

R

September 22, 1941

CONFIDENTIAL

CLASSIFICATION CANCELLED OR

CHANGED TO UnclassifiedBY AUTH. CG-DAR-1 4318BY J. S. [unclear] APC 4/1/96DATE 3/15/96
By W. E. [unclear] ADD 3/15/96

Dr. C. C. Furnas
Sterling Chemical Laboratory
Yale University
New Haven, Connecticut

Dear Dr. Furnas:

As requested in your telephone conversation, we are sending a statement of the present development of the cobalt oxygen compound and figures for the cost of the proposed extension of the work.

The theoretical uptake of the 2 to 1 form of the compound is 4.9 per cent O_2 . The density of the unoxidized material is 1.66. At present we have a small automatic cyclé in operation using 573 grams. This has been running for some time off of undried air. It produces 0.61 cu. ft. of O_2 every 25 minutes (20 minutes charge, 5 minutes discharge) when activated with air at 60 lbs. (total airflow 8 cu. ft. air). One pound of the compound thus produces 2.65 lbs. of O_2 per 24 hours. With air at 20 lbs. pressure and the same rate of flow the activation period would be about 1 hour. Higher rates of airflow decrease the activation period. The interchanger consists of a bundle of half inch copper tubes filled with the material in the form of pellets. The tubes are in a jacket so that they may be cooled with water or heated with steam (the pressure of O_2 at 100° is 80 lbs.). The heat of the oxygen reaction is 12 kcal. per mole O_2 .

In order that you can understand the manufacturing requirements, the following outline of the process at present used by Dr. Calvin is given.

Preparation of 2 to 1 compound. The preparation of useful active material in the form of pellets can be described in terms of the following eight operations.

1. Mixing stoichiometric quantities of the reagents in alcohol solution at $\sim 60^\circ C$. and permitting recrystallization of the initially formed gel.
2. Filtration and washing of this originally formed inactive material.
3. Recrystallization by boiling with three times its weight of pyridine and cooling in the absence of air.

1260133

CONFIDENTIAL

Dr. C. C. Furnas
September 22, 1941
page 2

4. Filtration of the pyridinate in the absence of air and washing with ether.

5. Drying off the ether by pumping through traps before complete exposure of the cake to air.

6. Pressing and granulating the pyridinate cake.

7. Converting the granulated material into pellets.

8. Activation of the pellets by depyridination in a rapid air stream at 160°C.

The pressing, granulating and pellet formation may not be necessary if the material can be used as a powder in a slow moving air.

Cost of manufacture. Our present cost of preparation is high as we have been using CP grade material bought in small lots and have employed no recovery of the various solvents. The following figures are for a 752 g. (theoretical) preparation.

	Price/Kg	Amts. in Kg	Cost(dollars)
Co(Ac) ₂ 4H ₂ O	6.88	0.576	3.97
Salicylaldehyde	6.00	0.565	3.99
Ethylenediamine 60% solution	5.83	0.139	0.82
			\$8.78

This is \$5.29 per lb. for 100% yield or \$6.22 per lb. actual yield. With the purchase of materials in large quantities, this figure should drop to \$2.00 to \$2.50 per pound of actual yield.

The process requires the following solvents for 752 g. of active material.

	Cost	Amts.	Cost
Ethanol	\$0.50 per gal.	2.02 gal.	\$ 1.01
Pyridine	2.11 per lb.	5.74 lbs.	12.10
Ether	0.64 per gal.	1.5 gal.	0.96
			\$14.07

CONFIDENTIAL

Dr. C. C. Furnas
September 22, 1941
page 3

\$14.07 for 752 g. or \$8.49 per lb. of material. This makes our present cost \$14.71 per pound. Thus the commercial production of the compound will amount to \$2.00 to \$2.50 plus operating cost and recovery of solvents.

Application of compound to Naval problem. For storage of oxygen a packing density of 0.5 would give approximately 1.5 lb. of O₂ per cu. ft. Thus 20,000 lbs. of O₂ would require 400,000 lbs. of cobalt complex and a storage space of 13,300 cu. ft. Interchanges which would give a density of 0.5 of compound would not be highly efficient and the period of charge might be an hour or more.

We would like to know if the Navy considers the above storage facilities a possible solution to their problem.

If used for the production of O₂ in connection with a liquefying cycle 17,000 lbs. of the cobalt compound working on a 25 minute cycle would produce 2000 lbs. of O₂ per hour. Such a cycle could be developed for Professor Giaque's liquefaction process.

Future work. Both of the above programs require a large scale manufacture of the cobalt compound. The first step should be a pilot plant handling about 25 lb. lots. This work should be accompanied by the study of appropriate interchangers for O₂ storage and also for rapid O₂ production.

Under our contract \$500 was allotted for material and equipment. We have now spent about \$1500 additional from departmental funds. It is highly essential that Dr. Calvin supervise the pilot plant for the manufacturing process. At the same time we would like to build interchangers to handle about 50 lb. changes. In other words we would like funds to manufacture about 100 lbs. of the material in 25 lb. lots and then use this in various designs of interchangers. This work would require about \$3000 for the preparation (including a pellet machine) and \$3000 for material and mechanics to build interchangers. We would not need additional assistants on our present contract which runs to March 1st.

If the work is to be pushed, plans should also be made for the large scale manufacture, probably as contracts with a chemical manufacturing company. To produce a ton of the compound we would have to purchase a large filter press, large driser, and stills for solvent recovery, total cost about \$7000.

1260135

CONFIDENTIAL

Dr. C. C. Furnas
September 22, 1941
page 4

Supplies and labor would cost us \$25,000 to \$40,000 more. We are willing to do this if it seems expedient. It might be possible to have the work done at the Dow Chemical Plant near Berkeley but an Eastern chemical company would have certain advantages, and some contact could probably be established between Dr. Calvin and their production. It should be emphasized that the preparation of the 2 to 1 compound without getting either the 3 to 1 or inactive form requires considerable familiarity with the preparation.

Sincerely yours,

Wendell M. Latimer

P.S. We have just received a letter from Dr. Gilliland of M.I.T. We are unable to see that his results are significant. Our equilibrium data are quite consistent when only one active form is present. The rate problem is of course a complex one.

copy to Roger Adams

1260136