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SECTION B-7

PHYSICAL-CHEMICAL

AND

CHEMICAL ENGINEERING PROBLEMS

Summary of Progress January 15 to February 15, 1942

Subsection B-7-B - Oxygen Problems - E. P. Stevenson, Chairman.

Problem AC-12 - The Manufacture of Oxygen While in Flight for Use of the Combat Crew.

Progress Report: Being prepared for distribution.

Problem NL-B42 - The Development of Chemicals and Apparatus for Regenerative Oxygen Production.

Summary: Strictly speaking the first of the problems listed above (The Manufacture of Oxygen While in Flight for the Use of the Combat Crew) might be considered quiescent because of the necessity of a great deal of background work before this problem, which is one of the most difficult in the whole program, can really be handled. However, the second of the problems (The development of Chemicals and Apparatus for Regenerative Oxygen Production) is very active indeed. The work on the chemical studies of compounds which may be used for regenerative oxygen production is progressing at three different university laboratories (University of California, Iowa State College, and the University of California at Los Angeles).

The compound which was first investigated in this study is a cobalt chelate compound made by reaction between ethylenediamine, salicylaldehyde, and a cobalt salt which will readily absorb oxygen in the temperature range

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of 0° to 35°C., and at air pressures of 20 pounds per square inch or higher, and will release pure oxygen at pressures above atmospheric and temperatures at 80°C. or higher. As far as this compound is concerned, the work at the three university laboratories is largely focused on preparing the compound in a more pure state than formerly and attempts to improve the physical and chemical characteristics by minor changes in structure. No significant improvements in the compound have yet been reported with the exception of the improvements in purity and percentage yield of the synthesis.

Work at two of the laboratories (Iowa State College and University of California) has also been focused on the preparation of compounds related to this one initially used. Various modifications in the organic part of the structure have been made as well as the use of metals other than cobalt in the metallo-organic complex. The metals iron and manganese have been studied especially in this respect. The results may be summarized briefly by stating that none of the variance either in the organic or metal parts of the molecule have yielded compounds which show any appreciable or hopeful characteristics as oxygen absorbers. This is rather disappointing particularly in the matter of substitution of other metals, for it would be very desirable in the event of large scale use of this material to have a more common metal such as iron which might be usable in the preparation of the compound. However, the possibility of variance in the field is so great that this should not be taken as an indication that there is no hope of developing other compounds which may be equally satisfactory and more practical for use.

The active life of the compound is a matter of great importance in its application. Preliminary studies show a definite falling off in the activity of the compound of about 15% in a life of 200 absorption and desorption cycles (University of California). Preliminary studies on the cause of the decrease in activity indicated that the presence of small amounts of water in the adsorbed air seemed to be largely responsible for this phenomenon. Operating on that suggestion, other life cycles were run with carefully dried air (M.I.T.), and it was found that the activity decreased about 10% in 200 cycles, then leveled off; at the end of 1300 cycles the total decrease in activity was about 20%. Other studies (Iowa State College) showed that the material which had been deactivated because of the presence of water could be reactivated by heating in a vacuum to a temperature of 180 to 190°. Studies are being continued there to see if this reactivation cannot take place in an inert gas instead of a vacuum. The results of the decrease in activity may be summarized by saying that the apparent decreasing life of the material in use may be somewhat troublesome, but the prospects are that it will be entirely satisfactory for practical application.

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Another difficulty, which it is hoped will only be minor, has been found in the evolution of small amounts of carbon monoxide in the regenerated oxygen. It has not yet been definitely decided whether this amount of carbon monoxide is in all cases below the toxic limit, but it is evident that this item must be investigated. A unit is being supplied to the toxicology section for animal tests to determine the biological effects of the oxygen produced by this chemical. Studies are also progressing (A.D.Little, Inc.) on the careful analysis of the carbon monoxide produced at different parts of the regenerating cycle. Studies on the ways and means of eliminating this small amount of carbon monoxide will be vigorously pursued. Apparently the source of it is the slow oxidation of the organic part of the compound, and it thus may not be possible to entirely eliminate its formation. This indicates that further studies must be made in devices for removing any carbon monoxide which may be formed from the gas stream. These precautions for the elimination of carbon monoxide are obviously necessary only for the case where oxygen is to be used for breathing. Where the application is that of welding or cutting or general repair work, it will not be necessary to take any precautions in regard to the elimination of traces of carbon monoxide (the construction of the first test unit for the use of the Armed Forces using the regenerative chemical has been held up because of priority difficulties, which seems to be an old story in most lines of activity. This unit is to produce 5 cubic feet of oxygen per minute for welding and cutting, and is to be tested at the Naval Research Laboratory).

A satisfactory method for the production of the cobalt complex has been developed without the use of any organic solvents (Rumford Chemical Co.). It has been found that by the use of this method the chemical can be produced in a state of uniform purity, and it can be brought into the proper physical state for use by putting through a standard pelleting machine. Material so prepared shows a small though not significant decrease in the rate of absorption of oxygen. The equipment for the production of this chemical at the rate of some 300 pounds per day is now ready for use, but here again a bottle-neck has developed in the priority situation in obtaining a machine for pelleting the final product for use. Steps are being taken to remove these priority difficuties as rapidly as possible.

The Air Corps and the Engineer's Board have come forward with definite recommendations for new units to be developed. As a consequence, three different units are now anticipated. One will produce about 1000 cu. ft. of oxygen per hour, which is to be compressed to 1800 pounds per sq. in. for delivery to oxygen bottles. This unit is to be designed to be completely self-contained, both as to power source, mediums for heating and cooling, etc., and is to be placed on a truck. It is the intention

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that the maximum weight of such a unit shall not exceed 6000 pounds. The second type of unit is one which will produce of the order of 300 to 500 cu. ft. of oxygen per hour compressed to 1800 pounds per sq. in., and to be so constructed that it can be easily dissembled, the maximum weight of any one piece being not over 250 pounds. This is to be a type of unit which may be transported by bomber or transport plane to isolated bases. The third type of unit under active consideration is one which will produce approximately 100 cu. ft. of oxygen per hour, which is to be assembled for use on a bomber while in flight. The weight restrictions here are quite severe, namely that the unit should not weigh more than 300 pounds. The space limitations are also very severe. The third unit presents problems which will be more difficult to solve than the other two.

Active work is going forward on the design of the first two types of units mentioned above by the M. W. Kellogg Co. The work has not yet progressed to the point where any definite predictions on the delivery date of the first unit can be made.

OXYGEN PRODUCTION BY MECHANICAL METHODS

Problem NL-B6 - Equipment for Manufacture of Liquid Oxygen.
Progress Reports: None

Summary: Three projects which were formerly under Section D-3 are now actively working under Section B-7. These are studies of (1) a cascade cycle using auxiliary refrigerants (University of California), (2) a cycle using an auxiliary gaseous refrigerant (M.I.T.), and a system using a modified Claude cycle (Air Reduction Co.). The work on the cascade system (University of California) is being confined at present to a study of a system using two refrigerants: methane and ethane. This cycle has the advantage of being quick starting and may have particular advantages in the production of liquid oxygen. A system using auxiliary gaseous refrigerants has been intensively studied some time back, and the work has been coupled with the development of four new and unique devices: (1) a free floating piston compressor, which can operate in a hermetically sealed system without lubrication, (2) a corresponding type of free floating piston expander, (3) a new type of heat interchanger which is very compact and should be very advantageous for use on board ship, particularly in confined spaces, and (4) a rotating distilling column, which from preliminary results should give high production of oxygen in a very small space, and should also be satisfactory for use on board ships where the effect of pitch and roll must be considered. This system and the devices are now in the state where further intensive engineering development is called for.

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The work at the Air Reduction Co. thus far has been confined to the study of distilling columns for the production of oxygen which might be satisfactorily operated on shipboard under conditions of violent roll and pitch. Various combinations of packing and bubble cap columns have been studied thus far, and satisfactory progress has been made, though the ultimate answer has probably not yet been found. The experimental work on the complete modified Claude cycle for the production of oxygen has not yet begun. It is anticipated that it will involve the use of a turbo-expander, which will require further development work although preliminary experimentation has been done on this device.

The original assignment of these three projects cited above was for the production of liquid oxygen for use on board a submarine for underwater propulsion. However, as the various uses of oxygen expand as evidenced by the demands from the Services, it is quite likely that use will be found for the mechanical systems in certain localities for the production of oxygen for field use. It is felt quite certain that an outgrowth of this experimental study of new cycles and new pieces of equipment will bring about a very significant reduction in the bulk and weight of portable oxygen equipment. It is intended that the study on both the mechanical and the chemical production systems shall be carried forward vigorously without any preconceived ideas as to the points where either type of system may be most readily applicable. At the present time the Air Corps has on order 45 field units for the production of 1000 cubic feet of oxygen gas per hour, the entire unit to be mounted on a truck or trailer. The units are to be constructed by three different commercial companies by the more or less conventional design, and the allowable weight of the unit mounted on the trailer as specified by the Air Corps may be 40,000 pounds. This weight is admittedly excessive, and the Air Corps realizes that such heavy and bulky units cannot be usable in a large proportion of the locations they have in mind, but they consider that it is necessary to get a few units which can be used in a limited area of operation, but they live in the hope that the total weight of an oxygen producing unit of this size can be cut down to a fraction of this figure. It is anticipated that the developments now going forward in the NDRC will be instrumental in the marked reduction in weight for both the chemical and the mechanical systems of production.

UNDERWATER PROPULSION OF SUBMARINES

Problem NL-B6 - Operation of Submarine Engines While Submerged.

(a) Engine Tests.

Progress Report: Being prepared for distribution.

(b) Absorption of exhaust gases.

Although it is anticipated that the eventual propulsion system for underwater craft may be a gas turbine, it seems quite certain that Diesel engines will be one of the modes of producing power, at least for a considerable time. Hence, a thorough study of the use of oxygen as the secondary fuel instead of air on Diesel engines is to be completed before any studies are directed toward the gas turbine.

Nearly all of the submarines operate on two-cycle Diesel engines, and it has been found that this type of engine offers much greater difficulty with the use of pure oxygen as a secondary fuel than is the case for the four-cycle Diesel engine or the gasoline engine. In the operation of these engines, it is necessary to recycle the exhaust gases in order to keep the total oxygen content of the mixture down approximately to that which is found in normal air in order to prevent excessive temperatures in the engine. In such a case, when oxygen is used as the secondary fuel, the working fluid becomes largely a mixture of carbon dioxide and water instead of carbon dioxide and nitrogen. For thermodynamic reasons, this lowers the operating efficiency of the engine, and the recycling of the gases also causes difficulty because, in the form of the loss of part of the oxygen supply, of the necessity of scavenging the cylinder between each power stroke. However, tests (The Standard Oil Development Co.) show that by proper adjustment of intake and exhaust pressures, it is possible to cut down the oxygen loss to about 6% of that supplied, though at a loss of a certain amount of power and efficiency of the engine. Systems for the injection of oxygen into the engine are to be investigated.

The second obvious problem in underwater propulsion is that of the absorption or disposal of exhaust gases in order to eliminate any trace of bubbles which might easily be detected while a submarine is submerged. The work on this problem (M.I.T.) is progressing along the same lines as previously reported, namely the studies of fundamentals in terms of the maximum bubble size which can be absorbed in the minimum depth of water which will occur in underwater propulsion; and the development of tactical dispersers for use in connection with engine exhaust. In general it is found that for a submersion of 30 feet, the maximum bubble size may range between .6 and 1 millimeter depending upon the content of the insoluble gases such as nitrogen and oxygen. The results indicate that a content of nitrogen plus oxygen of as much as 15% in the exhaust gases can be readily handled in the disperser system as now developed. Dispersers are essentially modified types of aspirators where water is pumped into the

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disperser, and gives violent shearing action across the gas stream, thus dividing the gas into very fine bubbles. Extrapolations and small scale tests indicate that a maximum of about 4% of the power of the engines might be required for operation of such an exhaust disposal system.

Noise detection tests, which have been performed on small scale, indicate that the noise attendant to the gas disperser is negligible in comparison with the other noises which would accompany the boat operation.

Another method of exhaust disposal which suggests itself is that of liquefying or freezing the exhaust gases by means of the liquid oxygen which would be on board. This, however, offers a number of difficulties, and it may be that the final system which will be most successful will be a combination of both of these methods. All phases of the problem will be thoroughly investigated before the experimental program is completed.

CENTRAL ENGINEERING WORK

Problem AC-12
NL-B60
NL-B42 - Engineering Development Work on the Production of Oxygen.

The M. W. Kellogg Co. is operating as a central engineering organization for the integrated oxygen program, and is actively engaged in the preliminary engineering design involved in the various projects. The work at this time is progressing along three lines: first, an intensive study of the engineering of the regenerative chemical system, particularly for the production of units to be mounted on trucks or to be portable by plane for the production of 500 to 1000 cu. ft. of oxygen per hour. There are various possible methods of operation. One is to pack the regenerative chemical into tubes; another is to pack the regenerative chemical around tubes which have metallic fins for rapid heat transfer; a third is to pack the material in large beds using outside cooling or heating for the absorption or desorption; fourth, to use the "floating p catalyst" type of technique for contacting a gas stream with finely divided solid in suspension in the gas stream. All of these methods of attack have been previously considered by the other investigators in this field, but it is now being brought down to a much more detailed consideration especially from the engineering point of view. The optimum conditions of this design will soon be available, and it is anticipated that the construction of the first test unit for the completely self-contained systems mounted on trucks will very soon be available.

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Another line involved in the engineering work is the consideration of equipment. The most important phase along this line is that of compressors and expanders. Work thus far is being carried through a very thorough investigation of all types of equipment both rotary and piston which are now available in the compressor and expander field which may be immediately adapted to both the mechanical and the chemical systems of oxygen production. Although such an investigation takes considerable time because of the large number of sources of information involved, it is anticipated that a program for the active engineering development of improved mechanical equipment will soon be going forward.

In conjunction with Professor B. F. Dodge (Yale) the theoretical analysis of the various possible mechanical cycles for the production of oxygen is being rapidly concluded. This outlines the field of possibility of operation for the various cycles under consideration.

In cooperation with the DuPont Company, studies are being carried forward on the matter of using "extended catalysts" techniques to increase the proportion of active surface of the cobalt complex for oxygen production. It it anticipated that the use of such an approach to increasing the rate of activity of the compound may very well involve the introduction of some inert material, and hence to cut down the ultimate absorption capacity of the compound. However, it is possible that such a step may lead to an improvement in the rate of absorption, and consequently make it possible to operate the compound at a lower total pressure. This is especially important in the consideration of a unit which may be used on board a plane.

In order not to overlook all possibilities in the production of oxygen field, calculations have been made on the requirements for a possible system which would be used for oxygen production by straight physical selective absorption in a liquid under pressure. This is one of the older systems which has long been considered, but it has not received active consideration in light of the new developments of industrial process equipment now available. The calculations indicate that even with the best equipment and processes now available such a system could not be successful in terms of power consumption, weight and bulk of apparatus unless a solvent for oxygen could be found which would absorb about 1 cc of oxygen gas per cc of liquid under a partial pressure of 1 atmosphere, and have the selective absorption for oxygen in the presence of nitrogen as much as 4 to 1. This effectively eliminates the use of these systems for any absorbing liquids now known, but it does indicate the limit which must be met for a liquid absorbing system which might contain an oxygen absorbing chemical such as the cobalt complex now being used in the solid form. Consequently these engineering calculations tie in with one of the active phases of the

program on the study of the regenerative oxygen chemicals. Two of the investigators (University of California and Iowa State College) are doing work on the consideration of incorporating the cobalt complex in solution or in suspension in order to be able to use it in a continuous cyclic system which would be analogous to the blood stream in the human body. Calculations on the liquid absorption system indicate that if as little as 4% of the active cobalt complex could be incorporated in a non-volatile, non-inflammable solvent, either in true solution or as a permanent finely divided suspension, then the continuous liquid circulating system would stand a chance of being successful.

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MISCELLANEOUS MATERIAL

A meeting with the representatives of the various branches of the Navy indicated an ever-increasing evidence in the oxygen program of NDRC and a willingness if not an expectation for the NDRC to handle all of the problems in connection with the production of oxygen and many of them in connection with the uses. It has been requested that some additional problems should be taken on by the NDRC. One of these is the investigation of the feasibility of a system for the use of liquid oxygen on board planes instead of the oxygen in high-pressure bottles as is now the system. The Navy is investigating and is having a preliminary plant built for the production of liquid oxygen on board a ship or airplane tender which will be used as the source of supply at sea for supplying planes which take off from carriers. However, in this case, the oxygen previous to loading on the plane is to be stored in high-pressure bottles, and it would be a distinct weight advantage if the liquid oxygen could be carried on board planes. There is also the possible application of the use of liquid oxygen for jet propulsion as an aid in take-off.

The Navy now standardizes on a rebreather system on some of their planes which involves the recirculation of the oxygen which is breathed by the aviator through a rebreather of sodium peroxide which partially supplies the necessary oxygen. One of the problems here is the build up of nitrogen largely through the nitrogen exhaled by the body. The Navy has presented the problem of investigations of means of removing this nitrogen from the rebreather system. Although this problem on the surface looks very difficult indeed, if not impossible, it will be given consideration.

A recent inquiry and request for information from the British indicate that a very active problem with them is that of conditioning the air of submarines before submersion by increasing the oxygen content in some parts of the submarine to at least 28 or 30%, for the use of the crew while submerged. The British consider this a very important problem, and the indications are that this might soon become a matter of high priority for the American research program also.

It is anticipated as the integrated oxygen program develops that at least one more of the large oxygen producing companies will be brought into the general program to become an active participant. The arrangements here are still in the stage of negotiation, but by the time of the next monthly report it is anticipated that they will be actively at work.

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The oxygen program now involves 15 individual contracts scattered from coast to coast and a large number of men and a large number of lines of a activity. Many complications and difficulties are involved, and hence the full-speed ahead does not come quite as quickly as might be hoped. However, it must be remembered that large things systart slowly.

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