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DATE 21 Feb 1958

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CHEMICAL CORPS

**CONSOLIDATED R&D ANNUAL
PROJECT REPORT**

(EXCLUDING GM) (REPORTS CONTROL SYMBOL CSCRD-16)

Edition A—AW, BW, CW
Vol 1
31 December 1957



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| R & D PROJECT CARD | | TYPE OF REPORT Rewritten | | REPORT CONTROL SYMBOL CSCRD-16 | |
| 1. PROJECT TITLE CW Agent Process Development (U | | | 2. SECURITY OF PROJECT [REDACTED] | 3. PROJECT NO. 4-92-03-013 | |
| 4. BASIC FIELD OR SUBJECT Processing & Manufacturing Methods & Techniques & Equipment | | | 7. SUB FIELD OR SUBJECT SUB GROUP Chemical | 5. REPORT DATE 31 Dec 57 | |
| 6. COGNIZANT AGENCY CmlC | | | 8. CONTRACTOR AND/OR LABORATORY Proc Dev Div, Dir/Dev, USA CW Labs | | CONTRACT/W. O. NO. DA-18-108-CML-6109 DA-18-108-CML-5725 and 5726 |
| 9. DIRECTING AGENCY CmlC R&D Cmd | | | 10. REQUESTING AGENCY CmlC | | |
| 11. PARTICIPATION AND/OR COORDINATION Army (AR) | | | 13. RELATED PROJECTS 4-04-15-032 | | 17. EST. COMPLETION DATES RES. DEV. Cont. TEST OP. EVAL. |
| | | | 14. DATE APPROVED 9 Dec 54, CCTC Item 2962 | | 18. FY. FISCAL ESTIMATES |
| | | | 15. PRIORITY 1-B | | 58 936M 59 360M 60 360M |
| | | | 16. MAJOR CATEGORY 5..2.. | | Continuing rate p/a 980M |
| 19. REPLACED PROJECT CARD AND PROJECT STATUS Subproject card 4-92-03-013-02, dated 31 Dec 56; revised CCTC Item 3388, 12 Dec 57. | | | | | |
| 20. REQUIREMENT AND/OR JUSTIFICATION (1) There is a requirement for the development of CW agents. Necessary steps between research and the ultimate production from CmlC facilities are the development of chemical process data; design, construction and operation of pilot plants based on such process data; determination of process and equipment design data through pilot plant operation and the pilot production of agents for field evaluation of agent and dissemination techniques. CDOG ref. par. 1212a. | | | | | |
| 21. BRIEF OF PROJECT AND OBJECTIVE | | | | | |
| a. <u>Brief.</u> (Applied Research) The object of this project is: Operation of a chemical-process laboratory for the development of process data and process-control techniques; design, construction and operation of pilot plants to develop process and equipment design data and experimental production of agents for field development and evaluation work. | | | | | |
| b. <u>Approach.</u> The approach taken in process and product development is: (1) Determine in the process laboratory the physical and chemical | | | | | |
| 22. OASD (R & D) | | SN. | CN. | C. | X. I. C. |
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characteristics of reactants and reactions leading to an end process or product.

- (2) Select a sound technical and economical process for development.
- (3) Develop the controls necessary for sound technical and economic processing.
- (4) Develop data for the design of piloting facilities.
- (5) Design and build piloting facilities for processing facilities.
- (6) Operate pilot plants to develop and furnish complete processing information, engineering data and process products for use in further development.

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c. ~~CONFIDENTIAL~~ Subprojects and/or Tasks.

- (1) Process Development Studies
- (2) V-Agent Pilot-Plant Studies

(C)

d. (U) Fiscal Estimates:

| Period | Total Estimate | Army R&D Funds | Proc & Prod Funds | Other Ident. Army Funds | Other Agency Funds |
|---------|----------------|----------------|-------------------|-------------------------|--------------------|
| FY55-57 | 3406M | 2990M | 415M | 1M(ORD) | =0M |
| FY58 | 1317M | 936M | 360M | 10M(ORD) 11(O&M) | =0M |
| T | 4723M | 3926M | 775M | 22M | =0M |

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Other Information. None.

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| PROJECT NO. 12-03-013 |
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Background History and Progress.

(1) Background History.

This project previously included subprojects 4-92-03-013-02, CW Agent Process Development; 4-92-03-013-03, CW Agent Filling Equipment Development; and 4-09-03-013-04 Smokes & Incendiaries. CCTC Item 3388, dated 12 Dec 57, revised this project by continuing subproject 4-09-03-013-02 as project 4-09-03-013. Work under 4-09-03-013-03 was continued as subproject 4-04-15-032-06 and subproject 4-09-03-013 was suspended.

(a) G Agents. In 1947 GB was selected as the G agent to be produced for general field use, and the DMHP process was also selected as the most satisfactory for the production of GB. Based upon pilot-plant development, production facilities were designed, constructed and operated.

Since 1951 several additional processes for the production of GB have been investigated. The process that offers the most advantages is a high temperature method (HTM process) of combining methane with phosphorous trichloride to form dichlormethylphosphine.

(b) V Agents. (b)(3):10 USC 130,(b)(2) HIGH

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(b)(3):10 USC 130,(b)(2) HIGH

Process development work was begun both in these Laboratories and by Olin Mathieson Corporation under contract DA-18-108-CML-6109. Process data developed by these sources were applied to pilot-plant design as fast as available.

A source of the organic phosphorus intermediate, dichloromethylphosphine, existed in the pilot plant built and operated by Westvaco Chlor-Alkali Division, Food Machinery and Chemical Corporation under contracts DA-18-108-CML-5725 and DA-18-108-CML-5726. This source proved to be limited to 22,500 lb. of the crude intermediate, which was purchased, and purification equipment was built at these Laboratories to further refine the material to a grade usable in the transester process.

(2) Progress.

(a) Process Development Studies of V Agent.

(1) Dichlormethylphosphine. Process and pilot-plant development was done by Westvaco Chlor-Alkali Division of Food Machinery Corporation under contracts DA-18-108-CML-5725 and DA-18-108-CML-5726 for the HTM process. Pilot-plant development work has been completed; unit-plant design phase is incomplete.

(2) Step I. Investigation of the process variables was continued in the process laboratory and under contract DA-18-108-CML-6109 by Olin Mathieson Corporation.

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PROJECT TITLE
GW Agent Process Development (U)

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Emphasis and most of the work has been directed toward the development of continuous process equipment and process controls.

The related problems of process control and analytical methods in this and in succeeding reaction steps are being worked on by processing-laboratory personnel. The many problems in these areas cannot be solved by effort of these Laboratories alone in the time available. Assistance will be obtained from industry under a contract, as described below.

(3) Step II. Investigation of the reactions in this step of the process have been continued in the process laboratory on both bench scale and prepilot-plant scale. The work has generally been done by a batch process and has been directed specifically toward a study of separation methods for the reaction components and purification methods for the transester end product. Here as in Step I, progress will be accelerated by solution of the problems of process control and analytical methods.

(4) Step III. In this step of the process, sulfur is added to transester followed by isomerization to produce VX. A small-scale, 8 lb. per batch, process has been developed to produce agent for munitions and dissemination development. Approximately 1000 lb. of agent has been so produced for these purposes. The process work has served as a basis for the design of the pilot-plant reactor system.

(5) Contract Proposal. Additional research and development is urgently needed in the determination of physical and chemical characteristics of reactants and reactions, in analytical methods for both process-stream constituents and intermediates. These are the first tasks proposed in the contract, and will be followed by a task to develop

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process-control instrumentation design. Several industrial firms, including chemical manufacturers and engineering organizations, have shown an interest in this proposed work. The scope for such a contract has been written and submitted for approval through U. S. Army Chemical Center Procurement Agency.

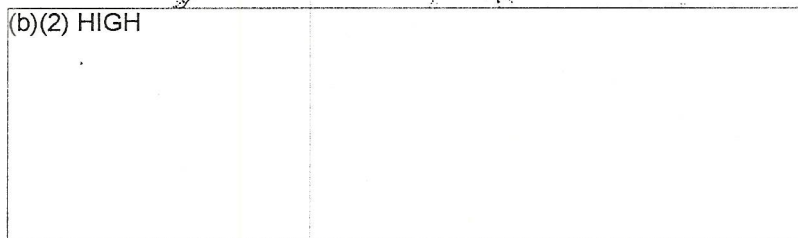
(b) V-Agent Pilot-Plant Studies.

(1) Design. Design has been completed on an HTM process unit to produce and purify by distillation approximately 1000 lb. of dichlormethylphosphine every 24 hr. This unit will produce material of approximately 95% purity as feed stock for the V-agent pilot plant. Design of the agent pilot plant is complete. Changes in design are being made as additional process information becomes available from the process laboratory, or from operation of the pilot plant itself.

(2) Construction. Construction of the HTM unit is underway. Operation of this unit is scheduled for 4th Q FY 58. Construction of Step I and Step II pilot facilities have been completed; completion of Step III is scheduled for 3rd Q FY 58.

(3) Operation.

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(b) Step I. Operation of this step in the pilot plant has been started; as designed it is continuous. Deposition

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of the solid ammonium chloride on reactor heat-transfer surfaces has so far limited the continuity of operation to 48 hr., or less. Purification equipment operated satisfactorily. The material produced was shown, by assay at the process laboratory, to be satisfactory. Other types of continuous reactors are being tried in the search for an answer to the ammonium chloride problem.

(c) Step II. Operation of this piloting equipment has been started. Some minor changes in design and construction have been made to correct deficiencies made apparent by the limited operation.

(d) Step III. Operation of this final step in the process is scheduled for the 3rd Q FY 58.

f. Future Plans.

- (1) Expedite the development, by contract and CWL effort, of basic data in the areas of process data, analytical methods, and process control.
- (2) Complete the construction of the HTM unit and Step III pilot facilities. Completion of the HTM unit will permit operation of the agent pilot plant unrestricted by inadequate supply of dichlormethylphosphine.
- (3) Continue operation of the agent pilot plant and complete process development.

g. References.

- (1) CWL Technical Memorandum 31-6, Agent 1701 - Comparison of the Transester Process and a Water-Wash Process for Production - Preliminary Report, 21 January 1957.

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21. g. Continued.
- (2) CWL Technical Memorandum 31-15, Preliminary Report on V-Agent Pilot Plant Section 5: Step O Process Detail, 14 February 1957.
 - (3) CWL Technical Memorandum 31-16, Process Laboratory Development of Transester Process, Step 1, February 1957.
 - (4) CWL Technical Memorandum 31-23, Preliminary Report on V-Agent Pilot Plant Part 2: Step O Data Book, 1 March 1957.
 - (5) CWL Technical Memorandum 31-24, Preliminary Report on V-Agent Pilot Plant Part 2: Step I Data Book, 1 April 1957.
 - (6) CWL Technical Memorandum 31-25, Preliminary Report on V-Agent Pilot Plant Section 5: Step I Process Details, 8 April 1957.
 - (7) CWL Technical Memorandum 31-26, Reaction of ROH with Methylchlorphosphine Without an Acid Acceptor, 12 April 1957.
 - (8) CWL Technical Memorandum 31-27, Preliminary Report on V-Agent Pilot Plant Section V: Step II Process Details, 1 May 1957.
 - (9) CWL Technical Memorandum 31-30, Preliminary Report on V-Agent Pilot Plant Part 3: Process and Engineering Program Step I, 17 June 1957.
 - (10) CWL Technical Memorandum 31-35, Pre-pilot Evaluation of Size O Turba-Film Evaporator, August 1957.
 - (11) CWL Technical Memorandum 31-41, Preliminary Report on V-Agent Pilot Plant Part I, Section 5: Step 3 Process Details; and Part 3, Process Engineering Program, 5 September 1957.
 - (12) CWL Technical Memorandum 31-42, Preliminary Report on V-Agent Pilot Plant Part II; Step 2 Data Book, September 1957.

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1. PROJECT TITLE
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- (13) CWL Technical Memorandum 31-43, Preliminary Report on V-Agent Pilot Plant. Part II: Step 3 Data Book, 18 September 1957.
- (14) CWL Technical Memorandum 31-44, Report on Modified HTM Plant. Section 5: HTM Process Details, September 1957.
- (15) CWL Technical Memorandum 31-45, Preliminary Report on V-Agent Pilot Plant. Part III: Process and Engineering Program Step II, 8 October 1957.
- (16) CWL Technical Memorandum 31-51, Pilot Plant Process and Engineering Reports on Agent EA 1701 by the Transester Process, 31 October 1957.
- (17) Olin Mathieson Chemical Corp., Final Report on DA-18-108-CML-6109, Project Gibraltar, 1 September 1957.
- (18) Food Machinery and Chemical Corp., Final Report on DA-18-108-CML-5725, Development of HTM Pyro Process, June 1957.

h. Modernization Code.

Not applicable.

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