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SPECIAL REPORT

SIGNIFICANT ACCOMPLISHMENTS
FISCAL YEAR 1953

Excerpt

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SIGNIFICANT ACCOMPLISHMENTS
FISCAL YEAR 1953

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I. INTRODUCTION.

This is the second consecutive annual report which summarizes the operations of the CmlC Chemical and Radiological Laboratories (C&RL) for an entire fiscal year. During the year a total of approximately $14,600,000 from all sources was expended, which is approximately $2,000,000 less than last year. At the end of FY 1953 combined military and civilian personnel strength stood at 1,384, a decrease of 46 from the beginning of the year.

The operations of C&RL were characterized by accelerated progress in the development of end items, as well as by the exploitation of new concepts in the fields of chemical and radiological warfare.

At the year's end, 38 items had passed the final engineering test and had been turned over to the CmlC Engineering Agency. This compares with 13 items completed by C&RL during the previous year. In addition to the 38 completed end items mentioned above, 16 items were developed to the final engineering stage. While C&RL has no responsibility for carrying end items beyond the final engineering test stage of development, it should be noted that 17 items which were developed originally by C&RL were standardized during the year.

During the year, 104 research and development contracts were awarded to outside agencies, representing a total value of more than $5,000,000. At the end of the year there were 149 contracts in effect, valued at approximately $15,600,000. These latter figures include the carry-over from FY 1951 and 1952. Anticipated budgetary reductions in FY 1954 are expected to reduce drastically the scope of the contract program.

II. SUMMARY OF PROGRESS.

A. Agents.

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1. Agent GB.

Work on Agent GB (isopropyl methylphosphonofluoridate) was continued under highest priority. Since production facilities are based on the DMHP (dimethyl hydrogen phosphite) Process, first importance was given to

process-laboratory and pilot-plant development in support of the production facilities. In this connection, process data needed for further piloting of Steps I, II, and III were obtained. Pilot-plant work on Step I is proceeding satisfactorily, a detailed study of the pyrolysis reaction (Step II) is under way, and a process for continuous chlorination in Step III has been successfully piloted. In view of difficulties which have developed in preparations for large-scale production of DMHP "Dichloro" (methylphosphonic dichloride), a laboratory study of the chlorination of DIMP (diisopropyl methylphosphonate) to "Dichloro" was undertaken in order to provide the basis for an interim process. Preliminary results, using thionyl chloride as chlorinating agent, indicate that a quantitative conversion is readily obtainable; feasibility of phosphorus trichloride and chlorine as chlorinating agent was also demonstrated.

The engineering evaluations of the various processes under study in FY 1952 pointed to the Salt Process as the most practical for use when additional GB manufacturing facilities will be required. During FY 1953, the five steps which comprise this method were carried out in the Process Laboratory. Research on improvements in the method are under way. Piloting the first four steps is being carried out under contract, and work on the first three is nearing completion. Work on Step IV, which is apparently the most difficult one, is in progress. Sufficient work on Step V has been completed here, using chloro-GB prepared in C&RL for this purpose by a batch process to demonstrate its operability based on this Step IV product. This work is continuing. Completion of Step V will depend on availability of Step IV product of usable quality from a typical pilot plant. This material has not yet been produced. Four thousand pounds of GB was produced in the Step V pilot plant.

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Investigation of several other alternate processes was continued. Further laboratory work on the TIPP (Triisopropylphosphite) Process resulted in a variation called the Ammonia Process, which promised initially to be competitive with the Salt Process. However, more detailed study and considerations of time in relation to the comparative progress already made on the Salt and Ammonia Processes led to the decision not to pilot the latter. The Kellogg Process has been improved to the point where it appears ready for larger-scale laboratory work and piloting if funds become available for this purpose. A process based on oxidation of phosphorus in the presence of methanol is believed on the basis of a laboratory study not to have sufficient promise to merit further study at present.

Thus, as seen above, the picture in regard to processes to be

compared is changed as of today, because of loss of interest in the TIPP (or Ammonia) Process, and the great promise of the HTM Process. However, a recent re-evaluation confirmed the 1952 choice of the Salt Process for an immediate construction program. Insufficient data were available to permit inclusion of the HTM Process in this evaluation. It appears probable, if the promise of the laboratory work on this process is confirmed in the pilot plant now under construction, that it will be recommended over all the others.

Some progress has been made on a comprehensive program on the chemistry of GB. This work is aimed at eventual improvement in existing methods of detection and decontamination, and is also in support of CmlC Medical Laboratories research on the mechanism of action of G Agents.

Further studies of radioactive GB were carried out, and samples of this and other radioactive G agents were furnished to CmlC Medical Laboratories for research on biological reaction mechanisms.

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2. Persistent Agents.

The most important factors to be considered in the choice of supplementary agents with greater persistence than GB are the actual use to which these agents are to be put, and the matching as closely as possible of pertinent physical and biological properties to this use. We have no accurate guide as to proposed detailed use of these agents. In this situation, the Advisory Committee on New Agents is considering, among others, the following important properties in its study of persistent "quick-acting" agents - volatility, percutaneous toxicity, speed of action, stability (especially when dispersed on soil), and ease of manufacture.

Among the higher-molecular-weight homologs of GB, the following toxics have sufficiently low volatility to be considered as candidate persistent agents, and at the same time, sufficient vapor pressure to give a possible vapor hazard:

GD (pinacolyl methylphosphonofluoridate)

GF (cyclohexyl methylphosphonofluoridate)

Compound 1211 (formerly GH, methylisobutylcarbinyl methylphosphonofluoridate)

These substances are comparatively rapid in their action.

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Compared with the group of GB analogs, they are quite slow in their action.

Further study of the above compounds is under way. All show high percutaneous toxicity, with the possible exception of Compound 1211. While the manufacture of any of the above candidate agents is considered feasible, preliminary evaluation indicates some variation in the ease of manufacture.

In addition to the above substances, laboratory study of which is fairly well advanced, new candidate agents for persistent use are constantly appearing in the lists of compounds synthesized in C&RL (EA Series) and those from outside sources (CS Series). These for the most part are phosphates and phosphonates which can be represented by some variation of the GB or paraoxon structure. They are at present under preliminary study.

Investigation of possible thickeners for GB did not reveal a more satisfactory material than "Lucite." However, because of length of mixing time required in the use of "Lucite," search is continuing for other thickening agents. Study of the physical properties of thickened GB was pursued, and investigation of its stability showed it to be satisfactory at normal temperatures. However, it was found that tributylamine-stabilized GB and plant-grade GB when thickened with "Lucite" are considerably less stable than pure GB, similarly thickened.

The results of the Advisory Committee on New Agents study on candidate persistent agents are expected in the near future.

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3. Agent Mixtures.

If toxic agents should be required for immediate use, it would be necessary to rely on stocks of materials currently on hand or available on very short notice. For this purpose, we are thus limited to Agents H (mustard), CG (phosgene), AC (hydrogen cyanide), and CK (cyanogen chloride). In order to increase the range of usefulness of these agents, particularly to extremely cold weather conditions, study of the eutectic mixtures, H/L and AC/CK, was undertaken. Samples of these mixtures were prepared for study of chemical and physical properties in Chemical and Radiological Laboratories, and for investigation of physiological effects by Chemical Corps Medical Laboratories.

A longer-range problem on agent mixtures is also under way.

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This involves study of the following mixtures:

GB/HD (isopropyl methylphosphonofluoridate-distilled mustard)

GB/CX is phosgene oxime)

The GB/CX mixture is of interest because of preliminary biological data which indicate that it is more toxic percutaneously to animals than GB alone and because of its very low freezing point.

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4. New Agents Search.

The work of the Advisory Committee on New Agents, consisting of personnel from Research and Engineering Command, Chemical and Radiological, Medical, and Biological Laboratories, as in the past, was focussed on toxics of chemical warfare interest and the mechanism of toxic action. Toxicity screening procedures have become well established and are functioning now on a routine basis. During FY 1953 the Committee requested 157 samples selected from lists totaling about 400 compounds, compiled from information supplied by outside sources. About two thirds of these substances were requested for the purposes of the screening program; the rest were intended for chemical and spectrographic studies in Chemical and Radiological Laboratories. Most of the compounds are organic phosphorus and fluorine derivatives. Other classes represented are mono- and bis-quaternary ammonium compounds, amines and nitrosoamines, carbamates, esters, acrylic derivatives, and phenylalkamines.

Chemical and Radiological Laboratories and its contractors synthesized 73 compounds for screening as candidate agents during this fiscal year. Analogs of GB and DFP (diisopropyl fluorophosphate), acetylcholine analogs, bis-quaternary ammonium compounds, aliphatic sulfonyl fluorides, fluorinated hydrocarbons, and boron compounds comprised this list.

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