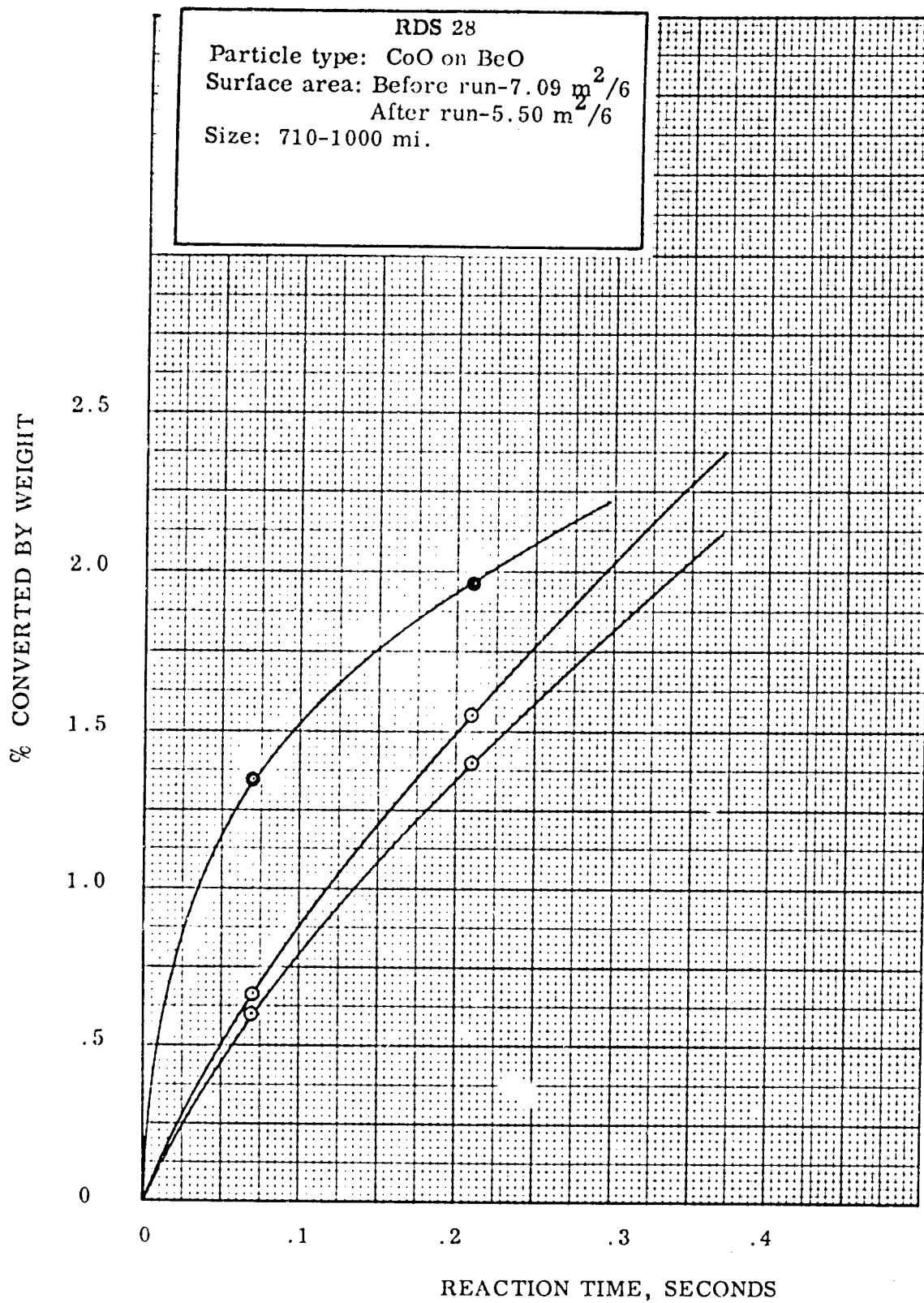


FIGURE 23

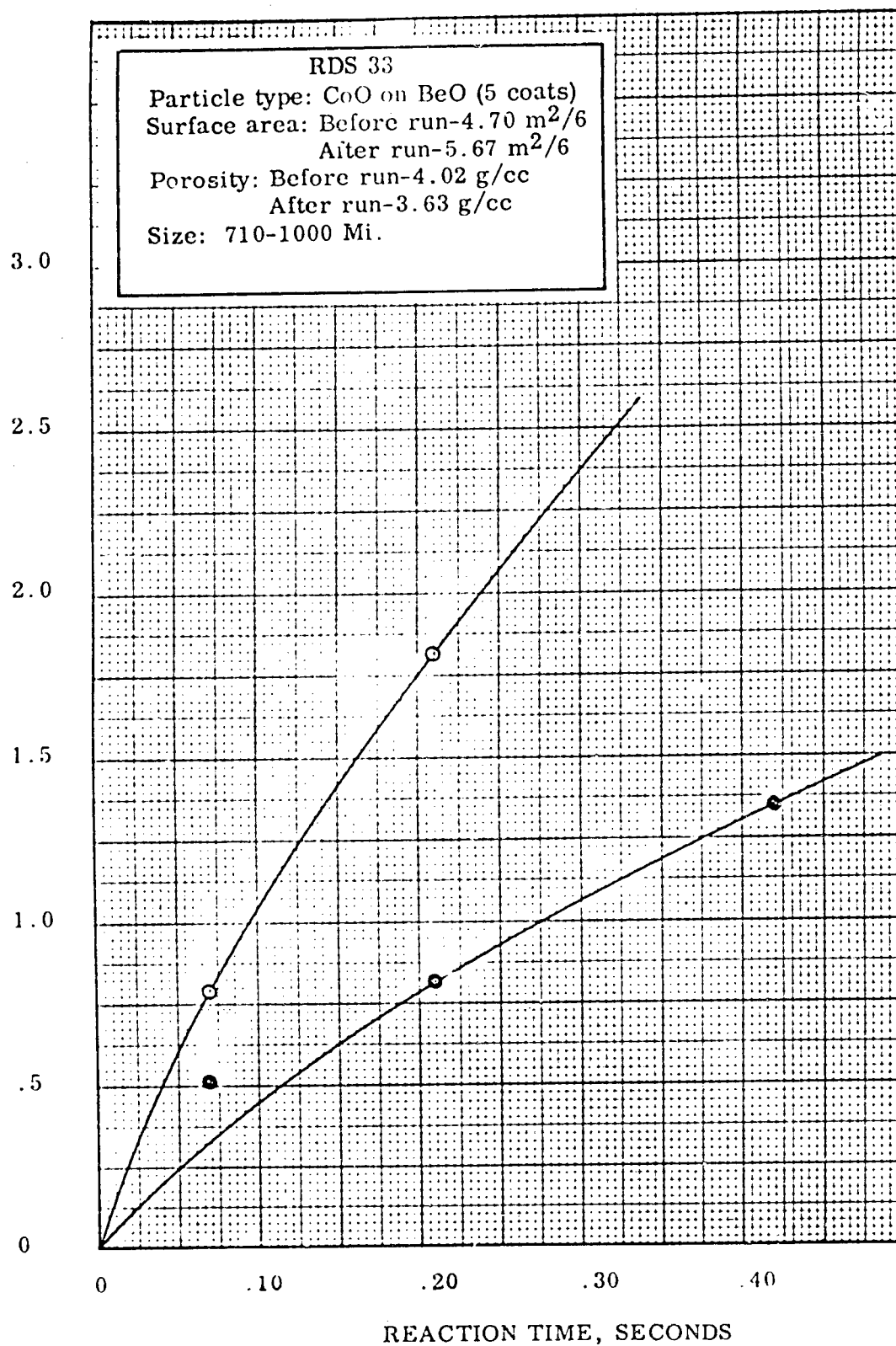


FIGURE 24

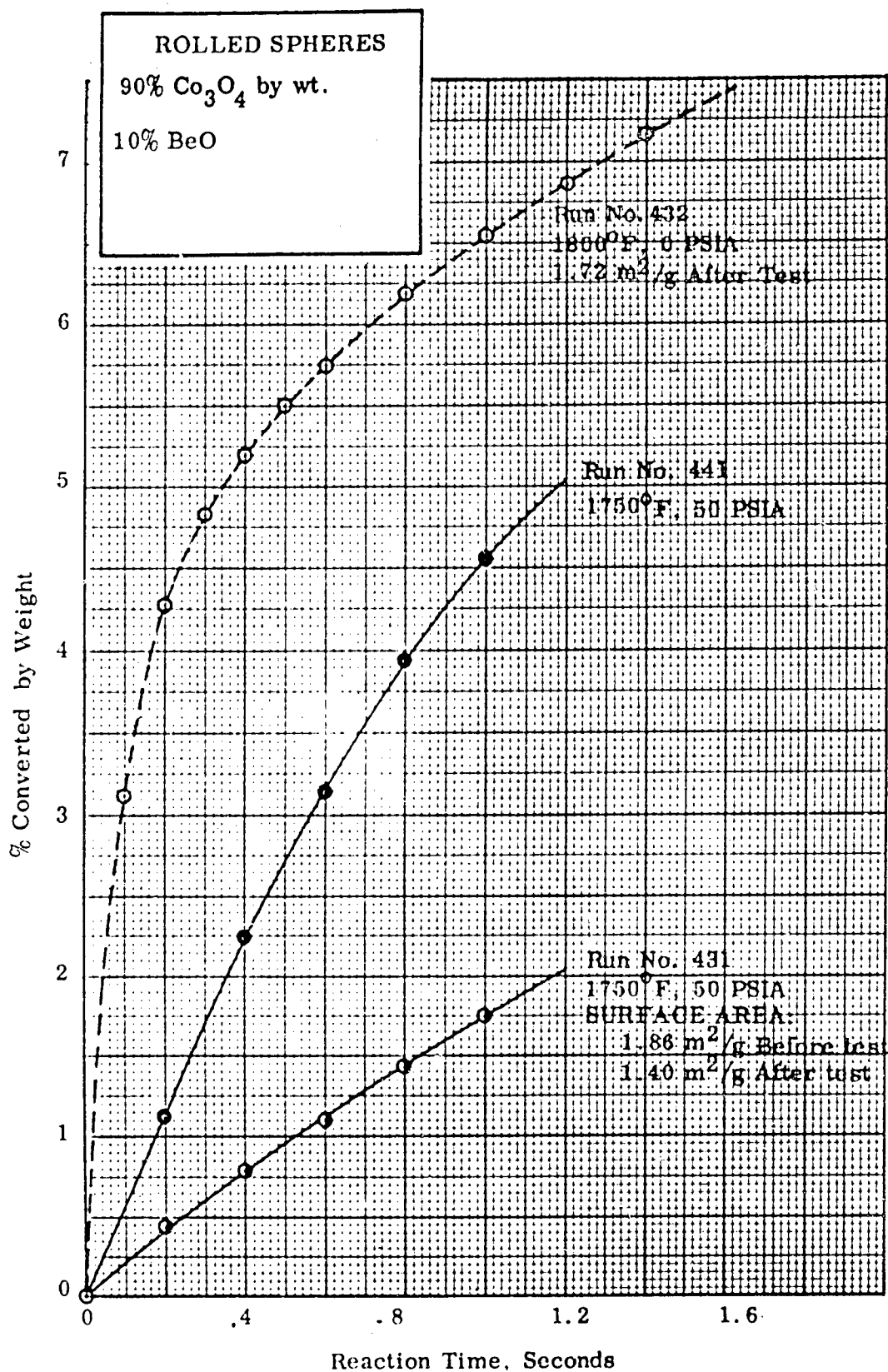


FIGURE 25

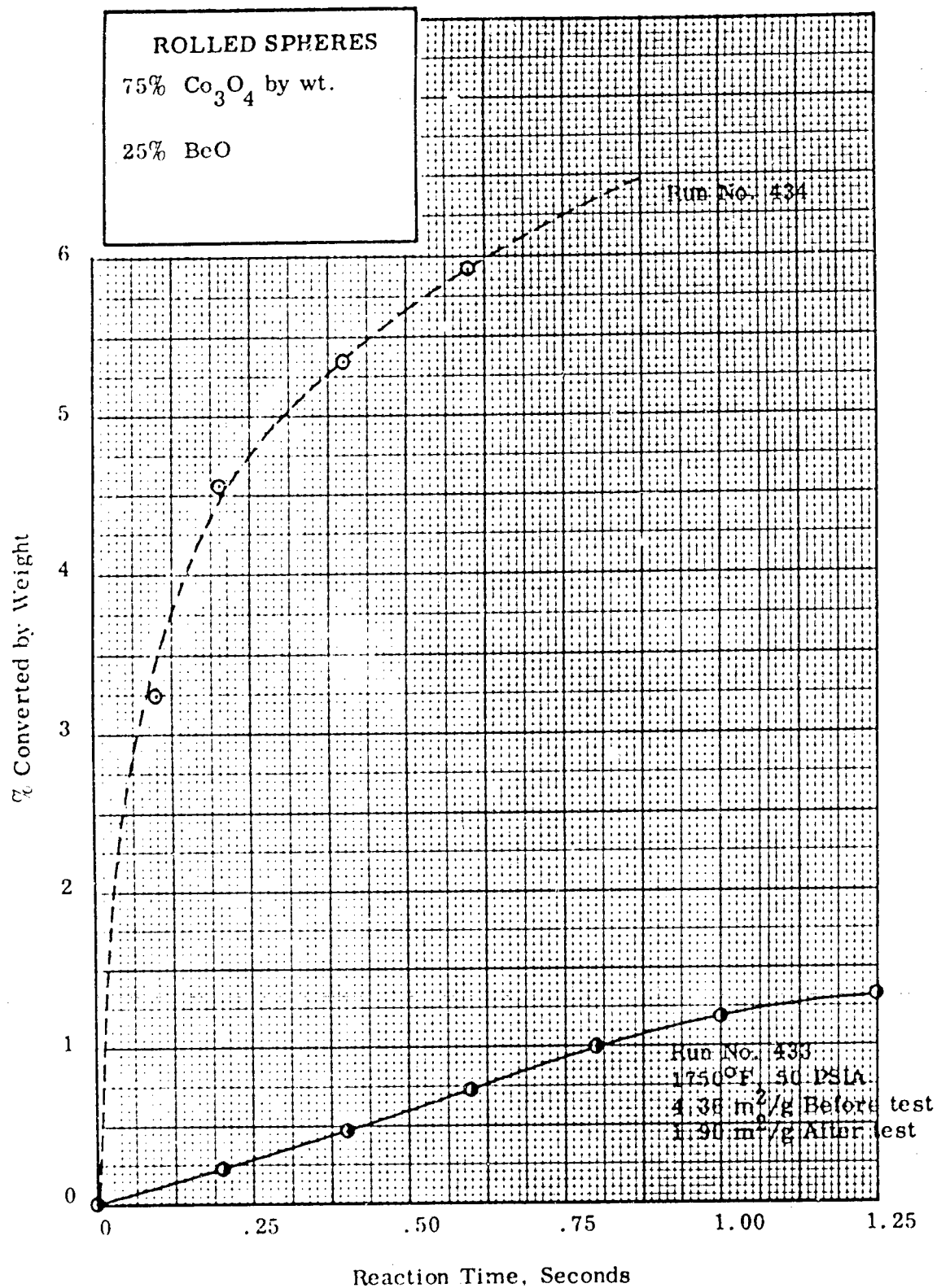


Figure 26

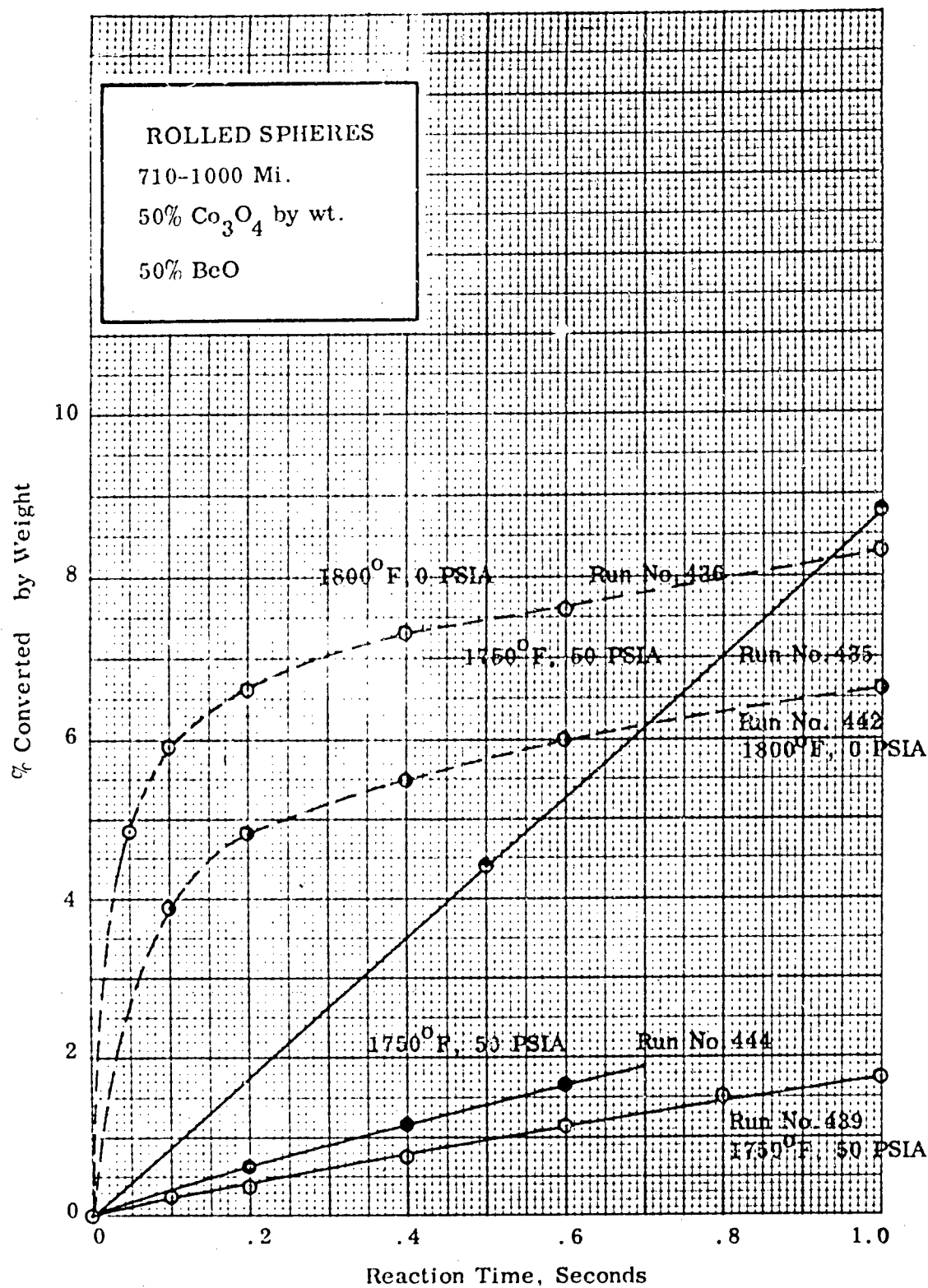


Figure 27

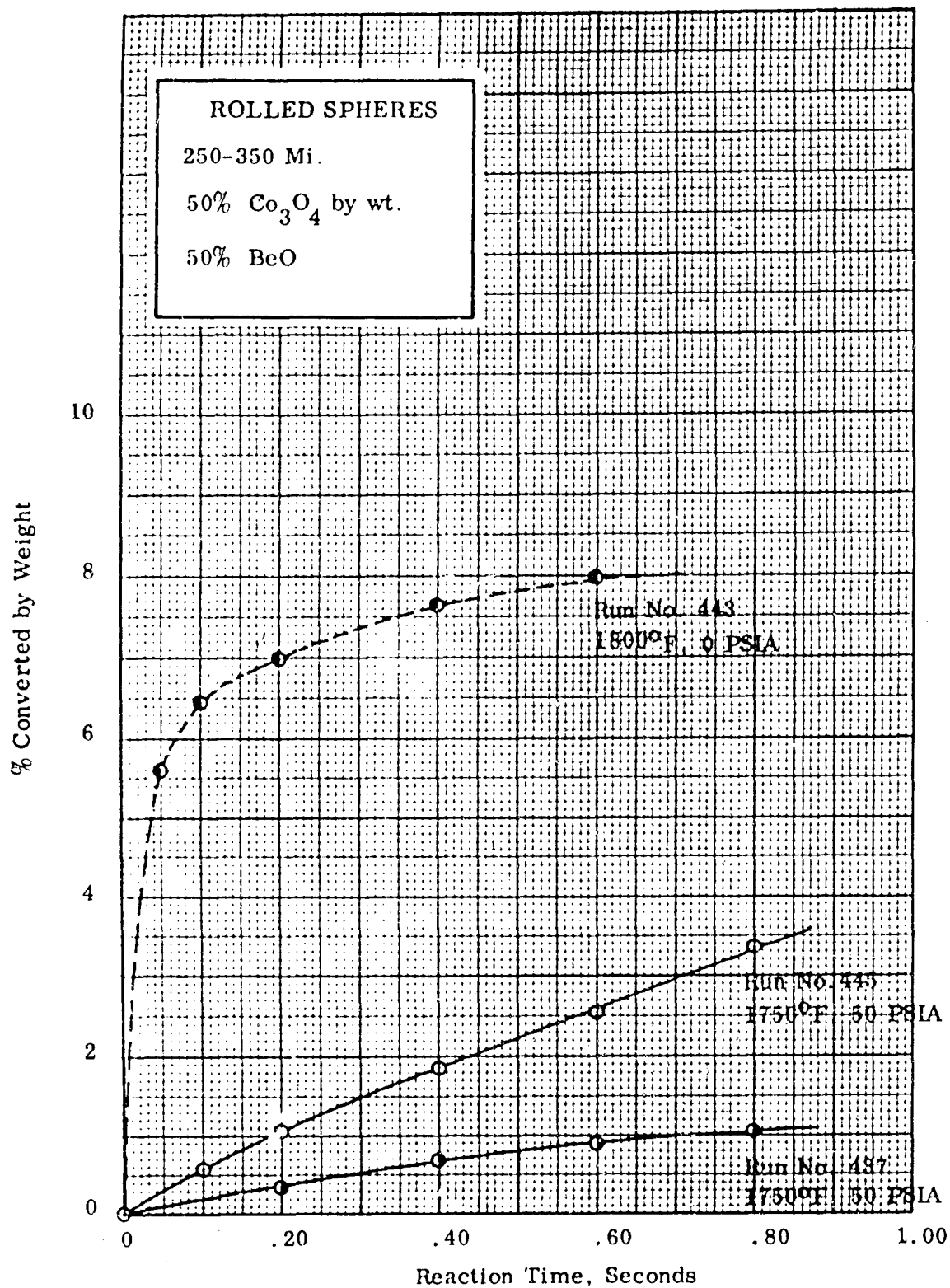


Figure 28

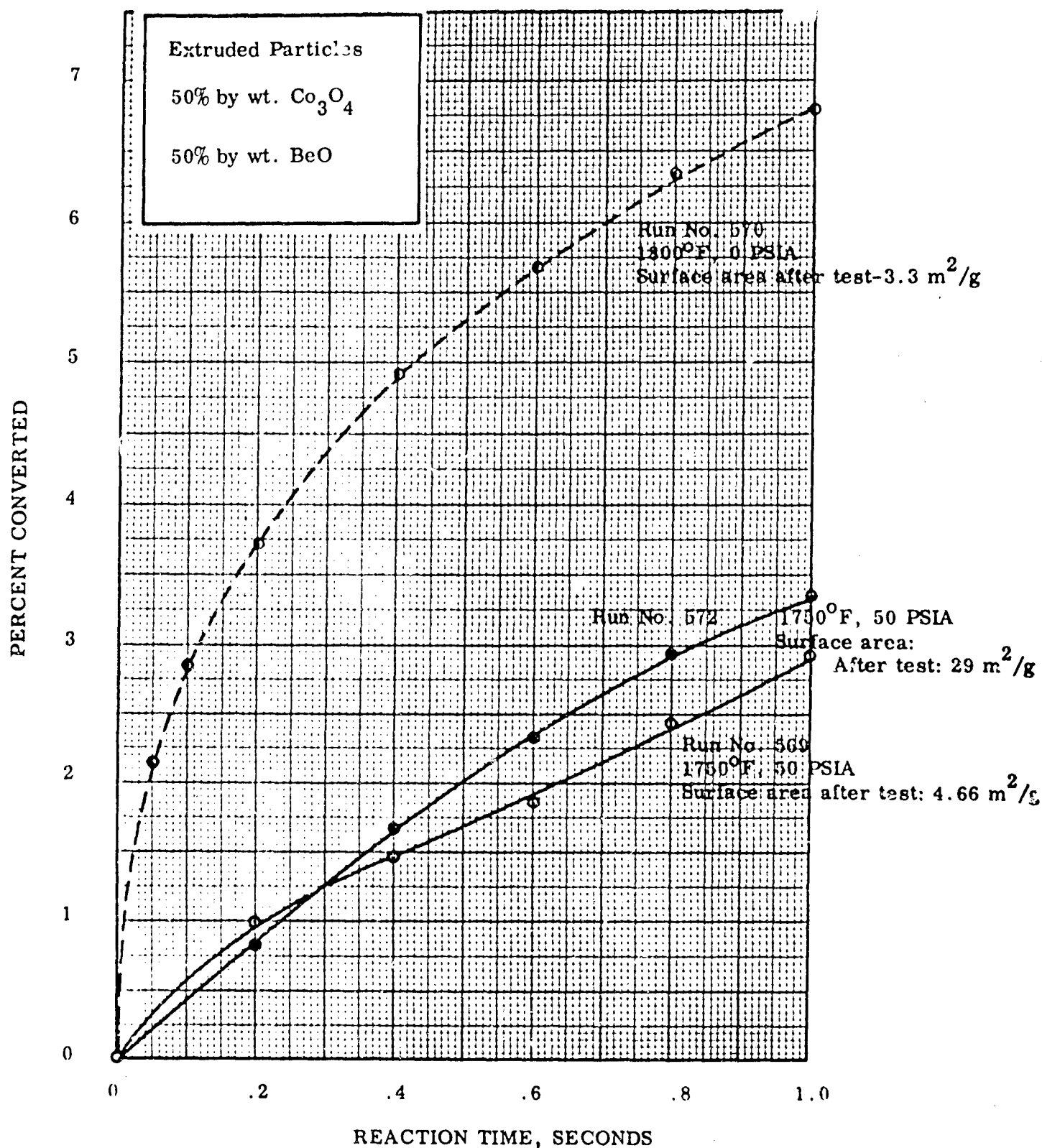


FIGURE 29

PERCENT CONVERTED

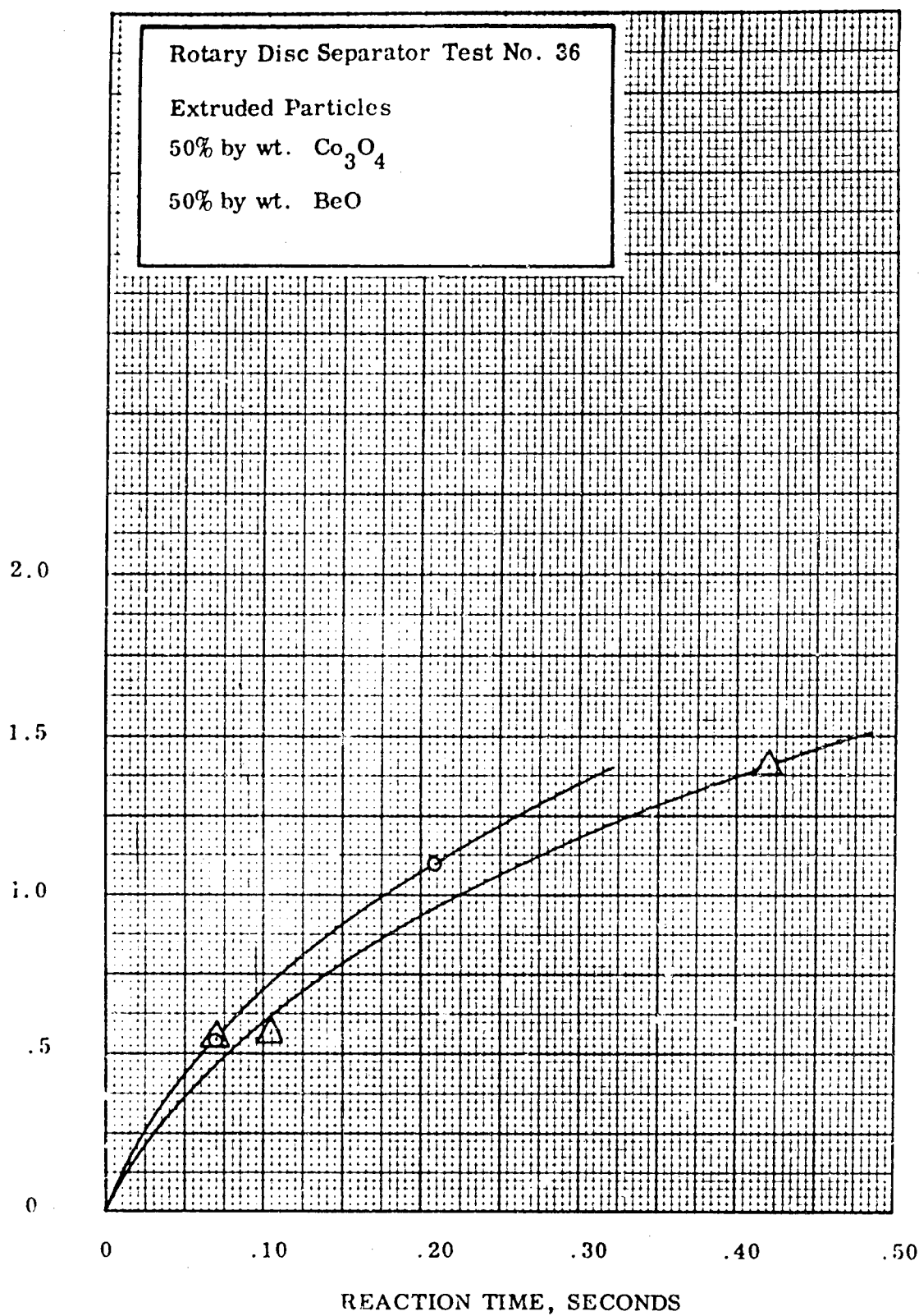


FIGURE 30

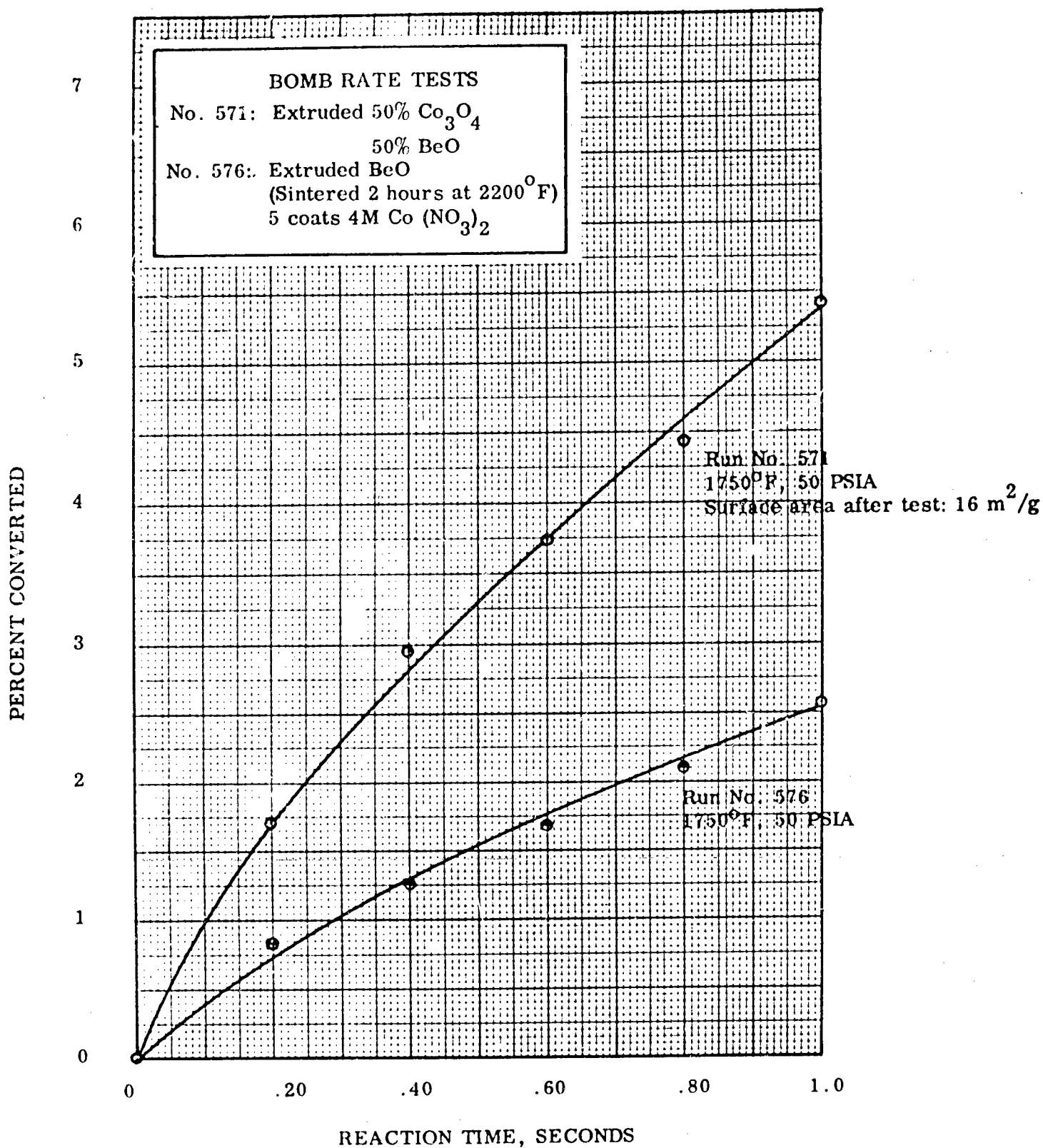


FIGURE 31

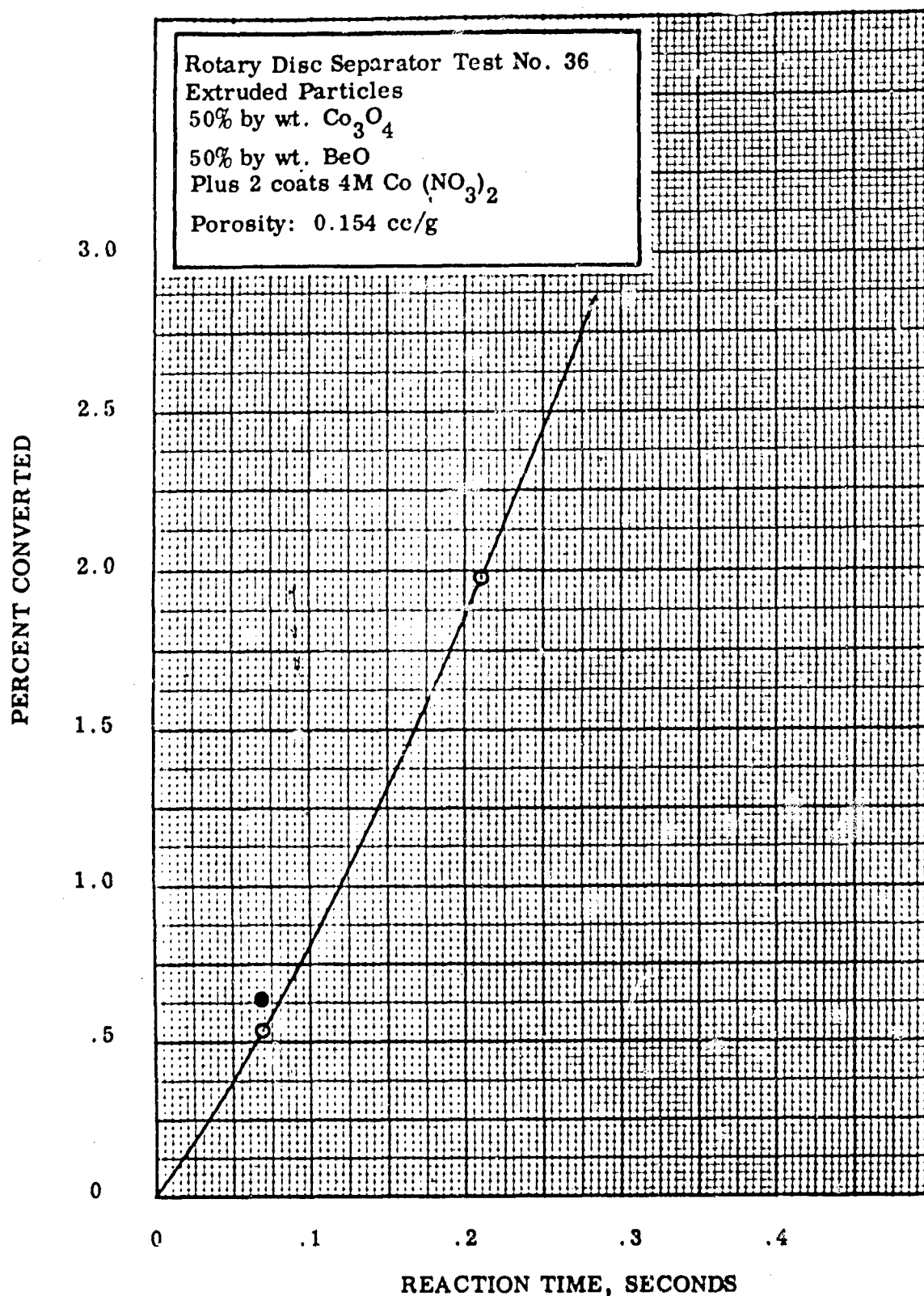


FIGURE 32