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CHEMICAL CORPS
ADVISORY COUNCIL

AD HOC COMMITTEE MEETING
of
ENGINEERING & PRODUCTION COMMITTEE

MUSCLE SHOALS, ALABAMA

5 - 6 April 1954

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SUMMARY

The Chemical Corps Advisory Council Ad Hoc Committee met at Muscle Shoals, Alabama, 5-6 April 1954, Dr. Allan P. Colburn, Chairman, presiding.

The Muscle Shoals Project has been given the highest priority within the Chemical Corps. The responsibility for the technical and operational control of Site A has been assigned to the Commanding General of the Research & Engineering Command. A task force has been formed, composed of representatives of the Chemical Corps, TVA, and contractors at Site A. Its mission is divided into three objectives:

(1) Sustained production of 450 tons of dichlor in one month.

(2) Sustained production of dichlor at full design capacity--900 tons of dichlor in one month.

(3) Production of a roundout design and a cost estimate thereof which would increase production at Site A to 62-65 tons of dichlor per day.

In 1950 an agreement was drawn up between the Army and TVA definitely establishing Site A at Muscle Shoals. It was specified that the Army would design, construct and conduct preliminary operations of the facilities at Site A. In addition to work on the Site, TVA has performed pilot plant work on the reduction of $POCl_3$ and on the aluminum chloride process which is now in operation at Site B.

To accomplish the necessary liaison with TVA, Chemical Corps engineers have been placed within the plant in all operations on all four working shifts to act in an advisory capacity. Ideas originated by Corps engineers are transmitted to the Corps superintendent of operations and, in turn, recommendations for changes and modifications in the plant are transmitted to the TVA engineer in charge of operations. The type of organization and the system of operation have been successful. Present personnel is considered adequate for accomplishing the three objectives.

TVA is preparing to take over the routine operation of the plant, including the responsibility for all routine laboratory work and chemical testing. Even after TVA has assumed responsibility for the operation of Site A, however, TVA and the Chemical Corps will continue to make changes in process design to improve the yield and quality of the product, improve the purity of the by-products and enhance the possibilities of their disposal. A small design engineering staff will be necessary for this purpose. It it is desired, and TVA can sell the by-products, they will do so, although the disposal of by-products is primarily a Chemical Corps problem.

Two types of funds are being used at the Site--Army Modification Funds and Air Force Production Funds. As of 1 July 1954 it is expected that $7.5 million will have been spent for training. It is thought that the funds currently available will support operations at Site A and Site B through August.

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In order for Site B to maintain operations, dichlor is now being purchased from the Shell Chemical Company which utilizes the aluminum chloride process. The original contract with the Shell Chemical Company called for the production of 200 tons of dichlor and was later increased to 900 tons.

The PCl_3 facility was started in June, 1952. The facility has been operated at 120% of design capacity using two of the eight reactors installed. The consistent rate has been 30 tons or 110% of design capacity for each reactor, sufficient to meet all of the plant requirements and 1250 tons of PCl_3 have been shipped to another Chemical Corps installation. Despite a feed control problem a good produc has resulted. To date 640 tons are in storage at Site A.

(b)(3):10 USC 130

Total requirements for PCl_3 are 189 tons per day, necessitating the operatic of seven reactors.

Operation of the DMHP production facility was started on 9 February at 50% of design capacity, increasing to 70% on the 10th and operated at 70% until March 4th with no great difficulty. During this period 74% yield was obtained. From March 26 through March 30 the plant was run at 110% of design capacity, after whic the plant was shut down because of trouble. During the last run, for the first time reasonable control of the reactants in the reactor was maintained and a produ of better than 90% purity resulted. There are approximately 690 tons of DMHP in storage at Site A.

(b)(3):10 USC 130

In Step II the pyro system, except for the feed and storage tanks, is constructed entirely of Hastelloy B and there has been very little corrosion in this facility.

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(b)(3):10 USC 130

Although TVA agreed to assume responsibility for marketable by-products it was also understood that any by-products which for any reason could not be disposed of might be turned over to the Army. At present the Chemical Corps is attempting to locate long-range sources of disposal that may be used by TVA when sustained production is achieved.

At half capacity Step I will produce approximately 50 tons per day of HCl and Step III will produce 25, making a total of 75 tons per day. The original plan for disposal of HCl was to neutralize it with caustic and use the salt in the chlorine plant at TVA, but owing to the high phosphorous content of the salt this is not feasible. Various solutions are now being considered. It is not believed that the disposal problem will prevent Site A from operating.

Methyl chloride is produced at the rate of 8-11 tons per day at half capacity, which at present is being shipped to Rocky Mountain Arsenal for use in the APC process. Since this source will terminate in July 1954, however, an attempt is being made to locate an industrial concern making methyl chloride from raw chlorine and then utilizing the methyl chloride for another purpose. A tentative outlet has been found for the present methyl chloride production.

Phosphorous oxychloride is produced at the rate of approximately 55 tons per day at half capacity. A facility to reduce $POCl_3$ to PCl_3, which could then be used in Steps I and III is located at Site A. It has not yet been operated and plans are underway to conduct a test operation of this facility. Continuous attempts are being made to sell $POCl_3$ where it will not affect the normal producers of this material or upset the market in any way. This disposal problem could prevent Site A from operating.

In order to produce the planned 450 tons of dichlor per month for shipment to Site B starting 1 July, it will be necessary to start production on 1 June. If it is found that production is possible, Step III will be operated during May, although no production goal has been established for that month.

Since there are only two to three weeks storage available for Step II material, Step II must be put into operation within one week after Step III and will run approximately in balance with Step III.

There is considerable Step I product already in storage so this step will not commence operations until the latter part of June. It will then be operated at 100% capacity until 1 September when storage will be full once more. The plan is to start production in Step III on or before 1 May, build up an inventory of Step III product and at the same time fill the storage space of Steps

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I and II so that all three units can be shut down after which all changes will be made to enable the plan to operate on a two-train basis.

Assuming that all plans go forward according to schedule by the middle of November the first two phases of Site A's mission will be accomplished. If all of the material is delivered when promised it is believed that Step III could be put into operation as early as 19 April.

Seven operating engineers from the pilot plant at the Army Chemical Center will be assigned to Site A on a temporary-duty basis and approximately 12 engineers from TVA, Vitro and Site A will be integrated into this group.

Research studies on Steps I, II and III are being conducted under contract and at Army Chemical Center. Work has continued on the building of a 450-pound-per day process laboratory for Steps I, II and III at Army Chemical Center.

The reaction system was started on 19 March and the highest priority was determination of the chlorination reaction. A general study is being made of the reaction in Step III. The process laboratory purification system for Step III is expected to be in operation on 15 April. It is anticipated that the Step II portion of the process laboratory will be in operation by 1 May.

An effort will be made to determine what effect by-passing the still in Step I will have on Steps II and III and the correlation of Standard Oil Development work and corrosion.

Under contract the Standard Oil Development has been studying Step II pyrolysis and in the light of its studies another contract for approximately $65,000 is being negotiated for further study of this step. Also under study is a modified Step II without a catalyst, utilizing undistilled DMHP, in order to determine operating conditions. A third phase of Standard Oil Development's study will be a modified Step II using methylating agent. It is anticipated that all of this work will be completed by 1 December 1954.

In addition to studies being made under contract a number of problems relating to Step III are being considered by the personnel of Army Chemical Center. The results of these studies will be directly applicable to operations at Site A.

Piloting of the HTM processes for oxidation-reduction of HTM intermediate, dichlorphosphine, to dichlor is being conducted under contract. A pilot plant is in operation, and it is believed that information for a unit plant design will be given to Engineering Agency on 1 September 1954.

The unit plant design for the salt process is being performed by Mathieson Chemical Corporation.

The pilot plant work at Army Chemical Center is continuing and should be completed by 1 September 1954.

Three instances of possible stream pollution have focused the attention of the public on both TVA and PDW. The problem of stream pollution has been consid-

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ered by practically every agency which has been involved in the design, construction, and operation of the facilities and an attempt is being made to provide proper controls. Several process changes and changes in the treatment of normal process waste are being considered. The idea of providing retention lagoons through which all waste and surface drainage from Site A would pass before entering Pond Creek and the Tennessee River has met with unanimous approval.

From February 20th to March 3rd Step I was operated at 70% capacity with the reactor operating at 15°F and the still on stream. Slightly better than 30,000 pounds of DMHP per day were produced and the yield was approximately 73-74%. By eliminating the still from operations and by effecting contemplated modifications to the reactor it is believed that the yield of DMHP can be increased to 87-90%.

The PCl_3 facility has been operated with little difficulty at 110% of capacity. Step I has been operated at 110% of design capacity on one train with little apparent difficulty. The next mission requires that Step I be divided into two trains and brought up to 30 tons GB equivalent. It is estimated that the present facilities can be changed over to a two-train operation in approximately ten weeks. Step II has operated on a two-cascade basis (one train) up to 110% with no difficulties involved. To date Step III has not been operated. All construction is expected to be finished between the 19th and 30th of April, after which operations will begin.

No plans have been made for the operation of the $POCl_3$ reduction facility for the following reason: (1) Materiel Command has stated that $POCl_3$ can be sold, and (2) major changes similar to those being made in Step III appear to be required in this unit.

To date no difficulty has been experienced with the waste disposal facility. However, the piping is being changed to allow greater flexibility and to have three systems instead of two.

The disposal of waste materials appears to present no problem. Disposal of by-products--methyl chloride, HCl, $POCl_3$--may be a source of difficulty if not promptly handled.

There are 2% unknowns in the salt produced and samples have been furnished to Huntsville to determine if the material can be utilized in the diaphragm cell. On an economical basis it will be necessary to determine whether it is more desirable to change the diaphragm more frequently in order to get cheaper salt or to continue buying salt from the mines in Louisiana at a total cost of $13.00 a ton.

After considerable discussion as to whether a pilot plant was necessary at this time, the consensus of the Chemical Corps and TVA personnel was that the pilot plant was neither desired nor needed.

High praise was given to the work performed by the ESPP personnel. Although it may be possible to decrease the number of technical people at some later date, it is thought advisable to keep Site A heavily staffed with this type of personnel until operations are on a routine basis.

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