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NATIONAL RESEARCH COUNCIL OF CANADA

Division of Chemistry

WAR HISTORY
OF
DIVISION OF CHEMISTRY

OTTAWA

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PREFACE

The scope and extent of the war-time activities of the Division of Chemistry were such that an all inclusive report would have been beyond the practical limits of this volume. Details of extensive and valuable work of a more or less routine nature have, therefore, been omitted from this account, except for passing mention. Nor is it possible here to discuss the considerable contributions which members of the staff have made in administering the work of various Associate Committees of the National Research Council. Discussion of work done for the Associate Committees has been confined for the most part to actual experimental investigations carried out in the Divisional laboratories. The tremendously valuable work of these various Associate Committees is to be treated extensively in another volume.

It is perhaps well to warn the reader that the importance or value of any project is not to be inferred from the space devoted to it in this History. Work which has been adequately described by publication in the literature has been treated very briefly here; whereas work which has not been published is treated much more extensively.

The primary purpose of this volume is to give the technical reader an informative summary of the contribution made by the Division of Chemistry during the period of the Second World War. It is also the purpose of this volume to guide those interested to more detailed sources of information. To this end a rather extensive bibliography of publications, internal reports, and patents has been included. It has been the policy to include references which describe work done during the period of the war, regardless of the actual date of publication or issue. It might here be mentioned, parenthetically, that work done after VJ-day has sometimes been included when it served to complete an investigation previously started, or to show the significance and value of previous work.

The publications listed in the bibliography are believed to be a complete record of all publications, descriptive of war-time work, published prior to June 15, 1946, except for a few minor articles of no particular scientific interest which were published in some of the trade journals. The patent references are believed to include all patent applications covering war-time inventions in the Division of Chemistry filed in Canada and the United States prior to June 15, 1946. The patent number and date of issue are given, when these are known, otherwise the application number and date of

filing are given. It should be noted that the Council has no direct knowledge of issue of a patent when the patent rights have been assigned before the patent issues. Therefore absence of a patent number is not to be considered as evidence that the patent has not yet issued, for, indeed, many of these applications actually have issued. This bibliography is not intended as a record of the Research Council's patent position, but rather as a record of the work done in the laboratories; as the Canadian and United States applications give adequate disclosures in this respect, United Kingdom and foreign patent applications and patents have not been listed.

More than ten thousand reports were issued by the Division of Chemistry during the war years. Obviously, it has been possible to include only a few of them in this bibliography. An effort has been made to include the more important reports covering investigations not recorded elsewhere and, in general, to include enough reports to serve as examples of the work of the various laboratories. Sometimes reports on typical investigations have been included in the bibliography even though that work is not specifically referred to in the text; and conversely work is sometimes referred to in the text for which no report references are given. Anything like a completely consistent record with respect to reports would be a task of impracticable magnitude, and such completeness has not been attempted. In arranging the bibliographies an effort has been made, in so far as possible, to list the references in the order in which their subject matter is discussed in the preceding text. A few miscellaneous publications which could not conveniently be classified have been grouped together as an appendix.

A final work regarding the arrangement of this History may be in order. No one method of classifying the material was entirely without faults, but it was found most expedient to classify the work of the various laboratories according to the nature of the work. Thus there are chapters on Chemical Warfare (both offensive and defensive), and on Protection of Personnel and Equipment (including fields such as textiles, corrosion, and protective coatings). These two chapters deal entirely with specific applications of chemistry to the problems of war. Other work was classified either as Inorganic or as Organic Chemistry, depending on the composition of the principal substance involved. While these two chapters also include direct applications of chemistry to war problems, they tend somewhat more towards the

study of basic materials and their production, or to chemical studies of a fundamental nature. In some cases this system of classification has largely coincided with the work of distinctive laboratories; in other cases many laboratories have contributed to one general field of endeavour. In any event, the bibliographies will give credit where credit is due; that is, to those who did the work.

A. HISTORICAL INTRODUCTION

The beginning of the Second World War in September, 1939, found the organization of chemical research in Canada already under discussion. In July of that year, the National Research Council had convened a conference of Heads of Industrial Chemical Research Organizations to discuss ways in which cooperation between industrial and governmental organizations conducting industrial chemical research could be facilitated. The conference was largely exploratory, and a steering committee had been appointed to consider further activities of the group. A resolution, dated September 8th, drafted by this committee and approved by the members of the July Conference, was tabled at the meeting of the National Research Council on September 15th. It called for the setting up of a War Research Committee with the President of the National Research Council as its chairman, "to receive and suggest research problems, to coordinate the available research facilities, to assess the relative importance of the problems to be dealt with, and to allocate these problems to the various laboratories". In view of the fact that these are specific functions of the National Research Council and that the Council already had facilities for effectively discharging these functions, it was decided not to appoint a committee with such executive powers, but to develop the services of the Council in every way that seemed appropriate, in receiving proposals for research, and in ensuring that such work was undertaken in those laboratories where it could be most effectively prosecuted; and, to this end, it was decided that the Assisted Researches Committee would be enlarged and reorganized.

On October 17th, General McNaughton, President of the National Research Council, assumed his duties as Officer Commanding the First Canadian Division, and on the following day Dean C.J. Mackenzie came to Ottawa as Acting President of the Research Council. Two weeks later, on October 31st, Dean Mackenzie met with a small group of key men in the field of industrial research, all of whom had been present at the July Conference. The manner in which the National Research Council could act as the liaison and coordinating body for war research was discussed, with particular reference to the chemical field. It was agreed that a chemical advisory committee should be set up to advise the Assisted Researches Committee of the National Research Council on problems relating to the war effort in the chemical field, and to cooperate through the Council with research organizations in Great Britain with a view to the correlation of

chemical research work on war problems. At this point in the conference, it was announced that Dr. Maass had been appointed to the National Research Council, whereupon it was decided that he should act as Chairman of this advisory committee, At the next meeting of the Council, on December 14, 1939, such a committee was formally established under the name of the Advisory Committee of Industrial Chemists, with Dr. Maass as its chairman, and its personnel consisting of those present at the October Conference, namely: Dr. A.F.G. Cadenhead, J.R. Donald, Dr. R.T. Elworthy, Dr. I.R. McHaffie, Dr. H.B. Speakman, Dr. E.W.R. Steacie, Dr. R.K. Stratford, S.J. Cook (Secretary) and Dr. C.A. MacConkey (Asst. Secretary).

At this same Council meeting, five Subcommittees on War Researches of the Standing Committee on Assisted Researches were set up on (i) Chemical Problems, (ii) Biological Problems, (iii) Physics Problems, (iv) Engineering Problems, and (v) Medical Problems. The Subcommittee on Chemical Problems consisted of Dr. O. Maass (chairman), Dr. E.W.R. Steacie and Dr. R.K. Stratford.

During the late winter and spring of 1940, Dr. Maass, as chairman of the Advisory Committee of Industrial Chemists, called together advisory subcommittees in the fields of explosives and petroleum technology. These High Explosives and Petroleum Subcommittees were very active, and ably carried out the advisory functions assigned to them. Later, it became desirable that these committees should administer the very extensive research activities which had been initiated; their membership wqs then broadened, and they were formally constituted as Associate Committees of the National Research Council. Under Dr. Maass' direction, and with the advice of these various committees, research work in the fields of chemical warfare, explosives, and petroleum were initiated, during 1940, in various laboratories, particularly at McGill University of Toronto. Thus an extra-mural research program of considerable extent was got under way, funds being supplied initially by the Assisted Researches Committee of the National Research Council. These Committees inevitably were associated in many ways with the activities of the Division of Chemistry; a detailed discussion of their work is reserved, however, for another volume.

Until the time of Dunkirk, the funds available for special war research were somewhat limited, as the full extent of the contribution which Canada could make to the war effort was not visualized until after Britain had lost her chief ally and stood alone, facing a powerful enemy across the narrow Channel. It was at about this time that a small

group of Canadian business men offered to donate a sum totalling about one million dollars in aid of Canada's war effort. This offer was first received by the Acting Deputy Minister for Air, who called a meeting of ranking officials of various departments of Government concerned, to discuss with representatives of the donors the question as to how these funds could most effectively be applied. At this meeting various proposals were presented, but when the Acting President of the Research Council outlined what the Council was doing both during peace and during war, it was unanimously agreed that these monies could most effectively be expended on research projects proposed by the National Research Council. A War Technical and Scientific Development Committee was therefore set up by P.C. 4260, August 27, 1940, consisting of representatives of the National Research Council, of the various government departments directly concerned in the prosecution of the war, and of the donors. This committee was authorized to receive funds from private donors and to transfer to the National Research Council funds for the carrying out of projects proposed by the National Research Council and approved by the Committee. The National Research Council representatives on this Committee were Dean C.J. Mackenzie, Sir Frederick Banting and Dr. Otto Maass. Later, in July 1941, after the tragic death of Sir Frederick Banting, the fund was renamed the Sir Frederick Banting Fund, and the committee, the Sir Frederick Banting Fund Committee. Dr. J.B. Collip was named to replace Sir Frederick on the Committee. By February of 1943 the sum available to the National Research Council through the fund had reached $1,300,000. This money made possible the development of the early work in such fields as chemical warfare, explosives, ballistics, radar, optics, aviation medicine, and aeronautics, up to the point of proven success where money from regular sources was readily available to carry the projects to completion.

One other event of that summer was of significance in the field of chemistry. The rapid expansion of the activities of the Research Council made necessary the appointment of a Special Assistant to the Acting President. Dr. Maass, who was already a key figure in the organizing of chemical war research, was appointed to assume the responsibilities of this position - a position which he filled with distinction throughout the war.

These events were, then, the background against which we must consider the history of the wartime activities of the Division of Chemistry, to which we will now turn more specifically.

When Dr. E.W.R. Steacie assumed the post Director of the Division of Chemistry in July 1939, projects were already under way - thanks to the foresight of the President, General McNaughton - which were to be of significance in Canada's war effort; most notable of these were the investigation of methods of producing metallic magnesium, and the proofing of active charcoal and containers for respirators. With the incidence of war in September, the program of the Division was rapidly converted to a wartime basis; several projects were terminated as rapidly as practicable and new ones were initiated

During the course of the war there was a tremendous increase in the activities of the Division of Chemistry, and the staff of the Division approximately tripled in size.

Throughout the war, administrative duties in connection with the Associate Committees on Textiles, Explosives, Petroleum, and Rubber engaged almost the full attention of two senior members of the staff.

A number of noteworthy changes in the Division took place during the years of the war:-

The respirator container proofing laboratory rapidly expanded, as interest in other aspects of chemical warfare developed, until it was taken over by the Department of National Defence early in 1941, becoming the Research Establishment (C.W.) of the Directorate of Technical Research. Early in 1943, laboratory and pilot plant work on refractories was transferred to the new laboratories of Canadian Refractories Ltd., at Kilmar, Que. A year later development work on metallic magnesium was taken over directly by Dominion Magnesium Ltd. Each of these were projects which, launched under the aegis of the Division of Chemistry, had developed into self-reliant organizations, able to carry on an independent existence.

Early in 1941 an Explosive Testing Laboratory was established as joint activity of the Department of Mines and Resources and the National Research Council. The laboratory was administered by the Division of Chemistry. In addition to functioning in its statutory capacity for the inspection of explosives under the supervision of the Department of Mines and Resources, the laboratory has conducted extensive researches under the direction of the Associate Committee on Explosive Testing and Research, which later, towards the end of 1942, became part of the newly formed Associate Committee on Explosives. These investigations will be discussed in the volume

of this History dealing with the work of the Associate Committees.

On January 1st, 1943, the staff and laboratory facilities of the Customs and Excise Laboratory were transferred from the Department of National Revenue to the Division of Chemistry of the National Research Council. Under this new arrangement the laboratory is operated jointly by the Council and the Department of National Revenue. It has continued to function in its statutory capacity for the examination of commercial products for customs and excise purposes.

The need for a Chemical Engineering Section within the Division had been becoming increasingly apparent as activities of a pilot plant nature increased in importance. The removal of the refractories and magnesium pilot plants provided an opportunity for the consolidation of such engineering activities, and, early in 1944, a Chemical Engineering Section was created, with the group working on sulphur recovery as its nucleus. This new Section was almost immediately asked to undertake heat transfer pilot plant studies in connection with the Chalk River atomic energy pile. When, at the end of that year, water treatment studies were commenced at Chalk River, the Chemical Engineering Section provided most of the staff and facilities.

These projects so completely engaged the staff of the new section that, when the Division of Chemistry was requested a few months later to undertake pilot plant studies of the chemical separation processes for the Chalk River plant, it was necessary to create what was, in effect, a new section, but which was more generally known as the "Pilot Plant Group". The "Design Group" which converted these pilot plant studies into full scale plant designs, was a similar temporary section. Later, and after the close of hostilities, when the plans for the chemical separation plant were largely complete, there was a considerable contraction in the size of the staff engaged in the work; the remnants were consolidated under the Chemical Engineering Section and formed the Chemical Engineering Laboratory at Chalk River.

In the meantime, in May 1944, Dr. E.W.R. Steacie had been appointed Deputy Director of the Council's Montreal Laboratories, which were the centre of the Atomic Energy Project, and responsibility for the direction of the activities of the Division of Chemistry was given to Dr. A. Cambron, who became Acting Director.

Following the close of hostilities a gradual reorganization of the Division of Chemistry took place, which was complete by the end of June 1946. Plutonium pilot plant and engineering work had been transferred to Chalk River as mentioned above, and the transition from war to post-war research programs had been completed. Because of the greatly increased size of the Division it had, in the spring of 1946, been divided into two parts, a Fundamental Chemistry Branch under the Director, Dr. Steacie, and an Applied Chemistry Branch under the Assistant Director, Dr. Cambron. The first of these will deal with such basic branches of chemistry as organic, inorganic, physical, and colloid chemistry, while the latter will deal with engineering and pilot plant operations, and with industrial chemistry related specifically to industries such as the textile, rubber, and protective coating industries.

B. INORGANIC CHEMISTRY

ATOMIC ENERGY

Before the completion of the Chalk River Laboratories, Canadian research and development work on atomic energy was centered in the Montreal Laboratories of the National Research Council, which were established for this purpose. However, other divisions of the Research Council as well as some other government laboratories were called upon to contribute to the program. The present account will be confined to the contribution of the Division of Chemistry of the Ottawa Laboratories of the National Research Council.

At first the full extent of the participation of the Division of Chemistry was not clear, and problems referred to the Ottawa Group multiplied more rapidly than laboratory space, equipment, and a staff could be assembled to handle them. In Ottawa, as elsewhere, a great sense of urgency attended the work, laboratories were rapidly blacked out, and those fortunate enough to participate in this epoch-making achievement began to burn the midnight oil behind locked doors.

The work done by the Division of Chemistry falls largely in that field of technology known as chemical engineering. For purposes of description, the organization at Ottawa may be divided into four groups, although actually there was an almost continual exchange of laboratory space, equipment, and staff. These groups were the Heat Transfer Group, the Water Treatment Group, the Pilot Plant Group, and the Design Group.

The Heat Transfer and Water Treatment Groups

The Canadian pile consists essentially of a large quantity of pure uranium and heavy water enclosed in a mass of concrete. The uranium is a natural mixture of the isotopes of mass 235 and 238. By fission of atoms of U235 enough neutrons are released to provide for absorption in the U238, for absorption in the other materials and impurities comprising the pile, for loss through the shielding surrounding the pile, and for enough residual neutrons to initiate the fission of more U235 atoms, thereby maintaining the chain reaction. Neutrons absorbed by the U238 result in the formation of plutonium according to the now well known reactions:-

$$^{238}_{92}U + {}^{1}_{o}n \longrightarrow {}^{239}_{93}Np + {}^{o}_{-1}e$$

$$^{239}_{93}Np \longrightarrow {}^{239}_{94}Pu + {}^{o}_{-1}e$$

In these equations mass numbers are shown as superscripts and atomic numbers as subscripts; Np and Pu are the symbols which have been adopted for the synthetic elements, neptunium and plutonium; $_on^1$ represents the neutron of mass unity and no charge; and $_{-1}e^o$ represents the electron of negligible mass and one negative charge.

The sum of the masses of the fission fragments is measurably less than the mass of the original U235, and the mass which is lost reappears as energy according to the well known Einstein equation $E = c^2m$, where c is the velocity of light. Expressed in common units, this energy equivalent of mass is 3.87×10^{13} BTU/lb. This energy appears initially as kinetic energy of the fragments of the disrupted atoms and as electro-magnetic radiation, but all of it is ultimately converted into heat within the shielding.

In brief, then, a chain reacting pile may be described as a machine in which U235 is burned to convert U238 into plutonium. Considered from this viewpoint, the heat associated with the process is a necessary evil and must be dissipated as efficiently as possible. The heat of fission of U235 has been measured by Henderson*, who reported a value of 177 Mev per atom; this is 1.74×10^{10} cals. per gram of U235 per day would be, therefore, about 380,000 KW. The removal of these tremendous quantities of heat becomes, then one of the major problems of pile design.

Since very large quantities of a cooling medium of relatively high specific heat are required, the choice naturally falls on water. And because a pile is a relatively compact machine compared with the amount of energy to be dissipated, the heat transferred per unit area of cooling surface must be exceptionally high. Water has a relatively high heat transfer coefficient for clean surfaces; but, in ordinary industrial practice, generous allowance must always be made for the scale which inevitably fouls the cooling surfaces, and the equipment must be overhauled regularly to remove the scale. However, in a pile, fouling of the cooling surfaces is a serious matter because of the high heat transfer rates which must be maintained, and because the surfaces are

* Phys.Rev. 58, 774, Nov. 1940.

not accessible for cleaning due to the intense radio-activity produced in the structural materials of the pile once it has been in operation.

Because of the great volumes of cooling water used, a water treatment plant is required large enough to serve a city. It is perhaps not generally realized that no two natural waters require quite the same treatment, and that a year or two of development work is usually required before satisfactory treatment is achieved. It was, then, the responsibility of the Water Treatment Group to produce a supply of water for cooling which would produce neither scaling of the cooling surfaces nor corrosion of any part of the water system. Considerations involving radioactivity phenomena also imposed certain restrictions on the type of water treatment employed. It was the responsibility of the Heat Transfer Group to test the water produced in order to determine that satisfactory heat transfer rates would be maintained under the conditions existing in the pile. They were also expected to keep a weather eye open for signs of corrosion and to investigate suitable inhibitors of scale and of corrosion for addition to the treated water.

Because this investigation had to be carried out on the actual source of water to be employed in cooling the pile, most of the work had to be done at the plant site; and because the program was necessarily a lengthy one, this work had to be started very early. Consequently, the buildings to house this work were the first permanent buildings constructed and members of this group were the first National Research Council personnel to take up residence at the plant site.

This little group spent the first winter in three "winterized" summer cottages, which were on the property about a mile from the actual plant site. It was November when they arrived, and winter was upon them almost before they were able to obtain a supply of fuel. Soon the bush road to the cottages was impassible to vehicles, and all provisions had to be hauled in on toboggans. During this early stage of construction, conditions at the plant site were most unfavourable for research activities. Water, steam, and electric power, all of vital importance, were obtained from temporary equipment which, suitable enough for construction purposes, was not sufficiently stable in output for the needs of a research laboratory and was subject to frequent, irritating breakdowns. A whole chemical and engineering laboratory had to be equipped in the wilderness, starting from scratch, and many discouraging delays resulted from the lack of simple items, unexpectedly required, which usually could not be obtained from Ottawa in

less than a week. So the group not only lived under difficult conditions, but also worked under difficult conditions.

Meanwhile, in Ottawa, the other half of the group worked night and day to design and fabricate the special equipment required, and to obtain the laboratory equipment and supplies so urgently needed at Chalk River. In those days even the highest priority was no guarantee of early delivery, and many of the items involved special or "critical" materials and unusual types of fabrication. By Spring, shortages of staff, laboratory space, materials, and equipment were largely overcome; more adequate supplies of steam, water, and electric power were available; many of the earlier problems of existing and of working had been resolved - and progress became much more rapid. Before the construction of the pile was complete, the Ottawa group were able to report that a suitable supply of water would be available and that the cooling provided would be satisfactory.

The Pilot Plant Group

The reactions by which U238 is converted into plutonium in a chain-reacting pile have already been described. When the charge is removed from the pile, it contains not only plutonium and unreadted uranium but also fission products in concentrations similar to that of the plutonium itself. When it is realized that the fission products produced include selenium, bromine, krypton, rubidium, strontium, yttrium, zirconium, columbium, molybdenum, antimony, tellurium, iodine, xenon, cesium, barium, lanthanum, cerium, and possibly other elements, the complexity of the separation and purification problem can be readily appreciated. Furthermore, these fission products are present in the form of highly radioactive isotopes, so that processing must be carried out behind several feet of concrete shielding, or its equivalent thickness of lead. This not only requires remote control of all operations, but prevents servicing of equipment, for the equipment itself becomes so radioactive that it cannot be approached, even when empty. The problem was, therefore, one of evolving a process capable of separating a fifth of all the elements in the periodic table from an element which had not even been discovered a few short years ago - much less its chemical properties understood - and of embodying this process in a plant which would be absolutley free of such normally tolerable inconveniences as leaking valves and pumps, and free of mechanical parts requiring lubrication. As only very limited information was available regarding the processes used in the United States, and entirely new process of separating fissile materials from the pile charge was developed by the scientists and engineers of the Canadian project.

It was the responsibility of the Division of Chemistry of the Montreal Laboratories to investigate the hundreds of possible operations and reagents for effecting these separations and to make a decision as to what process was to be used.

Once the Montreal Laboratories had sketched in the outlines of the chemical process, the task of filling in the details and of converting them into practicable plant operations was the responsibility of the Ottawa Group. Usually, when engineers are designing a plant, they are able to turn to standard handbooks for densities, solubilities, specific heats, and similar data. Because of the novelty of the materials involved, such basic data were not usually available in the literature, and the first task of the Pilot Plant Group was to obtain such fundamental information. They also had to make a more thorough, quantitative, laboratory study of the chemical reactions and systems encountered in the process, than had been attempted in the original exploratory investigations in the Montreal Laboratories. Two complete pilot plants were built and operated; in addition, many smaller pilot plant investigations were carried out to study specific items of equipment required to meet the unusual needs of such a process.

Unfortunately, no details of this work are available for release at this time, but the program may be summarized by saying that all the processes to be used in the Chalk River chemical processing plant were successfully operated on a pilot plant scale by the Ottawa Group, using non-radioactive material.

The Design Group

The actual working drawings for the construction of the Chalk River plant were prepared by the Special Projects Department of Defence Industries Limited. These drawings were made from detailed sketches and according to instructions prepared by the engineering staff of the National Research Council, and were at all stages subject to the approval of the National Research Council. In general, these sketches and instructions were issued by the Engineering Division of the Montreal Laboratories, but in the case of the chemical processing plants, this responsibility was assumed by the Design Group at Ottawa.

In the normal course of process development, laboratory and pilot plant investigations are essentially complete before plant design is seriously undertaken. In the present case, because of the urgency of the work, plant design was commenced before the pilot plant work was more than well started.

Indeed, it was not unusual for sections of the plant design to be tentatively completed on the basis of the preliminary investigations in Montreal, before reports were received on thorough laboratory investigation or on pilot plant operation. Changes had to be made in the designs as more information became available, but in this way a development program that would normally have required ten years was compressed into twice that many months.

Although, for purposes of discussion, the work of the various groups has been considered separately, in practice, much consultation and discussion was necessary between the Design and Pilot Plant Groups and the staff of the Montreal Laboratories, and between the Design Group and the Design and Operating Departments of Defence Industries Limited. All of these groups were working simultaneously, and problems arising in one group were continually being referred to another group, better able to cope with them.

The final culmination of this effort is a unique chemical processing plant at Chalk River, which represents a milestone in the industrial development of atomic energy. The plant has been designed so that additions can be made for the recovery of various by-products as this becomes desirable and construction will doubtless continue for some time.

REFRACTORIES

This industrial investigation was begun in 1925 at the request of the Dominion Government and on behalf of a key industry. Basic refractories are essential in smelting operations and it was considered in the country's interests to develop such an industry in Canada. The raw material available, a magnesitic dolomite, was at first regarded as unsuitable for this purpose. However, persistent research on the part of the group assigned to this project resulted in the development of a variety of useful products.

Chief of these, developed before and improved during the war, was a chemically bonded hearth refractory. During the war it came to be accepted as the best open hearth bed material, because it eliminated that lengthy burning-in-period and hence made a substantial contribution towards the rapid expansion of the steel industry, especially in the United States. Other products developed before World War II but continuously improved included spalling-resistant brick, refractory cements, chemically bonded brick and a refractory highly resistant to hydration.

One other major development was a refractory brick for use in rotary cement kilns. This has at least three times the life of any other competing product and is being sold all over the world.

During the war the nature of the research carried out changed substantially. There became available for the first time calcined brucite granules produced in a plant near Ottawa by a method worked out by the Bureau of Mines and much research was required to utilize this material commercially. The major applications have been in the manufacture of burned brick, the production of unburned brick by means of new chemical bonds, and the development of rammed, chemically-bonded hearth refractories. All of these have been important commercially, and Canadian and foreign patents have been obtained on the special processes involved.

Shortly after the outbreak of war it became doubtful whether the supply of high-grade magnesia available to the United Nations would be equal to the demand. In order to provide against eventualities, work was undertaken on the purification of Canadian raw materials, using a number of different methods. From impure material it was found possible to leach lime economically and to divide the residue of magnesia into two portions, one high in magnesia and low in silica, and the other relatively high in silica but nevertheless suitable for many applications. Patents were obtained on the process, but the necessity for its use has not yet arisen. Other methods novel in character have been developed through the initial experimental stage, and work will be continued as opportunity affords.

In keeping with the policy adopted early in the investigation, research has been actively continued in the improvement of products previously developed and processes already adopted. This work has resulted in many improvements which, although not of outstanding character individually, together represent important developments and have made possible the application of products in fields for which they were not previously considered suitable. As frequently happens, even small improvements in the properties of a material greatly enhance its commercial value and competitive position.

One of the major problems arising out of the war has been that of finding suitable raw materials to replace those cut off by the shortage of shipping. Of these the most important has been chrome ore, previously obtained by the Canadian refractories industry from India, South Africa, and Turkey. To a degree, substitution was found possible if

certain precautions were observed; the proportion of chrome ore consumed has also been reduced substantially. Certain chemicals previously used have been unavailable at reasonable prices, and satisfactory substitutes had to be found. As a result of such conditions and the research work carried out, the revision of manufacturing formulae has become almost a continuous process.

In this refractories investigation, as is usually the case, progress in research has been by no means constant, but may be said to have a wave form. When some important basic advance is made, it starts a series of fruitful experiments intended to discover all possible commercial applications, and the attention of the research staff is accordingly transferred to a considerable degree from fundamental to practical lines. Without fundamental research, however, practical applications may soon be exhausted, and in recent years as much attention as possible has therefore been paid to theoretical considerations, such as are involved in phase equilibrium diagrams. Recently some very promising new fields have thus been opened up, and these are now being explored in a practical way. It is expected that full patent protection can be secured, since the approach is distinctly novel.

Early in 1943 the research work carried out on refractories was transferred from the laboratories of the Council to newly erected laboratories at Kilmar, Quebec, near the mine and treatment plant of Canadian Refractories Limited. Operations are being conducted there on substantially the same scale as previously, and contact with the Council has not been completely broken.

During the twenty-year period in which this investigation has been carried on, well over one hundred Canadian and foreign patents have been issued on more than thirty different processes and products. The principal patents arising from work carried out during the war are listed at the end of Section B, together with the title of a paper contrasting the situation with respect to Canadian sources of basic refractory materials at the beginning of the first and second world wars. The patents listed are on different processes, with no duplication other than those noted.

RECOVERY OF MAGNESIA

When the war broke out it was obvious that the United Nations' requirements of magnesia would be greatly increased, both for refractories, to be used in the metallurgical industries, and for the production of metallic magnesium.

Fortunately the situation had already been given close study for some years, in connection with the refractories investigations carried out in the National Research Laboratories.

In 1940, at the request of the Metals Controller, a survey was carried out of potential sources of magnesia in Canada for all purposes, including refractories, metallic magnesium, and magnesium sulphate. This survey covered not only Canadian mineral deposits and a study of their commercial possibilities, but also an evaluation of many methods of recovering pure magnesia that had been suggested to the Dominion Government as being worthy of support. Later, the survey was extended to include the probable requirements of magnesia in Canada and the United States for 1942. It was evident that large new sources of magnesia must be developed.

As a result of this survey the following potential sources of magnesia for refractories and metallic magnesium were recommended to the Metals Controller as being of nearly equal interest:-

1. Magnesitic dolomite occurring at Kilmar, Quebec. Operations at this property were greatly enlarged during the war, and it was the country's major source of basic refractories, while exports were also made to many countries.

2. Brucite rock near Wakefield, Quebec, from which magnesia was recovered by a method worked out by the Bureau of Mines. This was likewise developed and became an important source of refractories.

3. Sea water treated with calcined dolomite. In this process magnesia is recovered in equal quantities from the two raw materials used. Although no commercial developments took place in Canada, a careful survey in Nova Scotia by the provincial Department of Mines led to the discovery of a deposit suitably situated for commercial operation, and experiments in the National Research Laboratories demonstrated that this raw material was entirely satisfactory for the purpose. Further, very large developments along this line took place in both Great Britain and the United States.

As a source of magnesium sulphate, it was indicated that serpentine treated with acid by a method developed in the National Research Laboratories held greatest promise. No commercial development has yet occurred.

Dolomite was recommended as the most promising source of magnesium metal in Canada. A commercial plant was built at Haley, Ontario, and was operated successfully until the end of the war. The evolution of the process employed in this plant is described in the following section.

MAGNESIUM

Although metallic magnesium was first isolated by Sir Humphrey Davy in 1808 and small amounts were produced commercially for photographic purposes as early as 1857, the industrial production of magnesium dates only from 1896, when a plant for the electrolysis of fused magnesium chloride commenced operation at Bitterfeld, Germany. Progress was slow, and even in 1937 world production was estimated to be only 27,000 tons per annum, of which half was produced in Germany.

In that year an Associate Committee on Metallic Magnesium was formed in Canada, consisting of representatives of the Departments of National Defence, Mines and Resources, and the National Research Council; and the Council was asked to conduct "literature and laboratory studies of the possible processes for the recovery of metallic magnesium, with a view to being able to assess the technical difficulties which might be met in the use of Canadian raw materials".

At that time most commercial processes were based on the electrolysis of fused magnesium chloride. The raw materials used were either carnallite ($MgCl_2.KCl.6H_2O$), magnesium chloride from natural brines, sea water, magnesite ($MgCO_3$), or dolomite ($MgCO_3.CaCO_3$); the process varied from place to place chiefly in the method used to obtain anhydrous magnesium chloride from the raw material available. Since magnesia can be obtained in Canada from magnesite, dolomite, or naturally occurring brucite granules ($Mg(OH)2$), the method of preparation of chief interest in this country was that represented in its simplest form by the equation:

$$MgO + C + Cl_2 \longrightarrow MgCl_2 + CO$$

The early experimental work carried out followed previous commercial practice in that fine magnesia was mixed with reducing agents, such as coal and either wood, flour or sawdust, and was briquetted with magnesium chloride solution, with which the magnesia forms magnesium oxychloride. The main purposes of the wood flour or sawdust were to produce a porous briquette, into which the chlorine could penetrate, and at the same time provide sufficient pore space to accommodate the voluminous magnesium chloride formed. Such briquettes, calcined to drive

off all contained moisture, were found to chlorinate rapidly at first, but the rate of reaction decreased with the progress of chlorination and became very slow by the time half the magnesia had been converted to magnesium chloride.

Experiments were then begun with granular magnesia of various degrees of density, and it was soon found that, if the granules were of a size sufficient to permit the chlorine gas to penetrate the mass and the magnesium chloride formed to run off in a molten condition, and were themselves sufficiently porous, chlorination took place with greatly improved rapidity and completeness. Patents were obtained on the optimum ranges of particle size and density. Calcined brucite granules obtained from the deposit near Ottawa by the method worked out by the Bureau of Mines (see p. B-16) were found to be particularly suitable in both of these respects. The anhydrous magnesium chloride produced experimentally was used in small-scale tests designed to familiarize the operators with commercial practice in the production of magnesium by electrolysis.

However, as the commercial production of an anhydrous magnesium chloride suitable for electrolysis is usually a complicated process, attention was given to the direct reduction of magnesium oxide by means of carbon, calcium carbide, aluminum, and silicon. Of these various reducing agents, the first and last had received most attention, and semi-commercial plants had, in fact, been operated intermittently using carbon, in what is now known as the carbothermic process. This has the disadvantages (i) that reduction with carbon requires a very high temperature (above 2000°C), (ii) that the vapours must be cooled very rapidly through the region of unfavourable equilibrium, and (iii) that the product is obtained as magnesium dust which requires further treatment to produce a compact metal.

The use of silicon, preferably in the form of ferrosilicon, has the advantages that (i) compact metal is formed in one operation, (ii) the operating temperature is only about 1200°C., and (iii) the much cheaper and more widely distributed dolomite may be used instead of magnesite. Indeed, in the ferrosilicon process, calcined dolomite is preferred. When straight magnesia is used, some of the silica first formed by the oxidation of silicon combines with magnesia; this not only reduces the utilization efficiency of the magnesia, but may, at the temperature used, lead to the formation of liquid silicates in the reaction retort, involving serious operating difficulties. With dolomite, on the other hand, the amount of lime present is theoretically just enough to combine with all

the silica formed, thereby leaving the magnesia free to be reduced by silicon, and the dicalcium silicate formed is so highly refractory that it can readily be removed from the retort in the solid condition. Further, the heat generated by formation of dicalcium silicate is sufficient to promote the desired reducing reactions

$$2MgO + 2CaO + Si \longrightarrow (CaO)_2SiO_2 + 2Mg$$

Work on the development of a commercial process based on this reaction was begun by the Council even before the experiments with electrolysis were completed. It was recognized that under Canadian conditions the advantages offered by it were substantial, particularly with respect to the availability of cheap dolomite and the fact that Canada was already a large producer of ferrosilicon in electric furnaces.

Although this reaction had long been considered elsewhere, and a number of patents on it had in fact been issued, it had not been found possible to overcome the numerous technical difficulties involved, and the process had consequently never been brought into commercial operation. Indeed, one authority after an extensive investigation had published a report stating that the successful application of the ferrosilicon process was impossible. This, then, was the situation when the Canadian group set to work.

It was soon found that, using calcined dolomite and 75% ferrosilicon, the reaction could be made to proceed at a satisfactory rate if (i) the constituents of the charge were finely pulverized, mixed and briquetted, in order to bring them into the most intimate contact possible, (ii) a temperature of about 1100°C. was used, and (iii) the process was carried out under a sufficiently high vacuum to remove as vapour all magnesium metal as rapidly as it was formed. Under these conditions the magnesium was found to distill out of the reaction mixture and to condense in the cool end of the alloy steel retort used.

One difficulty encountered in previous work was that the small amount of sodium, always present in dolomite, was reduced and condensed with the magnesium, thereby causing ignition of the condensed metal and consequent loss of the product when the retort was opened. Cooling the retort before discharging was both uneconomical and inconvenient. This problem was solved by fractional condensation of the sodium in the outermost part of the retort. A method was developed for opening the retort, removing the product and spent charge, and reloading, without removing the retort

from the furnace. The perfection of this technique made it possible to suggest commercial operation in a manner somewhat similar to that adopted in the established horizontal retort zinc process.

By the autumn of 1940, a small furnace with a four-inch retort was in regular operation. This furnace was examined by a group of industrialists from Toronto, who considered the development sufficiently promising that they formed a private company under the name Dominion Magnesium Limited. Under an agreement with the National Research Council, the company took over the work, and a pilot plant using larger retorts was constructed and placed in continuous production by July, 1941. In November of that year plans were made to build a commercial plant, and in December authorization for the construction of a two-ton per day plant was received from the Department of Munitions and Supply. A month later the plans were expanded to provide for ten tons per day. This plant was built at a cost of $3,300,000 at Haley, Ont., near the dolomite deposit from which raw material was obtained in the experimental work. Operations began in July, 1942, and the plant was later operated at a capacity of 15.5 tons of magnesium per day. It is interesting to note that the process and plant are also well suited to the production of metallic calcium, and calcium has, indeed, been produced by Dominion Magnesium Limited.

Planning for the Canadian plant coincided with the attack at Pearl Harbor and the subsequent rapid expansion of the magnesium industry in the United States. In November, 1941, the National Research Council's pilot plant was visited by officials of the United States War Production Board. After a special committee on magnesium examined the pilot plant in January, 1942, it was decided to adopt the process in the United States for a total daily production of 135 tons. All available information was given to the American engineers and the following plants were erected, at an estimated cost of $35,000,000.

	Capacity
New England Lime Co.	15 tons/day
Magnesium Reduction Co. (subsidiary of National Lead Co)	15 " "
American Metals Co.	15 " "
Permanente Metals (H. Kaiser)	30 " "
Ford Motor Co.	60 " "

Throughout 1942 the pilot plant at the National Research Council operated day and night, testing dolomites from various United States locations, and training men for plant operation. Since the process was new and had never

been operated commercially, many problems arose. Until the spring of 1944, when Dominion Magnesium Limited terminated its agreement with the National Research Council, development work continued, directed towards improving the process.

RECOVERY OF ALUMINA

Because of the enormous demand for aluminum metal during the war, and the great expansion of this industry in Canada, this country's requirements for bauxite were correspondingly increased. None is produced in Canada, and it became necessary to bring from the bauxite mines of South America 5000 tons per day, or more. The demands upon shipping were obviously very great, and thought was therefore given to other possible sources of alumina then bauxite.

The most obvious of these sources is clay, which, although it contains only about half as much alumina as bauxite, is available in any desired quantity at a cost little more than that of digging the material up with a steam shovel. Some alumina was, in fact, produced from clay in Germany prior to the war, and following tests in the laboratories of the Bureau of Mines in Ottawa a company was formed to undertake large-scale pilot-plant work in the United States. It was therefore known that recovery could be effected from clay, but it had not yet been demonstrated that the production of alumina of sufficiently high purity to satisfy the requirements of the industry could be obtained at a price competitive with that of bauxite.

Further work by the usual methods therefore seemed scarcely justified. However, two new approaches to the problem was investigated.

For some years The Consolidated Mining and Smelting Company of Canada at Trail, B.C., have been producing ammonium sulphate for use as a fertilizer by combining sulphuric acid made from the sulphur dioxide in their waste gases with ammonia produced by synthesis. It was thought that this procedure could to advantage be combined with the treatment of clay, the ultimate products being ammonium sulphate, as previously, and alumina. The latter, if not sufficiently pure for direct use in the production of aluminum, would at least afford a high-grade raw material for purification by the standard Bayer process.

Clay was first calcined to dehydrate it, to render the silica insoluble, and to convert the alumina to a more soluble form. The temperature range 550-900°C. was found satisfactory. Leaching was effected by using a sulphuric acid solution containing 60% of acid by weight. By this means it was possible to dissolve over 90% of the alumina from kaolin containing 40.6% Al_2O_3 and 1.0% Fe_2O_3, but some of the iron also dissolved. When, after filtering off the silica, ammonia was added to precipitate the alumina, this dissolved iron also came down, thereby contaminating the alumina. Three possible methods of purification were proved applicable:-

1. Magnesium powder was found to precipitate iron readily from the slightly acid solution resulting from leaching, and it could then be separated by filtration.

2. The addition of ammonium sulphate to the filtrate from the silica precipitated ammonium alum as crystals free from iron. By dissolving these crystals in water and adding ammonia, alumina was precipitated in a state of satisfactory purity, and, following filtration, the ammonium sulphate was recovered by evaporation.

3. The impure precipitate of alumina produced by the addition of ammonia to the original clear leach liquor was dissolved in caustic soda (the iron remaining insoluble) and the solution filtered. Alumina was then precipitated from this solution as in the usual Bayer process.

These methods were worked out only on a small laboratory scale, but they appeared to give considerable promise of the cheap production of alumina of high purity. There was no opportunity to continue the work on larger quantities in a cyclic process, such as would be necessary before a pilot-plant operation could be justified.

DRYING AIR FOR BLAST FURNACE USE

The exceptional demand for steel during the war and the shortage of scrap led to greatly increased requirements for pig iron, and consideration was therefore given by industry to methods of increasing production. Of these, one of the most promising was the drying of the blast used. The method had been introduced by Gayley in 1904, but owing to the high capital and operating costs involved in his refrigeration system most of the dry blast plants were discontinued by 1916. In the early part of the recent war, several plants were built using modern methods of drying blast, and in some

cases excellent results were claimed, production being substantially increased and coke consumption decreased.

Work carried out previously in the National Research Laboratories had indicated that one of the best and most economical drying agents was active silica, produced by the acid treatment of serpentine. Steps were therefore taken to investigate its possible application to the drying of blast furnace air. A search of the literature was made, and the subject was discussed with experts at steel plants at Sault Ste. Marie, Ontario, at Sydney, N.S., at Middletown, Ohio, and at Baltimore, Md. Experiments were carried out to determine (i) the resistance to flow of blast passing through active silica of various particle sizes, (ii) the rate of adsorption of moisture, and (iii) the conditions necessary for the regeneration of the silica. From this data an adsorption system was designed in outline.

The results of this work indicated that an adsorption system using active silica might be the cheapest in both capital and operating costs, and a paper describing the findings was published. Since that time, however, it is believed that no new dry blast plants have been erected, and there has consequently been no opportunity to try the system on a commercial scale.

UTILIZATION OF OPEN HEARTH SLAG

For nearly half a century a large steel plant has been operated at Sydney, Nova Scotia, and during the whole of this period the open hearth slag produced has been wasted. The magnitude of this waste will be realized from the fact that the phosphoric acid and lime contents of the slag have substantially exceeded the quantities of these materials used as fertilizer and soil amendment in all of the Maritime Provinces put together.

The major reason for this continued waste was the fact that, because of the use of fluorspar in the steel furnace operations, the phosphoric acid in the slag was of low availability to plants. Such growth experiments as were carried out with it indicated that its value as fertilizer was quite small, and its application to the soil was believed not to be justified by the results obtained.

This problem was brought to the attention of the National Research Council late in 1938 in the form of a resolution passed by the Maritime Board of Trade, then in session at Sydney. The Council shortly afterwards wrote

the Dominion Steel and Coal Corporation, suggesting that the availability of the phosphoric acid might be improved by rapid chilling of the molten slag, as by granulating it in water. Preliminary experiments carried out by the company shortly afterwards indicated that the idea might have merit, but no further action was taken until the spring of 1941, when the Nova Scotia Economic Council asked the National Research Council to make a complete investigation. This work was undertaken in co-operation with the Dominion Steel and Coal Corporation and a slag committee set up by the Premier of Nova Scotia under the chairmanship of Dr. G.R. Smith, Provincial Agricultural Chemist. Later, when granulated slag became available, comprehensive greenhouse and field tests were carried out over a period of several years by the Dominion Department of Agriculture, through its Central Experimental Farm at Ottawa and its experimental and illustration stations in Nova Scotia, and also by the Nova Scotia Department of Agriculture.

In the early experiments the slag was not chilled with sufficient rapidity and the availability of the phosphoric acid was, in fact, reduced during the succeeding drying operation. Although growth tests showed some increase in yield for the garden and field crops tried, the results were not regarded as conclusive. A complicating factor was an unfavourable growing season in Nova Scotia, as a result of which the effects of drought in some cases completely masked the effect of the fertilizers applied. In the spring of 1944, however, the Steel Company granulated several hundred tons of slag by a semi-commercial method, and this was tested on a large number of farms in various parts of Nova Scotia. Fair results were obtained in that season with grain, in spite of exceptionally dry weather, and in 1945 grass was grown on the same areas with very striking results. On many farms the increased yield of hay from the application of one ton of slag per acre amounted to 50-75% and, on the average for all farms, the application of slag gave a highly favourable result, both as to increase in yield and improvement in the quality of the hay crop. Higher applications of slag gave still greater yields, but not sufficient to pay in one year for the extra expense involved.

These results were regarded as extremely promising, and steps were immediately taken to put in a commercial plant for slag granulation and grinding. It is expected that this plant will shortly be in operation, and that one of the major waste products of the Maritime Provinces will thereafter be converted into a valuable fertilizer material.

The Sydney steel plant is the only one in Canada burning out an open hearth slag containing much phosphoric acid. In Britain, however, there are many such, and information regarding progress in this investigation was therefore sent currently to the British Ministry of Supply. They expressed great interest in the project, duplicated the literature review for distribution to all British steel producers, and undertook extensive experimental work to determine the applicability of the findings to British practice. To date, the final results have not been received in Ottawa.

VANADIUM RECOVERY

In the spring of 1942 there developed a critical shortage of vanadium, which finds one of its main applications as a constituent of tool steel. About this time the Council learned that the ore of the Wabana Mine, Newfoundland, contains about 0.05% vanadium, and that as a consequence the pig iron produced at Sydney, Nova Scotia, by the Dominion Steel and Coal Corporation, has a vanadium content of slightly over 0.10%. Although this proportion is not large, the total quantity going to waste amounted to more than one ton per day which, if recovered, would go far toward meeting Canada's most urgent requirements of this important alloying element.

Because of this situation, a study of the literature on vanadium recovery was made to determine whether there was any reasonable chance of obtaining the vanadium from Sydney pig iron in marketable form. Some encouragement was obtained and, as a result, an investigation was undertaken at the request of the Metals Controller in the summer of 1942, and continued for about six months. During this period a large number of experiments were carried out at Sydney in co-operation with the staff ofthe Dominion Steel and Coal Corporation. The oxidation of the vanadium in the pig iron was brought about by the addition of iron oxide to the blast furnace ladles at the time of tapping, and the slag so produced was resmelted with substantially complete recovery of both vanadium and iron. The results were regarded as sufficiently encouraging that work was done on the design of a rotary reaction drum which might permit the partial desiliconization of the pig iron and, at the same time, substantially complete oxidation of the contained vanadium. It was proposed to resmelt the slag produced and again concentrate the vanadium by treatment of the resulting pig iron with iron oxide in the same equipment. It was believed that steel production would be increased by the oxidation of silicon in

the reaction drum and the consequent shortening of the open hearth operation, and that vanadium could be produced in marketable form at a cost below the pre-war price of the metal.

Early in 1943 these proposals were laid before the Metals Controller. By this time, however, the vanadium situation had become much less critical, steps already had been taken to increase the production of vanadium in the United States and that country had agreed to supply Canada with her complete requirements of the metal. Under these circumstances it was felt that the capital expenditure required for the erection of a vanadium plant at Sydney by the Dominion Government could not be justified.

NICKEL, COPPER, AND PLATINUM METALS

Three projects were undertaken, all arising out of practical experience in the Canadian nickel industry, as follows:-

(1) The recovery of platimum metals from Monel metal matte. In the usual practice, the nickel-copper matte chosen for the production of Monel metal is one relatively low in platinum metals, and by the usual methods of treatment their recovery is not profitable. It was suggested that at least part of these metals could be cheaply recovered by blowing a charge of regular matte to produce a small quantity of copper and utilizing the selective solubility of this element for gold and the platinum metals. Preliminary experiments in the laboratory indicated that this method was promising, but there was no opportunity to follow it up on a larger scale.

(2) Selective separation of copper and nickel in the converting operation. Much of the copper reverberatory and Orford mattes produced by the International Nickel Company contains a little nickel, which subsequently enters the anode metal and considerably increases the cost of the subsequent refining operation. It was hoped that, because of the easier reducibility of copper, most of this metal could be separated in the converting operation substantially free from nickel. Preliminary laboratory experiments proved that some concentration took place under such conditions, but it was not regarded as commercially promising.

(3) Analytical separation of the platinum metals. One of the most difficult analytical or assaying problems is the separation of the platinum metals from one another and from

gold and silver, with which, in Canadian ores, they are invariably associated. Inquiries had been received from time to time as to practical methods of separation and determination. These eventually led to a review of the literature and to the preparation of a paper describing methods used by the writer in his own work, for distribution to inquirers.

ATMOSPHERIC POLLUTION

Several requests were made during the war for investigations regarding atmospheric pollution by heavy industry and the proper disposal of waste products resulting from such operations to prevent serious damage to agricultural and ornamental plants and forest species.

The Trail Smelter Question

At Trail, British Columbia, is located the largest lead-zinc smelter in the world, and in its operation large quantities of sulphur dioxide have been discharged into the atmosphere. In 1928 the United States Government officially entered claims against the Dominion Government for damages alleged to have been done by fumes from the Trail smelter to farms and forests in the northern part of the State of Washington. The Dominion Government thereupon asked the National Research Council to make a full investigation of the matter, and work was subsequently carried on by the Council almost continuously for a period of about ten years. Later, and during the war, work was mainly confined to periodical surveys of the area and assistance in connection with the prosecution of the case before the courts.

In 1940 reports were submitted to the Department of External Affairs, covering the atmospheric sulphur dioxide conditions over the period 1937 to 1940 from data obtained from the operation of a number of automatic recorders in the Columbia river valley, on both sides of the International Boundary, in the path of fumes emitted by the plant at Trail. Records of sulphur dioxide concentrations at three points in the Columbia River Valley, south of Trail, are still being regularly scrutinized by the staff. However, remedial measures undertaken by the Consolidated Mining and Smelting Company of Canada have resulted in a reduction in the amount of sulphur dioxide evolved, and have eliminated all damage south of the International Boundary.

Air pollution by smelter smoke in Sudbury region

The Sudbury area of Ontario has for many years produced 75-90% of the world's nickel, and has been at the same time one of the major copper producers and the source of more than half of the world's supply of metals of the platinum group. In conjunction with these operations great quantities of sulphur dioxide have been produced by the three large smelters in the Sudbury district. Unlike Trail, which lies in a valley that serves as a natural channel for the gases, Sudbury is in relatively flat country, and by the use of high stacks the operating companies have been able to keep to a minimum the damage done by smelter fumes. The Ontario Department of Mines has long maintained an arbitrator in Sudbury, and he has adjudicated any claims for smoke damage.

During the war a major increase took place in the scale of operations in the Sudbury district, and the evolution of sulphur dioxide to the atmosphere was correspondingly increased. The Ontario Government became concerned lest the important provincial forests should be seriously damaged and asked the National Research Council for assistance in studying the problem. Following consultation in the spring of 1944 plans were laid for surveys of the vegetation, for meteorological studies, and for the determination of the concentration of sulphur dioxide in the atmosphere and of sulphur in the needles of conifers. In several localities trees were bored, pencils of wood removed, and annual growth carefully measured, in order to determine to what degree, if any the trees had been damaged by sulphur dioxide in previous years. In 1945 automatic sulphur dioxide recorders were set up by the Council's staff in several districts, and general supervision was given to the investigation.

During 1945 the following studies were carried out in the course of this investigation:-

1. Continuous measurements of sulphur dioxide concentration, by means of automatic recorders, at four stations situated at distances varying from 20 to 60 miles from the smelters;

2. Measurements of the concentration at various altitudes in the path of the smoke stream by means of portable equipment on an airplane in flight;

3. The simultaneous determination of sulphur trioxide and sulphur dioxide by mobile portable equipment;

4. The extent of penetration of smelter smoke by a study of the abnormal sulphur content of various species of plants within the smoke area in comparison with similar species in normal control zones.

Three additional automatic sulphur dioxide recorders were constructed for use in 1946.

The observations are being correlated with studies of meteorological conditions at ground level and at various altitudes, and with biological investigations, in order to delimit the zones of smoke damage and arrive at a satisfactory solution of this problem. This work is still under way, although the decreased scale of operations resulting from the cessation of hostilities has substantially reduced the sulphur dioxide hazard to vegetation.

CHARCOAL FOR FUSE POWDER

Production

The Charcoal used in manufacturing certain slow-burning fuse powders according to British specifications is obtained by carbonizing wood from the species Rhamus frangula (alder). In September, 1939 the National Research Council received from the British Mission an enquiry regarding the availability of this tree in Canada. A survey, conducted by the Division of Applied Biology, resulted in the discovery of two stands in Eastern Canada, one of which was in the vicinity of Ottawa, and samples were sent to Britain.

While this search was in progress, it was suggested that carbonization experiments be carried out in Canada, and as no information was available regarding the special equipment used in this work or the qualities sought in the charcoal produced, the Department of National Defence was asked to obtain this information from England. Some details of the plant and manufacturing methods were received in January, 1940, but work from Britain indicated that no further action was required at the time.

However, British supplies of Rhamus frangula were cut off by the fall of France, and an urgent request was received in August for six tons of this charcoal, to be delivered during 1941. It was decided that this work should be carried out by the Chemistry Division of the National Research Council. Equipment was ordered at once and factory space rented nearby.

In view of the urgent need for charcoal there was no thought of trying to follow the British plan of air drying the wood. A new method of debarking the wood was developed in which the wood was predried at 110°C., making the bark hard and brittle. Crushing the wood then reduced the bark to a fine powder, which was rejected as undersize by the pre-carbonization screening.

Full scale, twenty-four-hour operation began late in May, and, despite minor difficulties it was possible to keep the Beloeil explosives plant supplied with its needs. In all, slightly over six tons of fuse powder charcoal were produced in Ottawa and shipped to Beloeil. Some of this charcoal was prepared from alder supplied by Defence Industries Limited.

In the meantime, Defence Industries Limited had been instructed to take over the charcoal production. Because of experience in assembling the carbonizing units, the Research Council was requested by Allied War Supplies to purchase and to assemble for Defence Industries Limited the carbonizing units required. Engineers from Defence Industries Limited visited Ottawa to observe the plant in operation, and were given all information based on experience in Ottawa to aid in designing their proposed plant at Beloeil. By mid-October, 1941, the Beloeil plant was able to take over all production, but the Research Council continued development work for a time.

Development and Research

From the very start of fuse powder charcoal production, the relative scarcity of R. frangula had caused concern. It was not a native shrub, there was a definite limit to the supply available, and plantation planting could not be arranged in time to bring in a supply for the years 1943, 1944 and 1945. The most promising approach seemed to lie in the use of alternative woods. Accordingly sample batches of carbonized R. purshiana, R. cathartica, Canadian alder, willow, sumac and chokecherry were made and sent to Defence Industries Limited. Final reports on the use of these charcoals was quite effective, and that after some preliminary trials an acceptable powder was made from it.

Owing to the scarcity of wood, the losses due to sawdust and fines caused some worry. Some reduction in the sawdust loss was made by using a chopping device instead of a saw for cutting branches of smaller diameter. Later it

was thought that the loss of fines could be eliminated by not grinding the pieces till after carbonization; it was also thought that debarking and carbonizing might be carried out in one operation. In this treatment, the short pieces of wood with the bark still on were chopped into the carbonizing drum and brought up to the desired temperature more slowly than when the wood was predried and ground. When the run was completed the charge was emptied onto a coarse screen and the fines, which were chiefly from the carbonized bark, were discarded. The charcoal remaining on the screen was crushed and used as usual. With this process, control was more difficult, but the product obtained was almost as uniform as that produced by the standard method.

As initial operating and control difficulties abated, there was some interest in determining the properties of charcoal which made it suitable, or otherwise, for fuse powder production. It was thought that if any such relationship could be found, it might be of value and save considerable time when alternative woods were being investigated.

The reactivities of charcoals were compared by passing a controlled flow of oxygen through a sample of charcoal, immersed in a bath where the temperature was being slowly raised, and noting the bath temperature at which the temperature of the charcoal rose above that of the bath. By microscopic examination in polarized light some carbons were found to be completely isotropic, while others were completely anisotropic; some were a mixture. The refractive indices also were measured. There appeared to be some correlation between these optical properties of the charcoal and the performance of the fuse powder. This work was reported to Defence Industries Limited, but was not carried further at the Research Council because it had reached a point where any further progress depended on making and proving batches of powder; at the time, production was much too urgent to permit following up experimental work, no matter how promising. Besides that it appeared, from proof of powders already made, that alder would prove in place of R. frangula, and indeed, this work on optical properties received some degree of confirmation thereby.

DEICING AND DEFROSTING

The accumulation of ice on the propellers and aerofoils of aircraft in flight constitutes one of the greatest remaining hazards to aviation. Many efforts have been made to prevent or remove such accumulations. As a corollary, it is also necessary, before the plane takes off, to remove frost

which has accumulated while the aircraft was grounded. These and related problems have been the subject of intensive investigation in the Division of Chemistry.

Frost on Grounded Aircraft

The application of suitable chemical substances to the exposed surfaces of the aircraft will cause the frost crystals to form like an inverted pyramid with only the apex attached to the surface. Such crystals are then readily removed by brushing. Work was started on this subject before the war at the request of Canadian Airways, and was continued in the hope that it might be of value for use with dispersed aircraft.

The compound used must ideally meet all the following requirements:-

(a) It must be cheap;
(b) It must be easily applied;
(c) It must not collect dirt;
(d) It must not produce a slippery surface;
(e) It must be effective on all classes of base materials;
(f) It must be insoluble, and not chemically changed by rain;
(g) It must not lose its effectiveness due to the usual accumulation of dirt, oil, and gasoline residues;
(h) It must not deteriorate due to ultraviolet light;
(i) It must remain effective for a reasonably long time;
(j) It must not damage wing paints;
(k) It should preferably be invisible, or at least nearly so, on all base colours and finishes;
(l) It must be effective over a great range in weather conditions, with the consequent variations in the type of frost produced;
(m) It must not interfere with other deicing procedures, and should still remain effective after such procedures have been used.

This is a formidable list of requirements, and, although over 200,000 tests have been made to date, no material has yet been found which is entirely satisfactory. No treatment has been found which is effective after alcohol or sodium nitrate solution has been used for deicing, and the coatings still tend to be slippery, although they have been considerably improved in this respect. Finally, all materials which are stable to ultra-violet light damage some wing dopes, while effective materials which do not cause paint damage are destroyed by ultra-violet radiation. For example, a mixture was found which was shown by ground tests to be very effective,

except for (d) and (m) above; however, one hour's dry flight in mid-winter entirely destroyed its effectiveness just where it matters most - on the upper wing surfaces. This remarkable difference at the higher altitude is thought to be due, not to the greater intensity of ultraviolet light there, but to the presence of shorter, very destructive, wave lengths, which are not present at ground level due to atmospheric absorption. Work on this problem is continuing.

Carburettor Deicing

Because of certain disadvantages in the use of ethanol blended with fuel for carburettor deicing, a study was undertaken of the properties of other substitutes. The significant properties are ice-melting capacity, freezing point, volatility, heat combustion, and general suitability as a fuel blending agent.

Methanol is obviously by far the best and cheapest ice melter, and in most chemical properties is superior to ethanol. However, it has a rather poor fuel value, and has the disadvantage of being rather insoluble in gasoline, especially if traces of water are also present. A number of materials were investigated for blending with the methanol, and their octane ratings were determined by the Gas and Oil Laboratory of the Division of Mechanical Engineering. N-propyl alcohol, with an octane rating of 138, proved quite outstanding in all respects.

Propeller Deicing

(a) Using Deicing Pastes:-

Deicing pastes for wings were again shown by ground tests to be ineffective, but similar use on propellers appears to have some value since centrifugal force then removes non-adherent ice. A feeder was designed which feeds soft deicer paste to the blades continuously and produces very good blade coverage, without appreciable expense or loss of power. A number of commercial pastes were tested and also a number of locally compounded pastes. This work has been continued since the close of hostilities and very promising results are now being obtained.

(b) By Heating Electrically:-

In co-operation with the Division of Physics and the Deicing Laboratory of the Division of Mechanical Engineering, a Neoprene heater shoe was developed to cover the leading edge

of the propeller blade. Incorporation of acetylene black in the Neoprene renders it electrically conducting, the degree of conductivity being controlled by the type and quantity of the acetylene black and by the method of incorporation. In cooporation with Shawinigan Chemicals Ltd., studies were made of Shawinigan acetylene black in natural and synthetic rubber. The difficulty that had to be overcome in this case was the non-uniformity of the electrical conductivity of compounds containing Shawinigan black. When the black was incorporated into the rubber in the ordinary way on the rubber mill, the structure of the particles was broken down considerably and in an uncontrollable manner, resulting in low and non-uniform conductivity. By determining beforehand the degree of structure of the black by an absorption test, and by adding the black to a solution of the rubber, the structure was preserved and a predictable and unfirm conductivity obtained. This method was used in the manufacture of the electrically conducting Neoprene heater shoes.

These heater shoes are effective in removing ice that has already formed, since the layer next to the propeller is melted, permitting the removal of the non-adherent ice by centrifugal force. Methods employing alcohol or similar fluids are ineffective against ice which has formed before the application of the deicing fluid. The electrical method has the additional advantage that it is not dependent on the consumption of material, the supply of which may become exhausted if the flight or icing conditions are unduly prolonged.

Successful flight trials have been made with propellers so-equipped, patents have been applied for, and the problems encountered by the rubber manufacturers have been largely overcome.

Rate of Icing Indicator

An instrument has been developed to indicate rate of icing, thus permitting prediction of the plane's future condition. Previous instruments have only been capable of measuring the thickness of ice after it had accumulated. The first model was flight tested by the Deicing Laboratory and gave satisfactory indications. Work is being continued to include a few desirable refinements.

Ice-Loosening Grease

At the request of the Navy, attempts were made to prepare a grease which would cause ice to form specially loosely on metal surfaces, so that accumulated ice might be removed more readily. Although greases were developed which were satisfactory with fresh water ice, the tough nature of sea water ice is so different from glassy fresh water ice that at sea there was no improvement by using such a grease.

RAIN REPELLENTS FOR AIRCRAFT WINDOWS

Since windshield wipers are not entirely satisfactory at high speeds of flight, it was hoped that a suitable water repelling coat for aircraft windows could be developed which would shed the rain drops without allowing them to run into a continuous film. The ripples on a continuous film, such as ordinarily occur during flight in rain, break up the light and prevent clear outlines from being visible, while separate drops leave clear glass between them, and, since the wind keeps the drops moving rapidly they can be more or less disregarded.

Early tests soon showed that the only substances having much promise for this purpose are the non-polar semi-solids, since polar substances do not shed water; solids, although they may be repellent at first, soon lose this property; and liquids, even in solid vehicles, are rapidly removed by the water droplets.

The main difficulty in this process, however, is in obtaining any degree of attachment between the glass and this non-polar surface film, since only polar substances adhere naturally to glass.

The first success in obtaining such a non-polar bond to glass was accomplished by contaminating the glass with antimony and adding a selenium compound to a wax film. The selenium is firmly bound by the antimony and the wax film is retained quite adequately. However, this method suffered from the great disadvantage that it required extraordinary care in cleaning the glass surface.

The next improvement was the use of dimethyl silicon dipalmitate. For twelve months all efforts had failed to obtain any valuable result from it, until it was found that when rouge was incorporated with the compound merely rubbing briskly on the glass caused activation of some sort, and a

strongly repellent film was readily produced which would not wet with water, and which was remarkably difficult to remove.

It was then found that this is a very general property of organic silicon compounds; friction on glass with rouge breaks them, and the fragments attach themselves to the glass. All organic silicon compounds available acted similarly, producing varying degrees of attachment and repellency.

From a general consideration of such compounds it was considered that if the friction could activate it, the finest result might be secured if the silicon compound had a chain of two or more silicon atoms, and all side chains were hydrocarbon. Consequently hexa-ethyl-di-silicon was synthesized, and it was found to produce very good results indeed.

At this point the use of powders other than rouge were tested, but the only ones found to produce the effect were barium sulphate, rouge, and lampblack, being better in the order given. Tests of a great variety of lampblacks and carbon blacks showed that acetylene black was markedly better than all others. Shawinigan Chemicals Limited made available a wide range of acetylene blacks, and of these "Super Hyflow" was selected as best. It is now used as standard.

Silicon bonding compounds were then synthesized containing silicon chains of one, two, three, and four silicon atoms, with hydrocarbon side chains up to sixteen carbon atoms. The butyl compound with three silicon atoms was found to produce the most repellent bond, while the butyl compound with only two silicon atoms was found to give the strongest attachment; in cases where it is desired that the residual bonding compound should be volatile, the ethyl compound with two silicon atoms, carefully purified from higher constituents was found to give best results.

The wax coating was then reinvestigated, and the purification of each ingredient proved to be of the greatest importance. It was found that even the cloth by which the materials are applied must be specially purified to give best results.

Further complications are also introduced by differences in the techniques required to produce satisfactory results under such different conditions as:-

(a) First application on new glass windows;
(b) Maintenance on glass windows;
(c) Application to plastic windows, both acetate and methacrylate;
(d) Application to high temperature;
(e) Application below the freezing point;
(f) Application to windows during rain;
(g) Possible renewal during flight if continued rain ultimately wets the window;
(h) Effects of alcohol or heat used for deicing;
(i) Effects of gasoline or oil.

At first, different materials or techniques were necessary for each of these conditions; but after a thorough investigation, with constant modification of the technique, the waxes and the purification methods, a simple kit and a uniform technique were developed which are entirely satisfactory under all conditions.

The kit consists of (i) a packet of purified pieces of cloth for the applications, and (ii) two collapsible lead tubes containing respectively the bonding paste and the wax paste. The instructions for use are as follows: clean and dry window with the bonding paste, and polish; rub hard all over again with the bonding paste, and polish; apply the wax, and polish at once.

This treatment leaves an invisible film, free from optical distortion, and provides very clear vision during flight in rain without windscreen wipers. It has the advantages that:-

(a) Application is rapid, requiring only a few minutes per window;
(b) The base material may be any kind of plastic or any kind of glass (including fused quartz);
(c) Plastic windows may be treated wet or dry; glass windows may be retreated wet or dry, although the original application to glass must be while dry;
(d) The coating can be revived in flight either wet or dry with a hand-operated wiper, or with a spray of special wax solution applied in a manner similar to alcohol deicing;
(e) The materials can be applied at any temperature from -40°F to +125°F;
(f) Curvature of the window does not limit the action as it does in the case of windscreen wipers;

(g) The treatment helps prevent scratching of plastic windows, and produces the visibility of existing scratches;
(h) Flight under dry conditions does not reduce the subsequent life under rain, and treated windows may remain dormant in or out of the hangar without damage;
(i) Alternate exposure to rain and to dry air does not accelerate deterioration;
(j) So far as is known, the coating is not affected by any deicing procedure;
(k) The coating is not damaged by oil, gasoline, alcohol or glycol;
(l) Oil may be removed in flight with the "reviver" spray;
(m) The materials needed are cheap, non-corrosive, free from chlorides, and stable in storage.

A standard ground test has been developed in which a blower forces air and water droplets against the treated glass in a manner corresponding to flight, at 300 m.p.h., through rain falling at a rate of one inch per hour. Under such test, the initial clear vision obtained by this treatment is retained unimpaired for seven hours; then deterioration becomes noticable, and after seventeen hours, performance is the same as for untreated glass. Flight through hail removes the film entirely, but as explained above, the film can be renewed in flight.

Tests in flight conducted by the Test and Development Establishment of the R.C.A.F. proved very favourable, and after extensive service tests the Air Force has adopted the treatment as standard.

ANALYTICAL LABORATORY

This laboratory did not of itself undertake any research problems, but it co-operated actively in the work of many of the other laboratories. During the five and a half years of the war, the Analytical Laboratory of the Division of Chemistry made some 50,000 determinations on approximately 17,000 samples. However, the activities of this laboratory can hardly be considered "routine" since the infinite variety of materials submitted for analysis was a continual challenge to the technical ability of the staff and required chemical proficiency of the highest order.

These were in addition to analyses of textiles, leathers, paints, detergents, corrosion products, metals and alloys, etc. The laboratory, on request, also prepared

bibliographies on a number of subjects for other laboratories of the Division.

SALVAGE

At the request of the Department of National War Services, the National Research Council undertook in 1940 to study the question of salvage with a view to determining whether it would be advisable to institute a national salvage campaign, and a member of the staff of the Division of Chemistry was asked to investigate this question and to prepare a report. At that time there was considerable public agitation for a salvage campaign, but some of the controllers of supplies were known to be opposed to it.

In the time available it was not possible to conduct an exhaustive enquiry, but each of the principal types of salvage materials was examined (i) as to its statistical position, (ii) as to the possibility of obtaining additional supplies through a salvage campaign, (iii) as to the ability of industry or export markets to absorb any additional supplies which might be collected, and (iv) as to methods of collecting and marketing.

At the time this survey was undertaken, no real shortages of supplies had developed. The Department of National War Services was advised that under the conditions then prevailing the collection of additional salvage material could be justified only if carried out on a voluntary basis, and that even then the financial returns would probably be small. If collections were made, it would be advisable to include all types of scrap of potential value, and not merely those of one kind, as was then being done. It was found that the collection of heavy iron scrap (in the aggregate of greatest value) and that of the common base metals and paper were already being fairly well taken care of, but that a moderately favourable situation existed with respect to aluminum, tin alloys, wool and cotton rags, and rubber. If a campaign were undertaken, it was recommended that the collection of scrap be handled through established dealers.

MISCELLANEOUS INORGANIC INVESTIGATIONS

A number of minor investigations in the field of inorganic chemistry were also undertaken in the Divisional Laboratories.

Purification of Sea Water

At the request of the Navy, several methods of producing potable water from sea water were investigated. Some work was done on synthetic resin ion exchangers. Eventually a still was developed which operated automatically if the relative humidity was less than 100%. Maximum distillation rates of 60 cc. per sq. ft. of still surface per hour were obtained.

Potassium Tetroxide

At the suggestion of the late Sir Frederick Banting, potassium tetroxide was produced in small quantities for experimental purposes. This material has been used in the ventilation of submarines to maintain the oxygen supply, and has also been considered for use in a special type gas mask in which it provides oxygen independently of the external atmosphere. Carbon dioxide exhaled from the lungs, coming in contact with potassium tetroxide, reacts to form potassium carbonate and to liberate oxygen.

Methods of producing potassium tetroxide were studied in the laboratory, and apparatus for making this chemical on a semi-pilot scale was built. In this apparatus potassium (vaporized by heating a mixture of potassium chloride and sodium metal) was blown by a current of nitrogen, through a burner, where, it was mixed with oxygen, into a larger chamber, where it burned. The product of combustion settled out in the chamber as a fine powder of potassium tetroxide. As this material in its original form was too finely divided for use in respirators and the like, it was granulated by stirring, while warming, just enough to make the particles coalesce.

Asbestos

The utilization of Canadian asbestos had been a subject of investigation by the Division of Chemistry for some time before the war. As this was a long term project not likely to have much bearing on the conduct of the war, these activities were soon terminated and the most recent results were published.

During the war Canada continued to export considerable quantities of certain grades of asbestos to neutral countries. The Department of Trade and Commerce feared that this material might contain quantities of longer fibres that could be recovered and shipped to Germany by pro-German neutrals.

A lengthy investigation was thereupon launched by the National Research Council, which led eventually to the placing of an embargo on those grades of asbestos which were found to be of potential value to the enemy.

Asdic Recorder Paper

The Navy experienced considerable trouble with their asdic recorder paper during storage. This paper was essentially starch-iodine paper and it deteriorated due to the liberation of free iodine. The addition of a small percentage of sodium bicarbonate or sodium phosphate were found to prevent deterioration of the paper.

Comfort Meter

At the request of the Navy a simple instrument was designed that gave an integrated reading of temperature, relative humidity, and air circulation.

Magnesium Bombs

The magnesium pilot plant, with the aid of the Mines Branch Laboratories, prepared magnesium powder for experimental flash bombs in connection with the research program in night photography for the R.C.A.F.

Practice incendiary bombs were constructed for the Navy and for civilian defence organizations.

Packaging Problems

Excelsior (wood wool) was shown to be specifically bad for the packaging of unwrapped, greased or ungreased, metal parts, the effect probably being one of holding moisture in actual contact with the metal.

Qualification testing of silica gel dessicants for use in tropical packaging, where moisture free atmospheres are required, has been carried out from time to time.

Goggles

Impact tests on hardened glass lenses for chippers goggles have occasionally been carried out, as have also tests on plastic goggles and protective (steel toe) shoes. When this work was first requested it was found that there were no accepted Canadian standards for such safety equipment. This led to the formation of a Canadian Standards

Association Committee to establish a Canadian Code for Head and Eye Protection.

REVIEW OF FOREIGN RESEARCH ORGANIZATIONS

At the request of the Acting-President of the National Research Council a member of the staff of the Division of Chemistry undertook, in 1942 and 1943, to prepare a review of the organization of research in the major industrial countries, with particular reference to plans for post-war research. Included in the literature survey were Australia, France, Germany, Great Britain, Japan, the U.S.S.R., and the United States.

For each country an attempt was made to give enough of the historical background to permit one to understand how the present system had arisen. The principal research organizations were listed, their fields of activities and available funds were indicated, the relation between them (if any) was set out, and references to more complete information were cited. In the case of the United States there were listed the chief research foundations and trusts, the many institutes and associations having laboratory research as a major activity, and the larger private industrial research laboratories; a brief review of the work of several of these was included. Special attention was called to the United States Government's research studies made by the National Resources Planning Board. A review was included of the Kilgore bills which, though not ultimately passed, nevertheless reflect the thinking of many Americans.

Letters were also written to about a dozen prominent research directors in the United States, asking their views on such questions as (i) the relation between research sponsored by governments and that of the universities and privately endowed organizations, (ii) means of promoting cooperation between research organizations, (iii) the degree to which planning for research should be carried out on a national scale, (iv) the advisability of centralizing government research, or otherwise, (v) the probable trend of research after the war, and (vi) how peacetime research should be related to national defence. The replies received differed widely from one another, as might be expected, but were definitely stimulating and helpful.

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C. ORGANIC CHEMISTRY

EXPERIMENTAL EXPLOSIVES ESTABLISHMENT

History of the Establishment

The evolution of the High Explosives Sub-Committee on the Advisory Committee of Industrial Chemists has already been outlined in the Historical Introduction. Under its sponsorship, research on explosives was started in several universities. Out of this research came several new explosives or promise of new methods of manufacturing existing explosives. No matter how promising laboratory tests may be, it is absolutely essential, in the case of a new explosive, to prepare several thousand pounds for field tests or, in the case of a new process, to carry it through the pilot plant stage in order to discover and solve manufacturing problems.

The High Explosives Sub-Committee brought the need for such an explosives pilot plant to the attention of the National Research Council, who, in the spring of 1942, recommended to the newly formed Army Technical Development Board that such facilities be provided. The Board thereupon allocated one hundred thousand dollars to the project.

About this time, it was learned that the Department of Munitions and Supply were considering the construction of a propellant development building and laboratory, but had not passed beyond the initial layout plans. It occurred to some of those interested that, if this plant were also needed, it might advantageously be combined with the experimental high explosives building. After thorough investigation, this suggestion was approved by the Army Technical Development Board.

It was the opinion of those interested in the plan that the project should be a permanent one and that, for this reason, the establishment should not be situated beside a war-time plant, which might be dismantled after the war. It was also thought that it should be wholly under government control and, therefore, that it should not be placed on property of a private explosives manufacturer.

Visits were made to similar establishments in the United States and it was found, from their experience, that explosives development laboratories should be situated close to a weapon proving range. A suitable site was therefore sought, and found, adjacent to the Artillery Proof Establishment at Valcartier. Land was made available there in February 1943.

In the meantime, the expanding program in explosives development and research had made reorganization necessary and towards the end of 1942, the National Research Council had set up an Associate Committee on Explosives to coordinate all work on explosives development, to supervise the extra mural research on explosives, and to supervise the new Experimental Explosives Establishment. In the meantime, too, preliminary plans and cost estimates had been prepared by the staff of the Division of Chemistry and funds had been made available through the Army Technical Development Board in January 1943. At the request of the Canadian Government, the United Kingdom provided an expert research director from Woolwich Arsenal to guide the establishment through its initial stages. After his arrival in April, plans went ahead more rapidly; construction was started in late summer, 1943, and continued throughout most of 1944.

While construction at Valcartier was in progress, the nucleus of a technical staff for the new establishment was collected at the National Research Council in Ottawa. Since it was virtually impossible to obtain men with training in the field of explosives manufacture, because of the existing demand for such men by industrial firms, an effort was made to familiarize the initial staff with explosives practice by sending them for various periods of time to places such as Nobel and DeSalaberry. In this connection, very excellent cooperation was received from Defence Industries Limited and from the various plant managers.

Coincident with this training period, the staff prepared plans and blueprints for the pilot plant manufacture of the newer high explosives. With the assistance of D.I.L. engineers, plans were prepared for the installation of the solvent propellant presses in the nearly completed building. Three surplus presses from Defence Industries Limited, Nobel, were secured and delivered to the site. Laboratory furnishings, such as work benches, fumehoods, equipment, and chemicals, were planned and ordered for the laboratories. Plant equipment, consisting of motors, mixers, controllers, and countless other items, were placed on order and eventually secured from numerous sources.

The staff likewise participated in experimental work insofar as facilities and conditions at the National Research Council would permit. The first experimental work undertaken by the staff, while in Ottawa, was the development of a climatic storage cabinet which could be used for the testing of ammunition and explosives under conditions of heat and humidity likely to be encountered anywhere in the world. A standard type of construction and control mechanism was sought, which would

be flexible, automatic and reliable. Two experimental units were constructed, and one in particular was subjected to a six months' test period, which illustrated its inherent soundness of design. Particular attention was devoted to developing a simple cycle control mechanism which would permit the automatic alteration of the test conditions between predetermined limits. Considerable success was achieved in this direction and very satisfactory units are now in operation. Apart from experimental work, the group took an active interest in all published papers and two of the members compiled a very extensive card index of all explosives documents and reports.

Late in 1944 members of the group began to spend more and more time at Valcartier and the job of fitting out the various buildings was commenced. By the spring of 1945 the three presses were in place and the laboratories were furnished. As soon as the rolling house was vacated by the contractor, the rolls and auxiliary equipment, including an automatic deluge system, were installed. The climatic test hut was furnished with three test cabinets and all put in operation. Control and recording gear for five more cabinets were placed in position.

By VJ-Day numerous dummy batches of propellant had been worked up in the kneaders and extruded in the presses to check the operation of the equipment, Some minor changes had been found necessary. A few batches of "live" propellant had been processed and further batches were going through from day to day.

The Experimental Explosives Establishment was suggested by the High Explosives Sub-committee, was financed by the Army Technical Development Board, was administered at first by the National Research Council, and finally was turned over to the Army - on September 1, 1945, it became Wing C of the Canadian Armament Research and Development Establishment. Although it was not, strictly speaking, an activity of the Division of Chemistry, the divisional staff made large and important contributions in the formulation of plans for it and the Division of Chemistry gave it a home until the premises at Valcartier were ready for occupancy.

ETHYLENE OXIDE

In addition to its use as a raw material in a wide range of organic syntheses, ethylene oxide shows considerable promise as a fumigant, bactericide, and insecticide. In all

these fields the chief obstacle to a much wider use of ethylene oxide is the present restricted available supply and cost. At the present time no ethylene oxide is produced in Canada, while Canadian imports of ethylene glycol (the most widely used derivative of ethylene oxide) for antifreeze purposes alone amounted to more than a million gallons a year in 1939.

An investigation of the vapour phase oxidation of ethylene to ethylene oxide with air over a silver catalyst, begun in the Division of Chemistry in 1938, has led to the development of an economical process for the production of ethylene oxide and ethylene glycol from readily available hydrocarbon fractions such as oil refinery gases, without requiring the isolation of the ethylene. The critical problem in the oxidation of ethylene to ethylene oxide is temperature control of the catalyst surface in order to prevent complete oxidation to carbon dioxide and water. When the ethylene is diluted with methane and ethane another problem is presented. Methane and ethane are themselves oxidized to carbon dioxide and water, under the optimum conditions for the oxidation of ethylene to ethylene oxide, and also promote the complete oxidation of ethylene. In the process developed at the National Research Council, close temperature control of the catalyst surface together with the addition of controlled amounts of ethylene dichloride to the reactor feed have suppressed the oxidation of the paraffins and have made possible the economic production of the ethylene oxide by the air oxidation of ethylene in hydrocarbon feeds containing as low as 10% ethylene.

The chief new features of the process are:-

1. The ability to utilize ethylene in a mixed hydrocarbon feed containing 10 to 90% paraffins. The hydrocarbon feed may also contain small amounts of hydrogen, carbon monoxide or acetylene.

2. A new silver catalyst prepared, under special conditions, by the thermal decomposition of silver oxalate in the presence of calcium oxalate, and to which is subsequently added a controlled amount of stannous oxide as a promoter.

3. A new, fin-type, catalyst support, affording close temperature control over all the catalyst surface.

4. The addition to the reactants of controlled amounts of ethylene dichloride, less than 0.005% by volume of the total gas entering the reaction tube, for the specific purpose of suppressing both the oxidation of the paraffins and their tendency to promote the complete oxidation

of ethylene. The ethylene dichloride rate required to give optimum conversion to ethylene oxide has been found to be a linear function of the amount of methane plus ethane in the feed, and to be independent of the amount of ethylene present.

Estimates indicate that the raw material costs, depending on the mode of operation, will be 0.8 to 2.3 cents per pound of glycol when the process operates on methane-ethane-ethylene mixtures obtained by processing depropanizer residue gas, or its equivalent, in standard refinery equipment.

Sufficient data is not yet available to estimate the operating costs, but it may be pointed out that there are no fuel costs involved in the oxidation step, since the reaction is strongly exothermic.

The process has been successfully tested on a laboratory scale at a Canadian refinery over a period of seven months. Plans have been prepared for a pilot plant in Turner Valley, Alberta.

ETHANOLAMINES

In 1943, following preliminary experiments in the National Research Laboratories, a pilot plant for the production of ethanolamines was erected and put into operation at the plant of the Canadian Aniline and Extract Company in Hamilton, Ontario. This work was undertaken to supply diethanolamine for research on explosives at the University of Toronto and for pilot scale production of new explosives at Valleyfield.

The reaction between ethylene oxide and aqueous ammonia gives a mixture of mono-, di-, and triethanolamines, the proportion of each constitutent depending upon the ratio of ammonia to ethylene oxide. Since the most valuable products in this case were the mono- and diethanolamines, it was necessary to use as large an excess of aqueous ammonia as was commensurate with reasonable production, in order to keep the triethanolamine formation to a minimum. The process as finally installed can be divided into two main parts - (i) reaction of ammonia and ethylene oxide followed by recovery of the excess ammonia, and (ii) fractionation of the resulting aqueous solution to give mono-, di-, and triethanolamine.

Reaction and Ammonia Recovery

A jacketed stainless steel autoclave, available in the plant, was charged with aqueous ammonia (28%) and then warmed to 30-35°C. to ensure rapid reaction of the ethylene oxide. Ethylene oxide was vaporized and passed into the solution. During the addition, which required about two hours, the reaction mixture was vigorously stirred and cooled.

On completion of the reaction, the excess ammonia was recovered by slowly heating the contents of the autoclave. The ammonia was thus driven out and was absorbed in a simple recovery unit built from drums and piping available in the plant. Providing the autoclave with a reflux head prevented the distillation of mych water during this period. The recovery was about 90 to 95% efficient and the ammonia obtained suitable for re-use (25 to 28%).

Following the "blowing" of the ammonia, the autoclave was cooled and drained to give about 150 lbs. of mixed ethanolamines in about 500 lbs. of water. Each batch required approximately six hours to prepare.

Fractionation of the Ethanolamines

The fractionating equipment for separating the ethanolamines consisted of two continuous stripping columns and one batch still. All these units, together with their receivers, condensers, and other auxiliaries, were built in Hamilton from readily available materials. A second batch still of small capacity was also used at times to purify diethanolamine for shipment to the University of Toronto. All these stills were operated under vacuum. In the first stripping column the water content of the ethanolamines was reduced to about 5% without appreciable loss of monoethanolamine. In the second stripping column 65 to 75% of the monoethanolamine was removed; the separation was not complete because only 70 p.s.i. steam and 27 inches of vacuum were available. The final separation was effected in the batch still.

The diethanolamine obtained was almost colourless and had a melting point of 24 to 25°C., but before shipment it was redistilled to bring it up to a melting point of 26 to 27°C. The monoethanolamine obtained as by-product was converted to di-, and triethanolamines by further treatment with ethylene oxide.

Triethanolamine was obtained as the residue from the batch still and hence was of poor quality, marketable only to a slight extent in spite of the severe shortage of this material at the time. It could, however, have been improved by installing equipment to permit its distillation under high vacuum.

This process was operated for several weeks on a 24-hour day, 6-day week basis. The daily output was of the order of 270 lbs. of mono-, 200 lbs. of di-, and 140 lbs. of triethanolamine. In all, some six tons of diethanolamine were supplied to Defence Industries Limited.

FREE RADICAL REACTIONS

An investigation of the mechanism of chemical reactions usually reveals that what we normally write as a simple reaction is actually a series of very rapid reactions involving free atoms or free radicals. Such processes are commonly referred to as elementary reactions. A knowledge of such elementary reactions is of the utmost importance in obtaining an understanding of ordinary chemical processes, particularly since these elementary reactions are in themselves complete entities and may appear as steps in numerous complete reactions.

Numerous elementary reactions of importance in the field of organic chemistry have been investigated by the Physical Chemistry Section in accordance with the policy of continuing fundamental research during the period of hostilities. Fundamental data of this type are of value to chemists working in such fields of applied chemistry as petroleum and synthetic rubber technology.

RUBBER

The wartime activities of the Rubber Laboratory involved a great number of rubber products; the research, compounding, and testing problems undertaken were so varied as to defy complete description.

The Rubber Laboratory cooperated in the rubber conservation program instituted by the Rubber Controller by attendance on committees, by giving technical advice, and by undertaking research to find means of replacing natural rubber in various products. There was cooperation with the Polymer Corporation Ltd., Sarnia, and the War Production

Board, Washington, in work on government manufactured synthetic rubber. Many research problems were undertaken for the Armed Services, for the Department of Munitions and Supply, for the Inspection Board of the United Kingdom and Canada, for the British Admiralty Technical Mission, and for companies in Canada and elsewhere.

Acceptance tests of rubber products were carried out for the Inspection Board and for the Armed Services according to a specification, when available. Many new specifications were prepared for special products, and recommendations were made for revision of current specifications as shortages in materials occurred, or when the product was required to function under conditions not previously encountered.

Numerous vulcanizing jobs were carried out for other National Research Council Divisions and for the Armed Services, consisting mostly of small, sometimes intricate, moulded parts for experimental apparatus. In the majority of cases this entailed designing special moulds, and often required the development of new rubber compounds to meet the varied and exceptional conditions of service.

Synthetic Rubber

The substitution of synthetics for natural rubber brought many research problems. As a preliminary to compounding research, a program to calibrate the test equipment and procedures of all the contributing laboratories was undertaken. The Rubber Laboratory cooperated in this calibration, which was coordinated by the Polymer Corporation Synthetic Rubber Technical Advisory Committee. The results obtained were about midway between the extremes found in the results of the various laboratories. This was felt to be an indication of well calibrated equipment. Various compounding studies were made for this committee, some of which are summarized below.

An early problem was the slower rate of vulcanization of GR-S synthetic rubber as compared with natural rubber; this, if not overcome, would tend to slow down production. As part of this investigation the Rubber Laboratory was requested to determine the effect of di-isopropyl xanthogen disulphide accelerator in producing rapid cures of pure gum GR-S stocks at room temperature. It was found that some improvement could be attained through the use of a relatively low quantity of sulphur in the mix with one part of piperidine and one part of di-isopropyl xanthogen disulphide.

An attempt was made to improve the characteristic, low tensile strength of pure-gum, GR-S compounds by the use of selenium as the vulcanizing agent instead of sulphur in this respect.

The possibility of improving the characteristically low, pure-gum, tensile strength of GR-S by using special accelerators of vulcanization was also investigated. The accelerators tested were BJF, Tuads, Selenac, and Tetrone A. None of these accelerators caused a significant improvement in the tensile strength.

One of the more extensive projects in connection with the use of GR-S rubber was the investigation of various materials added as softeners which might reduce the toughness and improve the tackiness of the rubber, and thus allow it to be processed as easily, and as rapidly, as natural rubber. The materials added were natural rubber, balata, gutta percha, Vistanex, Plastogel, and p-nitrosodimethylaniline. In some cases the process-ability was slightly improved, but none of these materials was found to produce a tackiness in the synthetic rubber compound approaching that produced in a similar natural rubber compound. Various oxidizing agents and heat treatments were also investigated as to their effect on the tackiness of GR-S. Some improvement was found for the rubber itself but in all cases the incorporation of carbon black during compounding largely destroyed any tackiness that had been produced by the reagents tried.

In cooperation with the United States War Production Board, studies were made of the effect of storage on GR-S in the raw state. As the synthetic rubber production increased, it became necessary to determine the conditions under which the rubber might be stored for a considerable period of time without deterioration, so that stock piles might be built up. GR-S rubber was stored for one year at four temperatures, 30°, 80°, 120°, and 160°F., and tested every three months to determine any change in the original properties. It was found that storage for periods up to 12 months at 30°F. and 80°F. had no deleterious effect. Storage at 160°F. adversely affected process-ability as well as the physical properties of the vulcanized rubber. The adverse effect on processability of storage at 120°F. for 12 months was relatively slight. It was, therefore, concluded that GR-S could be successfully stored for a considerable period of time at all temperatures encountered in the United States.

Cooperative United States-Canadian investigations were made of butyl rubber carbon black stocks. Butyl rubber has a slower rate of cure than natural rubber, and various accelerators and accelerator combinations were tested in an effort to increase the rate of vulcanization. It was found that 1% to 1.5% of Selenac gives satisfactory accelerations, particularly the higher amount. It was found possible to produce butyl rubber cements that would slowly vulcanize at room temperature through the addition of relatively large amounts of lead peroxide and GMF accelerator.

At the request of the Department of Munitions and Supply, a literature survey was made regarding the possibility of producing synthetic rubber from alcohol. It was found that a plant for producing synthetic rubber from alcohol can be built more quickly than a plant using petroleum as a raw material. The production of rubber from alcohol, however, is more costly than from petroleum.

In cooperation with the Division of Biology, tests were made of Kok-Saghyz and milkweed rubbers as substitutes for natural rubber. Milkweed rubber containing some natural resin was found to give at best a tensile strength of only 1500 lbs. per sq. in. in a tread type compound. It would thus not be particularly promising when used alone. In admixture with GR-S it acted as a softener and gave some improvement in processability. A rubber free from resin would be a satisfactory rubber substitute but the cost of purification would be prohibitive. Kok-Saghyz rubber, grown in Canada by the Experimental Farm, proved to be almost equal in quality to natural rubber although its cost is high. Its molecular weight appears to be somewhat lower than that of natural rubber.

Studies were made of blends of natural and GR-S rubber, and of fillers in natural and synthetic rubber, in cooperation with the National Research Council Radiology Laboratory. Many fillers in rubber were observed to give characteristic X-ray diagrams. Stretched natural rubber gives a pronounced characteristic X-ray interference diagram, whereas, comparatively speaking, GR-S gives none. Mixtures of natural rubber and GR-S prepared in the Rubber Laboratory were observed to give X-ray interference diagrams, under certain circumstances, even when only ten percent of natural rubber was present. The X-ray examination thus gives an idea of the degree of dispersion of natural rubber in GR-S and it would appear that the mixing of the two rubbers does not produce an absolutely homogeneous product but leaves small units of unchanged natural rubber dispersed in the GR-S.

Laboratory Test Methods

Laboratory test data were obtained in conjunction with road tests of synthetic rubber recaps on Army truck tires, and a valuable correlation was thus made available. The road performance was found to be closely related to the modulus of the rubber. The specification for camelback, prepared as a result of this work, called for an increased modulus. The modulus and other requirements in this specification tended to make the manufacturers' products more uniform, and a general improvement in quality resulted.

An improved correlation of laboratory abrasion resistance tests with road wear was obtained by modifying the standard test by extracting the test specimen with an ethanol-toluene mixture before abrading it. It was found that softener from the rubber was coating the abrasive in the abrasion machine and vitiating the results. The extraction removes this softener and enables a good correlation with road wear to be obtained.

A method was developed for the identification of rubber in sludge in aircraft lubrication systems. Rubber dissolved from oil hose can thus be distinguished from other types of sludge.

Rubber Applications

Acetylene black was studied as a reinforcing agent for GR-S in tire treads. Some advantages for this type of filler were indicated because of the lower hysteresis and consequent lower heat development resulting from its use. Static is readily discharged in an aeroplane tire containing acetylene black.

A study of lime hydrate, a by-product of acetylene manufacture, showed it to be a useful filler in insulating compounds.

In cooperation with the National Research Council Electrical Engineering Laboratory and the Directorate of Scientific Research and Development, Naval Service, the physical and electrical properties of insulation made from three different types of raw GR-S were investigated. The GR-S insulation compounds were prepared by a number of different wire and cable manufacturers for examination by the National Research Council. The electrical stability of the rubber insulant was found to be related principally to the water soluble ash content of the raw GR-S. Sarnia GR-S of

exceptionally low ash content has been prepared for evaluation in this connection.

A specification for rubber glands, or packings, used in hydraulic mechanisms in aircraft, was prepared for the R.C.A.F. Such glands must be resistant to the various oils and fluids used in aircraft, and also flexible at low temperatures encountered at high altitudes. Studies were made of the best synthetic rubber compositions for this purpose, and useful knowledge was obtained. A standard rubber gland material was prepared and, in cooperation with the Gas and Oil Laboratory, was tested in various aircraft hydraulic fluids and oils. This work resulted in specifications for fluid and oil that would function satisfactorily and at the same time not be injurious to the rubber. Numerous tests of glands prepared by the various rubber manufacturers were made against specification requirements for the R.C.A.F.

In cooperation with the Royal Canadian Air Force numerous types of rubber hose for conveying oil, fuel, coolant, etc., were tested with a view to producing a single general spedification which would cover all types of service and reduce the number of production items necessary in aircraft.

In cooperation with the R.C.E.M.E. a number of types of rubber repair patches for synthetic rubber inner tubes were evaluated. Amongst other physical characteristics, the flexing resistance of the patch was determined. Methods of application of a satisfactory repair were investigated.

A number of manufactured products for use as anti-skid deck paints were investigated for the Naval Service. A specification for a composition containing a plastic and a coarse mineral aggregate was prepared. Acceptance tests were carried out on a considerable number of lots of this type of material.

A number of experimental hard and soft rubber parts were prepared for non-metallic land mines. These were designed by the Directorate of Engineer Development to avoid detection by electrical means. In cooperation with this same Army Branch, pressure tests were carried out on 1000-ft. lengths of tubing to hold explosive. Such tubing, when thrown out, filled with explosive, and exploded, could be used as a means of neutralizing land mines.

Rubber belting for use in the track assembly of snowmobiles was found to shrink excessively in the longitudinal direction when wetted. Investigation showed that

the cause of this shrinkage was the tension placed in the belting during manufacture. This shrinkage, which was irregular and unpredictable, created difficulties in the assembly and maintenance of the snowmobile. Knowing the cause of the shrinkage it was possible to suggest means of overcoming the difficulty.

An investigation of the creep or set occurring in snatch plugs, used in wireless sets, was carried out for the Canadian Signals Research and Development Establishment. These are electrical plugs containing five terminals embedded in rubber, the rubber being used to add resilience and to make the plug fit tightly into its socket with some elastic deformation of the rubber. When the change to synthetic rubber was made, the rubber was found to have undue creep, or set, so that the plug after some time in the installation would come out of its socket too readily. An accelerated, compression-set test and a test to determine the force required to pull out the plug were devised, and suggestions were made as to the best type of natural or synthetic rubber compound to use in order to overcome the difficulty.

The Rubber Laboratory cooperated in road tests of bogie wheels for Ram tanks carried out by the Army Engineering Design Branch of the Department of Munitions and Supply, and by the Inspection Board. It was found that bogie wheels in which the solid rubber tires had the highest resilience were superior. This is because resilient rubber has relatively low hysteresis and consequently develops less heat on the road. For certain positions on the tank it was found not possible to use synthetic rubber bogie wheels because of the somewhat lower resilience than natural rubber and consequent higher development of heat.

RUBBER GOODS

Development work on various items of protective equipment of which rubber was a constituent was carried out in various laboratories of the Division.

It was found that the considerable drop in the bursting strength of fire hose caused by mildew can be prevented by treating the hose with zinc naphthenate. A test was developed to evaluate the shock absorbing properties of various types of sponge rubber for crash helemts. Rubber socks for field experiments were prepared from latex by the Rubber Laboratory. A portable tent flooring for cold weather use was urgently required, and a suitable material

was recommended in the form of fabric equal in weight to that used in ground sheets, but coated on both sides with an all-reclaim rubber compound.

Technical assistance was given in commencing the manufacture of gas masks in Canada, and all manufacturing problems were solved before the war started. Skin irritation may be produced by contact with rubber in gas masks or other equipment; the Rubber Laboratory prepared rubber compounds for toxicity tests by the Chemical Warfare Laboratories in order to detect the irritating ingredient.

In an attempt to find a substitute for natural cellular rubber in life jackets and in other buoyant products, many different materials were examined. During the investigation a thin-walled, air-filled Neoprene bubble was developed in cooperation with the Viceroy Manufacturing Company. These bubbles, tightly packed together, were shown to be of use as a buoyant material.

A number of designs for automatically inflatable life jackets were also developed. These jackets were made of a waterproof, air-tight, rubber and fabric construction, and consisted of a number of compartments sealed from one another and inflated from carbon dioxide bullets by pulling a cord. Successful trials were made by the Naval Service.

A considerable amount of work was performed in testing rubber coated and plastic coated fabrics for use in ground sheets, raincoats, ponchos, muzzle covers, hospital sheeting, etc. Numerous tentative specifications were written to cover the production and testing of such materials.

PLASTICS

The National Research Council was called upon to give advice to the Services regarding the selection of the most suitable type of plastic for innumerable items of equipment. This work, involving extensive physical testing of plastics and of plastics products, although unspectacular, was an important contribution in the evolution of efficient service equipment. Among the items investigated were: transparent acrylics for aircraft enclosures; battery box materials; mess trays; combs; shaving kits; razors; radio antennae insulators; caps for water, gasoline, and oil containers; service buttons; badges, and web strap tips; and parts of hand grenades and smoke boxes. In each case the testing ensured that a plastic material of adequate properties was used.

After extensive testing, formulations of melamine-α-cellulose were adopted by the Navy for plastic mess gear. Bayonet scabbards of various plastics were tested; a design using injection-moulded cellulose acetate-butyrate, was shown to have adequate resistance to extremes of temperature and to have toughness much superior to thermosetting plastics. Some testing was carried out in connection with plastic motorcyclists' crash helmets and modifications in the specification were suggested; this helmet of sisalphenolic material appears to have possibilities with regard to the development of a better miner's helmet. Testing, carried out to determine the possible usefulness of thermoplastic tubing for fluids in aircraft, showed that low temperature embrittlement is a limiting factor in most cases; this fact, together with solvent resistance considerations, suggested only limited usefulness in view of the small weight advantage compared to aluminum.

Considerable assistance was given the Army regarding the correct adhesive to use in the manufacture of bulletproof glass that would withstand extremes of temperature; ethyl cellulose was eventhally adopted. At the request of the Army, an identification disc composed of asbestos paper and phenol-formaldehyde resin was fabricated. This disc was virtually indestructible by fire and had good water resistance. It was never adopted, however, due to commercial production difficulties.

A laboratory test, which was later adopted as standard, was developed to determine the amount of acidity developed by urea resin glues during the curing cycle. Some work was done attempting to use dimethyl urea as an adhesive for wood wastes. Considerable assistance was given to the industry in the manufacture of casein.

A large number of composite-type periscope prisms had been produced by the industry when bubbles began to appear in the field of vision. It was demonstrated that the defect occurred in the laminating adhesive used to cement the glass protective plates to the acrylic body of the prism. The effect, probably due to loss of plasticizer, might have been avoided by adequate preliminary testing.

Considerable testing was done in connection with the plastic prism holders for the periscopes of tanks. Of box-like construction, these holders must have certain specific properties, and plastic construction was adopted in Canada early in the war, the plastic being chosen to fulfil the requirement that the holder must shatter completely so as to be readily replaceable and not jam the periscope when struck

by enemy fire. The holder must also be of vapour proof construction so that there will be no opportunity for internal fogging of reflecting surfaces. A long series of tests was carried out to check this last point, and tests were also made to compare various methods of silvering. Practical firing tests were arranged which demonstrated the superiority of the plastics type holder over an aluminum alloy type in that it shattered more satisfactorily when struck by rifle fire.

A problem of quite considerable interest was that of waterproofing military maps so that they could be given operational markings with ordinary pencil (colored or otherwise), and so that such markings could be erased, as troop positions changed, without injury to the map. Previous solutions only partly solved the difficulties, that is, gave waterproof maps from which marks were erased only after damaging the map, required special pencils, or required the use of sheets of plastic which could only be, at best, insecurely attached to the map. The answer developed was that of lacquering the maps with a heavy coating of plasticized polyvinyl chloride - acetate, including in the lacquer a pigment, such as barytes, of refractive index substantially the same as that of the plastic and of suitable particle size (3 to 25 microns). Such a coating is transparent, readily takes markings in pencil, allows repeated erasure without damage to the map, and provides excellent waterproofing. The procedure is moderately expensive but may also have peacetime uses where similar properties are desired. After the principles involved had been established, commercial development was turned over to the Aulcraft Paint and Varnish Company who produced a very satisfactory commercial product.

In the manufacture of large, wooden, laminated timbers a considerable amount of time is lost due to the fact that the member must remain in clamps for a considerable period after the flue is applied. This time may vary from eight to twenty-four hours. High frequency electric heating has been used to hasten the process, but, while this is quite satisfactory, the initial cost of equipment is high. In an effort to confine the heating to the glue line, and to eliminate high frequency equipment, considerable experimental work was done using carbon black mixed with the glue to make it electrically conducting and then applying ordinary 60 cycle alternating current to this glue line. To make the glue line a more uniform conductor the method was later modified by applying the carbon black to an open mesh fabric such as mosquito netting. This method showed some promise for commercial use.

Prior to the establishment of the structure laboratory considerable work was done on the moulding of plywood. This was given a great impetus by the announcement of the Vidal bag-moulding process. Models of a tail assembly, a gasoline tank, and an engine nacelle were made. After the supply of natural bamboo was cut off by Japan's entry into the war, attempts were made to produce cylinders from plywood which had strength properties approaching those of bamboo. It was found, when birch veneer was coated with urea-formaldehyde cold-setting adhesive and wrapped about a mandrel, that after the resin cured compressive strength equal to that of natural bamboo could be obtained. Difficulty was found, however, in maintaining this strength when two pieces were spliced. The work was initially done at the request of the British Admiralty, who use bamboo in quantity in the manufacture of anti-aircraft kites.

LUBRICATING GREASE - FUNDAMENTAL STUDIES

Late in 1938, under the sponsorship of Imperial Oil Ltd., an investigation was started of the more fundamental properties of lubricating grease, and this project was continued during the early years of the war. The work was concerned mainly with the physical properties of soaps in organic media, although some attention was given to practical problems. The results of this work have been disclosed in publications listed at the end of this Section.

ALKALOIDS

In addition to direct war research, the Organic Chemistry Laboratory continued its investigations in the field of alkaloids. During the period 1940-41 two alkaloids were synthesized, the chemical structures of two others were completely unravelled, and that of another partially so. Furthermore, close to twenty new alkaloids have been discovered and reported. In connection with the determination of the structure of new alkaloids, the bast is usually degraded, and it often gives rise to a methyl- and dimethyl-quinoline. Since only about 40% of the possible methyl- and dimethyl-quinolines were known, it was seldom possible to identify such a degradation product. Such a case was encountered, and the synthesis and characterization of all these quinolines was therefore undertaken and completed.

During the period 1932-42 a systematic study was made of the alkaloids of the Fumariaceous plants. This work was important both chemically and botanically because it constituted the first systematic investigation of the alkaloids occurring in the various species of one genus of plants.

Work started before the war on the synthetic preparation of plant hormones was also completed.

MICRO ORGANIC ANALYSIS

In research work in the field of organic chemistry the amounts of some substances isolated are often very small. As these substances are usually obtained in quantities insufficient for analysis by ordinary methods, they must be analyzed by a special system known as Pregl's micro-analytical methods. Before the war, all such substances obtained in these laboratories were sent to Germany to be micro-analysed. With the outbreak of hostilities, it became imperative to have our own facilities, so a micro-analytical laboratory was established, and, to familiarize him with the technique involved, one man was sent to New York University for a summer course in micro-analysis. The services of the National Research Council Micro-analytical Laboratory are available to research departments in those Canadian Universities not equipped for such work.

STARCH

Beginning in 1938, considerable assistance was given to a potato starch plant at Grand Falls, N.B., in adapting to their process American made machinery - chiefly continuous centrifuges. Equipment of this type was formerly supplied by Germany. Considerable assistance was subsequently rendered in checking the quality of the product.

Investigations of the physical and chemical properties of starch, which had been in progress for several years were terminated during the early years of the war. Interesting relations have been noted in the falling off of the viscosity of starch pastes with intensive drying of the granules prior to pasting. Thermal degradation of the starch granules, as is practiced in the preparation of dextrin, has yielded some very valuable information on the probable mechanism of dextrin formation. This work was extended to some of the lower molecular weight hydrocarbons, and the rate of thermal decomposition, as obtained by the amount of gaseous products formed, was followed for dextrose, maltose, cellobiose, and sucrose. Some work on the behaviour of starch fractions on the film balance was also started.

PEAT

Peat, in its various forms, is one of Canada's most abundant materials. With the interruption of the European supply during the war, Canadian peat was used in a small way for soil conditioning, for stable litter, for packing material, and, for a brief period, it was exported to one of the larger United States magnesium plants. In Europe, its chief use has been for fuel in areas where coal and wood are not readily available and labour is cheap. The only really large scale fuel development seems to have been in the U.S.S.R. Efforts to develop its use as a fuel in Canada have not yet proved economical.

The two great difficulties which have retarded its development are the high moisture content (up to 90%), and the small scale of operations which has kept up its cost of recovery. It appears that any major industrial use of peat must be on a scale sufficiently large to warrant low-cost mechanical recovery, and must be of such a nature that the high moisture content is not a disadvantage. For use as a fuel, the scale of operations would be large enough to warrant the use of mechanical handling equipment; but the drying difficulties were never surmounted - artificial drying was too costly and natural air drying too slow. This, then, leaves the utilization of its chemical constituents without predrying as the only probable means of exploiting this abundant natural resource. It was with this in mind that a study of peat was undertaken in the Division of Chemistry.

Peat is a complex mixture of various materials, including waxes, alpha-cellulose, hemicelluloses, lignin, humic acids, and ash. The proportions vary considerably between different deposits and between different depths in the same deposit. In commencing this work it was thought that if some segregation of these various components could be effected, it might be possible to isolate materials of higher unit value than the peat mixture. As it was desired to accomplish this objective without passing through the uneconomic drying operation, treatment with an aqueous solution was indicated. Two such treatments were known which might be applicable, namely, treatment with an alkaline solution to extract humic acids, and dilute acid hydrolysis to effect some degree of saccharification as, for example, in the Schoeller process for the treatment of weed waste.

Alkaline Treatment

Humic acid extraction was tried first, using dilute caustic soda solution; this worked very well except that the subsequent filtration was slow and difficult. Ammonium hydroxide was tried next, as there is a large production of ammonia available in Canada. This worked much better and, in the course of this work, it was noted that the solid residue from the extraction dried quite rapidly and became considerably tougher than raw dry peat.

This observation suggested that, if it were possible to dry peat rapidly, it might be economically possible to work on it with other than aqueous solutions. Several repetitions confirmed the original observation, and it was found that peat would dry, though slowly, at temperatures almost down to the freezing point. Treatment at an elevated temperature, on the other hand, weakened the tough structure.

Since a use for the humic acid extract had not yet been developed, drying experiments were performed on alkali-treated peat without separating the humic acid. Again the mass was found to air-dry quite readily. Other alkalis were tried, but sodium carbonate was the only one found which could compare with ammonia in effectiveness. The effect of adding other chemicals was investigated and ferrous sulphate was found to add strength to the dried product and to prevent shrinkage cracks.

The final preferred process was to add to wet peat, as recovered from the bog, ammonia or sodium carbonate solution until the peat slurry was neutral or very slightly alkaline, then to add about 0.5% (based on dry weight of peat) ferrous sulphate in solution. When the resulting pasty mass was formed into blocks about two inches square, it hardened and dried appreciably in air within three days - or within half that time if air was blown over the surface by a fan. The product then contained about 10% moisture. After a further ten days, drying was as complete as practicable. The product obtained had the following properties: density, about 1.0; fuel value, 9000 B.T.U. per lb.; ash content, 5 to 6%; and moisture content, about 4%. This product is quite resistant to handling but disintegrates if exposed to rain for any length of time. It should make a very satisfactory fuel, except that to produce one ton of dry material, about ten tons of wet peat have to be handled. In areas where other fuel costs are high, it might be produced cooperatively, and it is certainly much superior to the usual hand-cut peat blocks.

Acid Treatment

Experiments were also carried out on the hydrolysis of wet peat with dilute acid, although apparatus for high pressure treatment was not available. It was found that a substantial percentage of reducing sugars could be obtained, although the amount was less than from pressure treated wood. However, large scale harvesting of peat should be obtainable in many parts of Canada, whereas wood waste is not so widely available in the tonnage required for alcohol production. After concentration of the sugar-containing liquor, both alcohol and butylene glycol were obtained qualitatively by enzyme action but no actual yield figures were obtained.

MISCELLANEOUS ORGANIC INVESTIGATIONS

Low Temperature Grease

At the request of the Meteorological Services, sources were investigated for a grease which would give adequate lubrication at temperatures down to -60°F. or -70°F. for use with radio-sonde equipment. The pre-war lubricant used was a French oil prepared from porpoise fins and discussion with all oil companies had failed to find anything suitable to replace their minute and dwindling supply.

After a number of other sources failed to provide suitable lubricants, it was decided to test the use of fractions refined from ordinary fuel oil. This is a residue from the thermal cracking of petroleum and it was hoped the heat treatment might have destroyed those molecular types resulting in poor viscosity indices and high freezing constituents. In general, the paraffinic constituents are those with high freezing points, and it was hoped that this class in particular would largely be destroyed by the cracking, since the aromatic constituents responsible for poor viscosity indices can be removed chemically.

This was found to be entirely correct and from suitable fractions remarkably good oil can be secured. As first taken from the fuel oil by distillation, the material is naturally very crude, on standing in the air a tremendous amount of gum forms, oxidation tests are very bad and, owing to a high aromatic content, its low temperature properties are useless. However, sulphuric acid treatment removes large amounts of aromatic constituents and heating to 250° with sodium metal removes gum forming and other undesirable constituents. After acid and sodium treatment, the oil is

clarified with bentonite and distilled.

The product is an entirely stable pale yellow oil of slight, agreeable odor, forms no detectable gum even after several years, and remains very fluid down to remarkably low temperatures. Tests by the Meteorological Services showed that their very delicate mechanisms remained properly lubricated down to -80°F.

In order to retain this light oil in small bearings, it is made up in the form of a thin grease using lithium stearate and a specially developed selenium compound is also added to produce low starting friction.

This grease was also tested in anti-aircraft shell timers at the request of the Inspection Board and at -89°F. the mechanism was delayed by only a half second. A small supply of this grease was sent to the Fuse Division of the Inspection Board, together with all details of its production; it is understood that further work was undertaken elsewhere.

The Kinetics of Polymerization Reactions

An article on the kinetics of polymerization reactions was prepared, in 1940, in collaboration with Dr. H. Mark, to form a contribution to the series of monographs on High Polymeric Substances edited by Buck, Mark, Houwink, and Whitby. The manuscript was submitted to Professor Houwink of Leyden, but publication was delayed owing to the invasion of Holland. In addition to a discussion to the theory of the chain reaction in polymerization and of the kinetics of polymerization reactions, the manuscript contained an account of experimental kinetic investigations of styrene, of acrylic acid and its derivatives, of vinyl acetate, and of some miscellaneous hydrocarbons.

Butadiene

Towards the end of 1943, in cooperation with the Biology Division, work was undertaken on the production of butadiene from butylene glycol. This process was at that time receiving considerable attention from the United States Department of Agriculture as a possible source of butadiene for synthetic rubber.

In the National Research Laboratories an effort was made to develop a continuous process for the esterification of butylene glycol and the subsequent pyrolysis of the ester to butadiene. In the course of this work glycol esters were prepared from a large number of acids but none showed much promise of commercial application. Fair yields (60 to 70%) of butadiene were obtained from the adipic acid derivative by pyrolysis at relatively low temperature (350° to 400°), but work was stopped when no method was found for preventing the simultaneous pyrolysis of adipic acid to cyclopentanone. However, this pyrolysis of the adipic acid ester is probably the most convenient laboratory method yet found for preparing butadiene of high purity.

Vitamin A Assay

After the invasion of Norway, which had been practically the only source of cod liver oil for Canada, it became necessary for Canadian producers to manufacture cod liver oil. Since cod liver oil is priced according to its content of Vitamin A and Vitamin D, these have to be assayed on every batch of oil produced. The assays are made by the manufacturer but they have to be checked. The Dominion Experimental Farm undertook to determine the Vitamin D Content of these oils and the National Research Council agreed to carry out the determination of Vitamin A. This involved saponifying the oils, isolating a fraction containing the Vitamin A, and determining the Vitamin A content spectrophotometrically. The first two steps were carried out in the Division of Chemistry and the last one in the Division of Physics. On the average, four samples of oil were received per month.

Barium Phosphopyruvate

A request came from England for the preparation of phosphopyruvic acid in the form of its barium salt. The first attempt to prepare this salt by the use of a method given in the literature led to very poor results. The method was then modified and the substance was subsequently obtained in fair yields. It is prepared by the action of phosphorus oxychloride on pyruvic acid. The product is converted to the barium salt and the salt is purified by a lengthy process of repeatedly dissolving in dilute acid and reprecipitating by proper adjustment of the pH.

Starch

A polarimetric method for the estimation of starch

in wheat was extended to other materials in collaboration with the Association of Official Agricultural Chemists and adopted as a tentative method by the Association.

Isothiocyanates

Arising from the isolation of a sulphur-containing plant substance, some related compounds were synthesized in order to study the connection between the sulphur grouping and bitter taste.

Chlorohydrins and Glycols

The reaction of sodium hypochlorite with ethylenic compounds was studied. α-Cyano-phenylglyceric acid was prepared and its decomposition products were identified. During this work it was found that sodium hypochlorite is an efficient reagent for nuclear chlorination of certain aromatic acids and aldehydes. Some physical constants of 2,3-butylene glycol were determined.

Fatty Acids

Work on the occurrence of 11:12-eicosenoic acid in a vegetable oil was completed. The acid was identified by means of derivatives and degradation products.

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D. PROTECTION OF PERSONNEL AND EQUIPMENT

SPECIFICATIONS, TESTING, AND TEST METHODS

The routine testing of equipment and supplies for the Armed Services was a function of such agencies as the Inspection Board of the United Kingdom and Canada. However, considerable testing was done by the Division of Chemistry of the National Research Council before suitable facilities were established by the Inspection Board or because the Research Council possessed special equipment or experience requisite to the task. In general, the Research Council did not undertake routine testing when other suitable laboratories were available. The burden of this routine work fell particularly heavily upon the Textile, Rubber, Corrosion, Leather and Plastics, and Protective Coatings Laboratories.

A number of cases arose in which no specifications existed or in which existing specifications required modification due to the necessity of using substitute materials. In such cases the Research Council was often called upon to provide a suitable specification. This in turn often required the development of test methods appropriate to the case under consideration. Such activities occupied a considerable part of the time and effort of the Textile, Rubber, Corrosion, and Protective Laboratories, and constituted an extremely valuable, although unspectacular, contribution to the national war effort. The specifications prepared number several hundred and the tests performed number well over ten thousand. Although some reference will be made in what follows to particular aspects of this work, no effort will be made to discuss it in full.

Advisory Panels on Textiles, on Leather, and on Rubber were set up under the National Research Council Co-ordinating Committee on Protective Equipment, for the purpose of examining the technical requirements of specifications, to ensure that they were in order. All specifications received from the Directorate of Interservice Research and Development were transmitted to the members of the appropriate Panel; when necessary, meetings of the Panel were held to discuss them, and suggested changes were incorporated in new specifications. In all the work a very close liaison was maintained with the Directorate of Interservice Research and Development.

TEXTILES

Parachutes

When it was realized, early in the war, that there would be a shortage of silk for parachute canopies, and of linen for harness webbing, a Committee on Substitutes for Parachute Materials was formed, under the auspices of the National Research Council, with representation from the manufacturers and from the Air Force. An investigation was made of the properties required in parachute materials, and various possible substitutes were tested for strength, for elastic properties, for weight, for effect of light, and, in the case of canopy cloth, for air permeability. It is fortunate that two years before the war there had been introduced on the market a new synthetic textile fibre - nylon - from which a light material with high strength and good elastic properties could be produced, and which in this time of emergency proved to be the ideal material for parachute canopies. It was found possible to construct from it parachutes which were not only lighter, but also stronger, than the previous silk ones. In these parachutes the canopy and shrouds were of nylon, and the harness webbing of cotton. It speaks well for the work of the Committee that these new materials were first adopted officially in Canada.

The development work was handicapped by lack of definite information regarding the stresses to which the parachute is subjected when it open, hence decisions had to be based largely on full-scale field trials. Until recently the important question of impact strength had been neglected and work is in progress to compare the impact strength of nylon with that of fortisan - the only material with a reasonable degree of elasticity and at all comparable in strength.

The parachutes studied included those for flares as well as those for personnel. Since the requirements for flare parachutes were less critical, it was possible to substitute rayon by using a somewhat heavier fabric and reducing the strength requirements to a minimum. With the assistance of the Aeronautical Laboratory, a gauge was built to register the opening shock loads of the flare parachutes, but the gauge was found to have too low a range for the purpose, and the work was not continued because existing flare parachutes had proved satisfactory.

Routine Tests for the Subcommittee on Wool

Throughout the war years, testing work was continued for the Sub-Committee on Wool of the National Sheep Committee. This involved testing, each year, about 300 samples of wool clipped from sheep in experimental flocks in the West. These samples were all tested for clean yield of wool, for fibre diameter, and in some cases for fibre length and other special characteristics.

Aviation Medical Research - Protective Clothing

Problems assigned to the Textile Laboratory by the Subcommittee on Protective Clothing of the Associate Committee on Aviation Medical Research had to do mostly with the comfort of certain items of clothing. The first essential was to develop and standardize methods of evaluating fabrics and clothing for comfort, and methods of measuring thermal insulation. This was done in collaboration with the Ontario Research Foundation and the Banting and Best Institute of Medical Research. The National Research Council co-operated with the United States authorities, in standardizing thermal insulation test methods, by measuring the thermal insulation of samples already measured by them.

The thermal insulating properties of various fabrics were the chief subject of investigation. It was found that heat losses due to radiation could be reduced by applying a bright flexible metallic coating to cloth; and apparatus for the continuous electroplating of cloth was built and operated. Later, when it was found that double pile fabrics afforded the best means of attaining the insulation required, with a maximum of flexibility and a minimum of weight, work was largely confined to a study of the properties of these double pile fabrics, which were finally adopted as standard for lining air-crew suits. Quilted materials were also studied, but it was found that the required degree of insulation could be obtained only by sacrificing flexibility or lightness.

The early work showed that the thermal insulation of double pile fabrics was dependent upon their thickness; the construction of pile fabrics was therefore studied with a view to obtaining maximum resistance to compression without loss of insulation or lightness. The materials investigated included fabrics with piles made from mohair, alpaca, cotton (candle-wick), rayon, and Aralac, in single and double pile construction, as well as the sharling formerly used in airmen's jackets. The effect of various factors, particularly

that of humidity, were studied in connection with the compressibility of these materials. The best ground construction had to be determined as well as the most desirable quality of wool for it. It was also necessary to initiate water absorption studies of these fabrics.

Not only was it important to study the behaviour of the double pile fabrics, but it was also necessary to investigate the type of cover-all used over the double pile, and to investigate the variations in the properties of the coverall with wind conditions. This was an extensive piece of work requiring determinations under a wide variety of conditions. The permeability of coverall fabrics was shown to be important only with high winds; with low wind velocities the effects were independent of the type of coverall. The relationship between wind velocity and protection afforded by coveralls of various permeabilities was completely investigated. With this information, it is possible to evaluate any fabric with regard to wind protection. This study is still being pursued to determine the effect of wind on any free space behind the coverall materials and thence to calculate the thermal insulation in areas where the clothing is loose. With reference to this problem, the effect of air leakage, or ventilation, through clothing is one that deserves further study; information on warmth of clothing will not be complete until such experiments are carried out.

A study had to be made of the behaviour of the interlining materials during dry cleaning, laundering, and service. By varying the methods of determining thickness it was found possible to gauge fairly accurately the effective thickness of the material during service use, and hence to determine its insulation effectiveness, which had been shown to depend upon thickness.

Methods for evaluating the flotation properties of filler materials in life jackets were also studied. In this connection, the compression characteristics of milkweed, kapok, and polystyrene fibres were determined.

Tests on the waterproofing properties of various materials were also carried out for the Subcommittee on Protective Clothing. A project has been started for the purpose of determining the role played by various factors in controlling the water repellency of materials but this in only in its initial stages so that there is nothing to report as yet.

Investigations for the National Research Council Coordinating Committee on Protective Equipment (Research)

One project undertaken for the Coordinating Committee on Protective Equipment was the study of the present general service sock for the purpose of recommending a better wearing sock, and of determining the best method of reducing shrinkage. The study involved testing the products of fourteen different manufacturers, and developing proper test methods for shrinkage and for determining damage to socks resulting from any treatment given to reduce the felting properties of the wool. It was the responsibility of the Subcommittee working on this problem to make recommendations regarding the best method of producing this shrink resistance and regarding the standardization of sock construction. Although it has not yet been possible to make any decision regarding the best method of producing an anti-shrink sock, definite recommendations have been made regarding improved sock construction. Samples made according to these recommendations have been tested, and it has been found that by using the proper type of wool and by proper mechanical construction a very great reduction in shrinkage can be effected, so that chemical anti-shrink treatments are no longer necessary in order to meet the specifications.

Another investigation for this Committee indicated that no apparent advantage in preventing stains is obtained by providing a water repellent finish for clothing when the clothing is used under conditions in which the type of soiling is predominantly of an oily or greasy nature. This would be expected to be the case with overalls worn by mechanics. On the other hand, there is evidence that the use of a water repellent finish is a very great advantage in reducing the tencency to stain by aqueous types of dirt. While only one of the soils used in the present investigation was of the aqueous type, the results obtained, together with data gathered from previous work along this line, indicate quite clearly that for aqueous types of soil the use of a permanent water repellent finish (Velan) is advantageous.

Another investigation indicated that a satisfactory rotproof vamp-lining fabric could be produced by substituting a permanent-type cellulose finish containing a rotproofer for the previously used starch-type finish, which was known to promote the rotting of the lining. The rotproofers used in this investigation were commercial preparations in which the active ingredient is phenyl mercuric

ammonium acetate, and which do not appear to be irritating to the skin in the amounts used. The use of a permanent cellulose finish markedly increases the breaking strength of the fabric, and hence tends to increase its serviceability. A suggested specification for vamp lining and for vamp and quarter doublers was given in the report to the Committee.

An investigation was undertaken to determine whether satisfactory dyeings to match British Scamic shade No. 207 could be carried out on certain made-up items of undyed Army clothing. The dyeing trials were conducted at various cleaning and dyeing plants. The dyed garments were examined for shrinkage, for tendency to produce skin irritation, and for colour fastness to laundering, light, and perspiration. There was considerable variation in the fastness properties of the different dyeings, but it could be concluded that the process was a practical one, if satisfactory dyes and methods were used. No evidence of skin irritation was obtained.

Work was carried out on the development of an improved anti-gas tarpaulin, since it had been found that one meeting the requirements of the British specification was unsatisfactory. In cooperation with Canadian Industries Limited, a cotton duck fabric, impregnated and coated with butyl rubber, was developed. The fabric showed a very high resistance to liquid mustard gas and Lewisite, and possessed satisfactory flexibility at low temperature.

Other minor investigations carried out for the Committee are included in the bibliography.

Rotproofing

A limited amount of work on the rotproofing of textiles was begun in 1941, when it seemed that the war would spread to tropical areas. At that time considerable amounts of textile materials such as motor transport covers were being manufactured in Canada for use in the Middle East, and the question of rotproofing first arose in this connection. It should be emphasized that damage to textiles through microbiological attack occurs not only in the tropics but even in temperate climates under favourable conditions of temperature and humidity, such as occur in Canada in the warmer parts of the country and in poorly ventilated storage during the summer months.

The outbreak of war in the Pacific, at the end of 1941, gave additional impetus to research in the field of rotproofing, especially in the United States where large research programs were quickly set up by various agencies - e.g. Army Quartermaster Depots, the Bureau of Standards, and the Department of Agriculture. The National Research Council has been constantly in touch with these agencies. Since the beginning of 1942 there has been an ever-growing list of items requiring rotproofing. One instance of such a material, manufactured in Canada in very considerable quantity, is waterproofed duck, used for covers for motor transport vehicles. Several million yards of this material have been produced in Canada during the last four years. When the material was to be used under conditions in which microbiological attack might occur - for example, in the tropics - it was essential that it have an adequate degree of rotproofness. The publication by the Scientific Liaison Bureau, Melbourne, Australia, of the confidential "Report on the Condition of Service Material under Tropical Conditions in New Guinea" drew the attention of Service supply agencies to the great need for proper packaging and adequate protection against the ravages of moisture and microbiological attack. Although this report may have presented the story of the deterioration of Service material in the tropics at its worst, there was little doubt that, as long as hostilities in the tropics continued, there was the possibility of this type of deterioration.

Laboratory work in this field was therefore pursued with increasing intensity. A large amount of information on rotproofers was collected and compiled in an index covering the composition and properties of most of the rotproofing compounds on the market.

The first requirement for a program of investigation was to have reasonably satisfactory methods for assessing rotproofness. In general, use is made of two methods; one involves the use of pure cultures of one or other of the cellulose destroying fungi, and the other involves burial of the treated material in biologically active soil under controlled conditions of temperature and moisture. Arrangements were made for carrying out the pure culture tests in the Division of Applied Biology, while the soil burial tests were conducted in the Division of Chemistry.

As information on the efficacy of various rotproofing agents was collected, it became possible in the case of certain of these compounds, for example, copper naphthenate, to dispense with the biological tests, and to assess the degree

of rotproofness of the treated materials by means of a chemical analysis for the amount of rotproofer present.

The National Research Council has cooperated with the American Association of Textile Chemists and Colorists in the work of their subcommittee on test methods for assessing microbiological attack. Much helpful information has been obtained from research organizations in the United States in connection with work on the estimation of various rotproofing agents.

The types of rotproofing agents studied include aromatic phenolic derivatives, organic and inorganic copper compounds, dihydroxy-dichloro-diphenyl methane, organic mercurials, and chromium treatments, The aromatic phenolic derivatives are not sufficiently permanent due to their volatility and water solubility; the inorganic copper is too water soluble and appears to cause appreciable photochemical tendering of cellulosic fabrics; of organic copper soaps such as the oleate, oleo stearate, and naphthenate, the latter has been most used on this continent owing to the adequate supplies of naphthenic acid from the petroleum industry - it has been found quite satisfactory in use; where the rotproofer comes in contact with rubber, zinc naphthenate must be substituted for the copper compound; copper tannate has been successfully applied in Canada to the rotproofing of sewing thread; dihydroxy-dichloro-diphenyl methane has been widely used and found to possess satisfactory properties when used in conjunction with waterproofing waxes; the organic mercurials are not particularly effective by themselves and are rather easily removed by weathering, but when used with copper naphthenate they prevent the growth of copper tolerant bacteria, which, although harmless to cellulosic materials, remove the copper and leave the fabric open to attack by other organisms; the so-called "mineral khaki" treatment, obtained by precipitating iron and chromium hydroxides or carbonates in the fabric, has been found to be extremely fast to leaching and to offer considerable protection against actinic degradation, but to be poor protection against cellulose destroying fungi and bacteria.

It cannot be too strongly emphasized that it is necessary to carry out weathering tests on rotproofers in order to determine their stability under conditions of use. In many instances rotproofers have been placed on the market without due regard to this need. While it is customary to submit rotproofers to the action of leaching with running water, the importance of the effect of combined exposure to sunlight, rain, and wind has frequently been overlooked. In this connection there is also the possibility that certain types of

rotproofing compounds may cause degradation of cotton fabrics due to catalytic actinic action. For this reason the National Research Council has attached considerable importance to weathering tests.

A limited series of such tests were carried out during the summer of 1944. These dealt chiefly with the resistance to weathering of copper naphthenate, oleate, and "tallate", and of copper hydroxy naphthenate. It was found that copper naphthenate and copper hydroxy naphthenate both showed good fastness to weathering when present in association with waterproofing waxes. The weathering characteristics of the other compounds was of a considerably lower order.

Recommendations and specifications have been prepared regarding the rotproofing of numerous items of Service equipment, including tentage, cordage, sewing thread, paper, linen fire hose, insect netting, and cotton duck for haversacks, kit bags, hammocks, and assault boats.

Waterproofing

Resistance to water is important in a number of Service items such as protective clothing, cotton duck covers of all kinds, and canvas for assault boats. The textile laboratory's early activities in this field had to do with the setting up of test methods for assessing the water resistance of heavy fabrics.

As the demand for heavy waterproofed ducks for transport vehicles increased, the textile laboratory was instrumental in arranging for the production in Canada of this material, which had formerly been imported from the United States. The laboratory cooperated with the Army in the production of water repellent battle dress, and, in cooperation with the Rubber Laboratory, work was undertaken for the R.C.A.F. on the development of an improved plastic-coated fabric for raincoats.

Flameproofing

Work in the field of textile flameproofing was confined chiefly to giving advice on methods of treatment and methods of test.

Early work carried out by the textile laboratory had to do with flame resistant treatments for cotton clothing worn by workers in munitions factories. Recommendations were made covering the use of mixtures of borax and boric acid

applied at the end or the laundering operation. Since other types of water soluble, flame resistant materials were on the market, it was of interest to determine the possible damaging effect of all the commonly used water soluble flame-proofers when applied repeatedly to cotton garments after laundering, for it was considered possible that the effect of heat in the finishing operation might promote chemical tendering. It was found that certain of these materials - e.g. ammonium sulphamate - caused considerable degradation when applied repeatedly to the same fabric.

A considerable amount of testing was carried out on antiflash hoods and gloves treated with borax-boric acid mixtures, and recommendations regarding suitable amounts of the flameproofing mixture were made to various manufacturers.

Much attention has been given to methods of testing flame resistance. The usual type of test, involving the application of a flame to the lower edge of a fabric, under controlled conditions, was regarded as inadequate in the case of materials which in use might be subjected to the action of a flame impinging on their surface. Hence a surface burning test was devised and has become a standard method.

Assistance was given to the industry in applying permanent flame resistant treatments employing mixtures of chlorinated paraffins or aromatics in conjunction with antimony oxide.

Experimental cotton coveralls for tank crews were developed, in collaboration with the Chemical Warfare Laboratories, using vinylacetate-vinyl-chloride copolymer with antimony oxide, a treatment developed by the United States Chemical Warfare Service.

Bacteriostatic Treatments

At the request of the Associate Committee on Army Medical Research, laboratory and plant-scale studies were made of methods of testing army blankets to provide a fabric possessing a bacteriostatic or bactericidal surface, which would prevent the dissemination of airborne infection from blankets in hospital wards and dormitories. The problem was also of interest with respect to the possibility of producing a bactericidal fabric for use in ordinary items of combat clothing.

Methods of treating blankets after laundering were worked out which were satisfactory for this purpose. These methods were a decided improvement on those formerly available.

Laundering and Dry Cleaning

Assistance was given to the Armed Services in setting up their laundry and cleaning activities, and extensive assistance was rendered in solving various problems which arose concerning the laundering of Service clothing,

Close contact was maintained with the laundry and dry cleaning industry during the war, although it was necessary to discontinue research for private industry. It was considered advisable to continue the test bundle service, since this gave a measure of the washing efficiency of laundry and dry cleaning plants, and was, therefore, of importance from the point of view of prolonging the life of clothing. The referee service has also been maintained and has been of value in bringing to light certain shortcomings of wartime textiles. One such difficulty has had to do with an apparent reduction in the quality of dyestuffs used in consumer clothing, despite the fact that it has been well established that there is no need for such a reduction.

Work was completed on the development of a modified short formula for use in the laundering of white cottons, using silicated alkaline builders. This formula was put to use in a number of plants throughout Canada, and has effected a considerable saving in time and washing supplies, in addition to its beneficial effect in reducing the amount of mechanical action suffered by clothing during washing.

Substitute Materials

In addition to the work on parachutes, already discussed, other problems arose due to shortages of various textile fibres.

Work on cordage substitutes arose as a result of the critical shortages of hard fibres, such as manila and sisal. Substitute rope, of cotton, satisfactory for certain purposes, was developed in cooperation with the industry.

Another problem, initiated at the request of the Inspection Board, had to do with finding a substitute for the silk fabric used in ammunition exploder bags. Recommendations

regarding the use of a fabric woven from spun viscose yarn and carrying a special finish had been received from the United Kingdom. Considerable work was carried out on this project in cooperation with the industry, with particular reference to the finishing treatment, which employed a constituent (urea formaldehyde resin) designed to suppress deterioration of the viscose yarn caused by nitrous vapours. The rate of burning of the fabric was also controlled by the use of ammonium dihydrogen phosphate. The suitability of cotton as a substitute for the silk and linen thread previously used in these bags was investigated, and its use for this purpose was recommended.

Considerable work was carried out on the relative buoyancy properties of Canadian milkweed as compared to Java kapok. At a packing density equal to that used in life-saving equipment (3 pounds per cubic foot), the buoyancy of milkweed was found to be equal to that of kapok after immersion periods up to forty-eight hours. The loss in buoyancy over longer periods of immersion was slightly greater for milkweed. The effect of buoyancy of mechanical shock was also studied. There is no doubt that Canadian milkweed can be used as a substitute for kapok in buoyancy equipment but, unfortunately, no action was taken in this matter. Studies were also made of the buoyancy properties of cat-tail floss (typha), Bubbfil (viscose filaments carrying entrapped air), and water repellent cotton fibre. Cat-tail floss was tested as a substitute for kapok and considerable promise was shown.

Miscellaneous Investigations

Early in the war an investigation was started to determine the reason for the deterioration of cotton fabrics at the water-line when they are partly immersed. It was found that the effect was apparently due to the selective absorption of ions by the cotton, resulting in an acidic reaction at the water-line. Work was also initiated on a study of the constitution of wool; the wool was broken down by acid hydrolysis under various conditions and the relative proportion of liberated amine acids was determined. Both of these projects were abandoned because of the pressure of urgent war problems.

At the request of the Navy an effort was made to prolong the life of awnings used on shipboard, which had deteriorated very rapidly in the past. An examination of the old canvas indicated that weakening of the cotton was due to acid damage rather than to oxidation or mildew. Substantial lengths of canvas were treated (a) with cutch, (b) with a cellulose acetate coating, or (c) with sodium bicarbonate

followed by calcium chloride, which left calcium carbonate deposited on the fabric. These samples were subjected to service trials but were not returned, so it was not possible to compare the relative merits of the three treatments. However, all reports from the ship on which the pieces were under test indicated that after two and a half years of service they were still in good shape.

Tests were made to compare the degree of ventilation of standard bell tents with those fabricated from canvas proofed against fire, rot, and moisture; and to determine whether there is any hazard to personnel, or effect on their efficiency, due to carbon monoxide, carbon dioxide, or lack of oxygen resulting from occupying such tents when closed and heated under winter conditions. The ventilation rate was determined by observing the rate of decay of gas concentration after release of measured amounts of sulphur dioxide condensed in glass bulbs. The hazard to personnel was assessed by analyzing the air in the tents at definite intervals during the period in which they were occupied, and also by the examination of blood samples collected from the men before and after occupying the tents. There was no marked difference shown by the use of the proofed canvas, even under winter conditions, with a low outside wind velocity, the heater in operation, and the tent inhabited by six men.

LEATHER AND ALLIED PRODUCTS

A considerable amount of work was done in the Division of Chemistry in connection with leather products and equipment for the Services. The bulk of the work was concerned with the chemical and physical analysis of various kinds of leathers used in Service footwear, such as vegetable-tanned sole leathers, chrome-vegetable-retan upper leather, and chrome-tanned upper leather. From the laboratory viewpoint, comparatively good and uniform quality leathers have been produced throughout the war. In one case only, the laboratory confirmed the suspicion of the Inspection Board that a tanner was incapable of obtaining a satisfactory product, and his allocation of hides was redistributed by the control authorities to those who could. Similar analyses were carried out from time to time on garment leathers (chrome-tanned horsehide or cowhide) for such items as gloves, mitts, and flying helmets.

Various leather washers - e.g. washers for torpedoes, fuse cap washers for shells, and hydraulic leathers for the buffer mechanism of twenty-five-pounder and other guns - were

continually being tested to ensure a satisfactory product, free from constituents which might have a deleterious effect on the lifetime or performance of the stores involved.

During the period of acute fat shortages, tanners had great difficulty in obtaining suitable stuffing greases for Army upper leather, and physical tests on proposed mixtures enabled the quality to be maintained. Shoe polishes, dubbins, and other waterproofing materials for Service boots were frequently tested. Since the establishment of Service boot rebuilding depots, analyses of the various oils (straight and sulphonated fish oils, mostly) used in revivifying the leather have been carried out to ensure a uniform and satisfactory product. The causes of the cracking of chrome-tanned shoe vamps, and the causes of shoe insole deterioration were investigated. A technique for the measurement of the real density of leather was developed.

Abrasion resistance measurements on soleing materials have frequently been made. While it is recognized that abrasion is only one of the factors involved in the wear of soleing materials, and while the small variations in abrasion resistance between different sole leathers are not very significant, useful results have been obtained from such measurements in establishing, in a general way, the relative values of various synthetic soleing materials, and in indicating the superior wearing qualities of such materials over the average leather.

At the request of the Department of Munitions and Supply work was undertaken to produce a substitute for leather shoe soles. The material found most satisfactory for this purpose was fabric coated with polyvinyl butyral. Sufficient layers of this material laminated under heat and pressure gave a sheet of any desired thickness, which had a flexibility similar to leather. Wear tests on the product showed that it outwore leather by a factor of about two.

At the request of the Navy to develop a rubber shoe that would not slip on wet decks, various designs were cut in soft rubber, and the angle at which they slipped on a piece of wet steel, when weighted with a fixed load, was noted. A "V" design eventually was recommended.

Various minor problems encountered by the Services were investigated from time to time.

CORROSION

Corrosion is a relentless enemy against which an unremitting struggle must inevitably be fought - in war even as in peace. During the war the solution of specific problems submitted by the Armed Services occupied about three quarters of the efforts of the Corrosion Laboratory; the balance of the time was directed toward more fundamental investigations of corrosion phenomena.

Fundamental Research

Just prior to the outbreak of war, the laboratory began an experimental study of cadmium and zinc coatings on steel. Panels were exposed at the Atlantic and Pacific coasts at the half-tide position, and other panels were exposed in the laboratory. Cadmium plating and zinc plating were found to be about equally resistant to tap water; cadmium plating was the more resistant to sea-water at half-tide position.

A study was made of the corrosion resistance of low-alloy, high-strength steels in sea-water, exposures being made at both sea coasts. At the same time a study was made of the sea-water corrosion resistance of five aluminum alloys said to be quite resistant to sea-water. It is thought that this work should be extended because marine growths appear to have a bad effect on certain alloys, while others survive with but little or no attack. The type of growth and its incidence appears to be important. The evidence is quite clear that anodizing contributes no significant advantage to these alloys in resistance to corrosion by the sea. Of course, anodizing is an excellent base for paints.

One of the chief research activities of the laboratory is the devising of new and better corrosion test-methods. A new apparatus for continuous, completely submerged testing of metal specimens in liquids has been devised, its merits have been evaluated, and a description of the apparatus has been published. Preliminary work was done on a stirring corrosion test. Some work was done on methods of testing with humidity effects, but this was set aside pending provision of more space.

Another very important field of research is that of corrosion inhibitors. A search is being made for better inhibitors for antifreeze liquids, mostly with negative results so far. Inhibitors of corrosion of ordinary pipes by water presented a number of important unsolved problems; in particular, the published work on the effects of sodium

hexametaphosphate ("Calgon") appeared to be quite inadequate. An investigation revealed the important fact that sodium hexametaphosphate has little, if any, inhibitive effect in still or stagnant water - it is only active when the water is in motion. Furthermore, the amount of sodium hexametaphosphate that gives best inhibition is considerably greater than that alleged in published articles. These articles, unfortunately, present no quantitative data. When there is inhibition, the potential of iron against a calomel half cell drops. A few preliminary experiments have been made with sodium nitrite. A number of inhibitors will be studied in this connection - e.g. sodium silicates, phosphates, lime, soda ash, caustic, chromates, and organic compounds.

Metal cleaners were tested for corrosion effects, and some work was done to determine the relative efficiency of cleaners. This latter problem seems to have two aspects, namely, rate of cleaning, and amount of permissible contamination of the cleaner before discarding. The first aspect was solved: it was found that consistent and reproducible results could be obtained using steel panels which had been annealed by heating to 600°C. and cooling overnight in the furnace; the panels were then pickled to remove scale, and finally were polished electrolytically. With such surfaces, the times, or rates, of cleaning of oiled surfaces in the various cleaners were closely reproducible, usually checking within 15 seconds. The second aspect of the problem requires development of a test for cleanliness. Many cleaners contain wetting agents which make it possible to wet oils with water, so the water break test fails to measure cleanliness in such cases.

Incomplete studies were made of metal spray-coatings on steel, exposed in the sea at Miami, Florida. The object was to learn whether zinc protection blocks on ships may be replaced by patches of zinc spray-metal, applied repeatedly over old sprayed metal without more than wire-brushing the previous zinc surface. Time has not permitted further work on this project.

Experiments were done on electrolytic polishing of duraluminum. Invariably the electrolyte etched the work during removal from the electrolyte and before washing could be accomplished. Pulling the work through a layer of Nujol on the surface of the electrolyte reduced the etching. Further work is planned on this problem.

Very considerable test work was done, early in the war, on temporary protective coatings, that is, on oils,

greases, and waxes. These results were later very useful in making approvals and in preparing specifications. Upwards of a hundred of these materials were tested.

Specific Problems for the Armed Services

Early in the war the problem of a specification for dishwashing compounds for aluminum ware was presented. Such proprietary compounds usually consist of mixtures of soda ash, sodium metasilicate, trisodium phosphate, and sodium pyro-phosphate. Investigation showed that soda ash is the ingredient cheifly responsible for corrosive attack of aluminum, and that increasing the sodium metasilicate content of the mixture to 25% almost completely prevents this attack.

Many corrosion tests for the Armed Services were made on innumerable items of equipment such as ball bearings, flash lights, automobile lights, water filter barrels, webbing hardware, silver plated ware, coffee urns, strapping for shipping boxes, camouflage materials, and fuse boxes.

A long-term study was undertaken of drums of various metals containing aviation gasoline. The preliminary studies were useful in choosing metals for gas tanks on patrol-torpedo boats.

An extensive experimental study was made of the corrosion of steel by used crankcase oil.

A number of antifreezes were submitted for corrosion tests and, in cooperation with the Army, a practical road test was run on trucks to make a comparison between ethylene-glycol-type antifreeze and calcium-chloride-type antifreeze. The latter proved to be very corrosive.

Experimental studies were made of protective coatings for bomber exhaust manifolds of low carbon steel. On the basis of experiments, the Laboratory suggested consideration of the use of a single base coat of ferro-enamel, which could be applied inside as well as outside the manifold after fabrication. It is understood that this was used later in the United States as a substitute for unavailable alloys.

Considerable attention was given to coating smoke cups for twenty-five-pounder guns, a problem submitted by the Inspection Board. However, before proven success was attained, a modified method of filling solved the difficulty. A problem in rusting of large calibre shells was also studied.

Corrosion of corrugated steel huts for storage of shells near Montreal was investigsted. Measurements showed that the steel should last long enough without further protective treatment.

Some work was done for the Chemical Warfare Laboratories on corrosion by acids and by war gases.

Some attention was given to electrical insulation of dissimilar metal joints. Insulation of ship's propellors by using a grease loaded with mica between shaft and propellor was found ineffective in laboratory trials.

During the last two years of the war, work was done on packaging of metal articles for export to tropical regions. The Corrosion Laboratory early advocated testing of final packages in humidity. All containers, except tin cans or galvanized cans soldered closed, let moisture get inside; once inside, it cannot get out, and, in time, the article rusts, even though coated with a rust preventative. Rust prevention of metal articles is better with a heavier coating of rust preventative than with most wrappers. However, mechanical protection of the coated article is considered requisite.

Tropicalization of trucks has been considered by the Laboratory. Carburetors are made of die-cast zinc, which, in sea-water and sea air, corroses very rapidly in contact with the variety of dissimilar metals comprising a truck. A high temperature paint was found to give considerably enhanced protection to the zinc, and is now understood to be used in practice. The ball and socket joints of front steering wheels also corrode, so the Laboratory made up, and tested, a corrosion-inhibiting grease for this use. This grease has also been suggested for factory application to all nuts and bolts of vehicles, in peace as well as in war, as bolts and studs break due to rusting, particularly in the Pacific tropics. Brakes and brake drums also presented a serious problem of deterioration, the drums being alleged to perforate in a month opposite rivets of the lining. Tests were started in this latter connection but the alleged perforation effects could not be substantiated in laboratory simulated trials.

A number of problems encountered during operations were investigated, such as the corrosion of the lifting cables on aircraft engaged in North Sea patrol. This was found to be due to soldering flux, carelessly left by workmen assembling the work. In a number of cases of plane crashes, the Corrosion Laboratory was asked to investigate certain aspects of the case to help determine the cause of the accident.

Specific Problems for Commercial Firms

In 1940 the Laboratory studied the problem of severe sporadic corrosion of rivets in the hull of the 5000-ton train ferry, "Charlottetown". It was found that the badly corroded rivets showed Neumann lines in polished and etched sections, an evidence of finishing at too low a temperature - so low, that stress relief did not occur sufficiently. It was recommended that any future rivets be finished at as high a temperature as possible and that the rivet lines be sandblasted, spray-coated with metallic aluminium, and then painted, in order to try to prevent further deterioration. This was done, but the "Charlottetown" was sunk less than a year later, so there was no opportunity to learn the effects of the spray coating. It was learned subsequently that zinc should have been used because it repels fouling better than does aluminum.

Recently the problem of severe corrosion by cold brines in the new type of overhead-icing refrigerator cars of the Canadian National Railway was presented. Some preliminary suggestions were made which should eliminate some of the deterioration, and work is continuing on this problem.

Another problem recently presented by a commercial firm concerns treatment of mine hoisting cables with the firm's proprietary rust preventative. Preliminary experiments have been outlined in this difficult and awkward problem.

Upwards of one hundred reports were issued each year by the Corrosion Laboratory so that it is impossible to give a complete bibliography. Some of these are listed among those at the end of this Section.

PROTECTIVE COATINGS

The importance of protective coatings in the production of war equipment was indicated by the large volume of enquiries received by the Protective Coatings Laboratory of the National Research Council. This Laboratory was frequently called into consultation by the Armed Forces regarding various aspects of paint design, specification, and use, so that frequent meetings, discussions and job inspections were necessary. Many of the recommendations required lengthy investigations and laboratory trials. The employment of substitute materials also necessitated much investigation and proving in the Laboratory.

A large volume of approval testing was carried out for the British Supply Board and for the Inspection Board of the United Kingdom and Canada, from 1940 to 1943. As part of this work it was necessary in some cases to design special apparatus and to prepare new test procedures. Experience accumulated by the Laboratory enabled the staff to offer useful recommendations leading to the revision of specifications. For example, investigations of priming paints for shells resulted in a recommendation to adopt, for this purpose, an entirely different type of primer which would eliminate much of the trouble previously caused by premature corrosion.

Supervision of paints for motor transport constituted one of the most important war functions of the Laboratory on account of the great volume of production, which, up to the end of 1944, totalled 745,000 vehicles, valued at over two billion dollars. Thousands of gallons of vehicle paints were used daily throughout the period of the war, and many problems arose in connection with their use. Investigations were necessary in connection with the approval testing of paint, the standardization of colours, the determination of durability in service, the adjustment of difficulties in use, the cleaning and preparing of the metal for painting, and the selection of priming and finish paints and of special paints for wood bodies.

As paint is one of the chief materials relied upon for camouflage of buildings and permanent ground installations, camouflage paints and camouflage materials were studied in detail. Paints were selected suitable for Canadian use on coastal defence installations, buildings, and air fields; these included types for application to concrete, and to asphalt roadways, as well as to wood and to metal. Specifications were written to control their purchase and inspection. Experiments were conducted, and recommendations made, in respect to camouflage painting of motor vehicles, packing cases, galvanized roofs, Airforce buildings, and runways. When the use of steel wool for camouflage purposes introduced painting problems, since this material must be well protected by paint to prevent its destruction by corrosion, the Laboratory made recommendations as to paints and painting procedures, carried out tests of paints, and adjusted difficulties in early production. This made possible the production of considerable quantities of steel-wool camouflage nets in Canada in 1942. Recommendations were made as to methods of finishing other camouflage materials such as paper garnishing, camouflage cloth, camouflage nets, and tents. Accelerated weathering tests were conducted on a number of these materials. Close

study was directed to the matter of infrared reflectivity of camouflage paints, so that, when the Army requested information early in 1943, the Laboratory was well prepared to deal with the matter. Infrared reflectance standards had been assembled, and equipment for measuring infrared reflectance had been set up and calibrated. In this connection a number of samples of paint from production were tested, and recommendations were made to manufacturers as to choice of pigments and manufacturing methods.

In 1943 a request was received from the Department of National Defence for a material which would make canvas tentage resistant to water, flame, and mildew, and would also be satisfactory for comouflage. A survey of commercially available materials was made without finding one that was entirely suitable. Accordingly, development work was undertaken to formulate such a material and to provide test methods for determining its effectiveness. When satisfactory samples had been prepared in the Laboratory, International Paints (Canada) Ltd. co-operated by making pilot plant plant batches from the most promising formulae. The development of suitable materials and test methods permitted the writing of a specification for the preservative compound in collaboration with the Textile Laboratory. The preservative material chosen consisted of a chlorinated hydrocarbon base containing fire retardent pigments such as antimony oxide. Practical trials of the material on Army tents, carried out by Army personnel under the supervision of the Laboratory staff, demonstrated that the treatment was satisfactory. More recently, a large sample was supplied to the British Army Staff in India for experimental work.

Proper painting of landing barges constructed of plywood was an important problem on which the advice of the Laboratory was requested. After a study of the construction of the vessels and of the conditions of use, a painting scheme was prepared and put into effect. The Laboratory also assisted in providing decontaminable paints for use on folding boats and pontoons when the existing specifications were found to be unsuitable.

Occasional inquiries regarding the usefulness and availability of luminous paints were received throughout the war period. Shortly after commencing work on phosphorescent paints, it was realized that there was an urgent necessity for a method of measuring the brightness or luminosity of the paint coatings and the duration of such brightness. There was also need for the establishment of minimum

requirements for brightness and for the preparation of a specification which would include all of the essential features of a phosphorescent paint. It was soon discovered that physicists had not yet been successful in devising a simple and rapid method for measuring the brightness of surfaces at such low levels of brightness. The use of the MacBeth photometer was tedious since the observer must spend at least forty minutes becoming dark-adapted before taking a reading; furthermore, the accuracy obtainable was unsatisfactory. Considerable time was spend endeavouring to reproduce and to improve a photographic method originated by the United States Army, but it was eventually abandoned. About this time new methods were introduced in Great Britain and in the United States. The American apparatus was based on the use of photoelectric cells; this method was not considered acceptable because the relationship between the human eye and the photoelectric cell at low levels of brightness is not adequately known. The British method proved satisfactory and was adopted. It consisted essentially of a modified MacBeth photometer with a built-in device for subjecting the sample to a standardized irradiation with ultraviolet light. It did not, however, eliminate the necessity for becoming dark-adapted before making measurements. The necessary apparatus was constructed by the Optics Laboratory and, after a study of available commercial products, a specification (No. PR-2) was prepared to cover a luminous paint having high initial brightness and to cover one having long after-glow.

Extensive work was carried out on fire retardant paint for ships. Methods for the removal of heavy, oil-paint coatings were studied, and ships at Halifax were inspected to determine the condition of the painted areas with respect to fire hazard. Recommendations for stripping and refinishing were made. Specifications for fire retardant paints were written, methods of test were compared, and commercial products were evaluated. Other Naval paints considered included an interior coating for drinking water tanks, interior anti-condensation coatings, insulating varnishes, and exterior camouflage paints. A detailed study of gloss requirements, gloss standards, and measurements of gloss was carried out.

Aircraft finishes of many types were investigated. A major project was the selection of a suitable coating for the interior of plywood gasoline and alcohol tanks. This study was completed in collaboration with the Structures Laboratory of the Division of Mechanical Engineering.

Protective coatings for tropical use were studied in detail, particular attention being given to the evaluation of rotproofing coatings for wood. The evaluation of wood preservatives and rot resistant materials had always been difficult and time-consuming. During 1944 the Protective Coatings Laboratory developed a new method of test for such substances, involving the determination of loss in tensile strength of wood veneer strips dipped in the preservative and subjected to soil burial. By this means a numerical index of the efficiency of rotproofing agents can be obtained. In addition, performance testing of packaging waxes, mildew-proofing lacquers, and canvas preservatives was carried out.

TREATMENT FOR "TRENCH MOUTH"

In the clinical treatment of "trench mouth", officers of the Dental Corps had observed that Fowler's solution (i.e. 1% arsenious oxide in water, neutralized with potassium hydroxide) and hydrogen peroxide - both of which are old remedies of none too great effectiveness - when mixed together, became much more effective than would be expected. Efforts were made to utilize this discovery, but the mixture was found to have defects which could not be corrected: It etched the teeth badly in its original condition; if made sufficiently alkaline to prevent etching, the peroxide decomposed and the mixture was not effective; and, if made neutral, or faintly acid enough to be stable, etching reappeared. When the matter was referred to the National Research Council, it was felt in the Division of Chemistry that further study in the light of the above observation might lead to a solution of the problem.

The etching was clearly caused by the fact that arsenates, produced from arsenites in Fowler's solution by oxidation with the hydrogen peroxide, are isomorphous (that is, crystallize in identical form) with the phosphates of the teeth. Thus a solution containing arsenate, placed in contact with the teeth, removes a layer of phosphate, and deposits in its place a layer of arsenate. This is later removed as a foreign substance by the saliva, and the effect is repeated at each use of the solution.

A study of the underlying causes shows that there is only one way of combatting this difficulty, namely, the use of a solution actually supersaturated with respect to tooth substance, largely calcium and magnesium phosphates. Since such supersaturated solutions can be prepared only by special methods, it is clear why earlier efforts had not succeeded.

The ultimate objective was a useful treatment in tablet form but, since the need was urgent, a recipe was first provided which involved mixing a number of separate solutions. By this procedure it is quite easy to secure several-fold supersaturation and, fortunately, in the case of calcium phosphate such supersaturated solutions are adequately stable, if mixed only as needed for immediate dispensing.

The development, however, of a dry powder which would dissolve to provide a supersaturated solution was hardly expected to be successful, since it is axiomatic that solids will not dissolve past the saturation point. After much experimenting, however, it was discovered that a mixture of sodium or potassium phosphate with a calcium salt such as the benzoate would readily produce solutions considerably supersaturated. This, no doubt, is due to the organic nature of the calcium benzoate; it apparently dissolves as intact molecules and ionises later only after adequate dilution has been secured to prevent immediate precipitation.

The next recipe therefore used these salts but still required that the preparation be left as a more or less loose powder, for when this powder was pressed into compact tablets the intimate contact resulted in the production of insoluble calcium and magnesium phosphates. The powder was therefore packaged in capsules, to be dissolved in peroxide at the time of dispensing. A large number of tests were then made on methods of preparing a tablet which would still remain soluble after long storage. It was found that a frothing agent added still greater efficacy to the mixture, so different ways of incorporating this frothing agent were tested in attempts to stabilize the tablet. The best method found was to coat all particles of the powder with the frother by adding it to the powder as a benzene solution, rather than by incorporating it by grinding. These coated grains could then be mixed, without further grinding, and pressed into tablets.

The stability of the tablets produced in this way was tested by subjecting them alternately to a temperature of 60°C. for forty-eight hours, and then to 98% humidity at 20°C. for an equal period. After fifteen such cycles they were found to retain over 90% solubility, although they had swelled and crumbled slightly. Yet one drop of liquid water will cause a tablet to set immediately to a hardness resembling concrete.

All clinical tests during this work were conducted by the Canadian Dental Corps. The Department of Pensions and National Health made a valuable contribution by studying the toxicity of this form of arsenic. From these toxicity studies it would appear that the minimum lethal dose for man would be about one tablet every day for a week; and since each patient is issued only one tablet, already dissolved in peroxide, there seems no danger of accident in this respect. Although many thousands of cases have now been treated with this prescription, no single case of any reaction attributable to the arsenic has yet been noted. Many cases of sensitivity to the peroxide are found but these are readily adjusted by using it rather weaker, and the symptoms rapidly disappear after discontinuing its use.

In many cases twenty-four hours' treatment will arrest, and very markedly improve, serious conditions of the mouth; and only rarely is treatment as long as a week needed. Patients who cannot eat are frequently comfortable after one day, that is, after four applications of the treatment. Many conditions other than that commonly known as "trench mouth", benefit markedly, for example, several cases of Vincent's infection of the tonsils have been cured by its use. Indeed, many inflammatory conditions of the mouth appear to involve organisms against which this treatment is beneficial.

MISCELLANEOUS INVESTIGATIONS

Anti-Dim Materials

Anyone who has had his spectacles fog over in the winter-time upon entering a warm room will understand how serious the fogging of goggles or gas mask can be for a soldier or airman in combat. Anti-dim materials commonly in use are all soluble substances and are, therefore, removed readily as soon as condensation occurs. During the defrosting investigations it was noted that the polyethers of myricyl alcohol have properties which make them valuable as anti-dim agents. If the ether chain is not too long, that is, if it is less than about forty units long, these ethers are hardly soluble in water at all. They are, however, quite active anti-dims with any chain length above about eight units. Consequently, within this range, effective anti-dims may be obtained which are sufficiently insoluble that one application of material may be used over long periods. The Chemical Warfare Laboratories tested these materials and found them very satisfactory.

Plastic Armour

Early in the war, at the request of the Navy, an attempt was made to improve the plastic armour then in use as a substitute for steel armour plate. This plastic armour consisted of a mixture of gravel, limestone, and asphalt. About a year of continuous effort was spent experimenting with various shapes and mesh sizes of the gravel, different types of gravel and different ways and means of fabrication. In addition to this, a new type of non-metallic armour was developed, which consisted of crushed rock bonded with sodium silicate.

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E. CHEMICAL WARFARE

EVOLUTION OF ORGANIZATION WITHIN CHEMISTRY DIVISION, NATIONAL RESEARCH COUNCIL

During the First World War no investigations on chemical warfare, either offensive or defensive, were carried out in Canada, nor was there any manufacture in Canada of offensive or defensive supplies or equipment. Furthermore, Canada was one of the signatories of the 1925 Geneva Protocol outlawing gas warfare, so that, in the years following the armistice of 1918, the subject was permitted to become chiefly one of academic interest.

About 1934-5, after the Manchurian incident of 1932 and the changes in the German government, it began to appear desirable that some thought be given to chemical warfare and, at the request of the Department of National Defence, a member of the staff of the Division of Chemistry was authorized to devote some time to reviewing the technical literature in this field and to keeping informed regarding new developments; for, while there was no thought of Canada building up a military establishment for carrying out this type of warfare, it was definitely in the national interest to give thought to defensive measures against it.

In 1937 the Department of National Defence considered the assembly of respirators in Canada and the manufacture of at least some of their components.

In 1938 the Department of National Defence asked the National Research Council to send a member of its staff to England to inspect the various plants making respirator components and assembling complete respirators. While in England he also attended a course at the Chemical Defence Experimental Station at Porton. On his return to Canada it was considered that our knowledge was now sufficient to warrant the Canadian manufacture of respirators, importing only a few of the components from the United Kingdom. This work was taken in hand by the Department of National Defence with our staff member acting as consultant, and a small laboratory was set up in the National Research Council to test the respirator containers and the active charcoal used therein.

During this time our contacts with the Department of National Defence were becoming more numerous, and to improve liaison, an Associate Committee on Container Proofing

and Research was organized. Its terms of reference covered only the defence aspect, and the chief attention was given to the manufacture of respirators. The advent of war found the respirator production programme well advanced and the National Research Council with the nucleus of an organization. The Research Council staff had also gained some experience in 1938 by the preparation of small amounts of mustard gas, chloracetophenone, and brombenzyl cyanide, for use in summer training of troops at military camps.

About April 1940, plans were made to start a reference library. This took in charge the small number of British chemical warfare reports which up to that time had been kept in the Council's administrative section and it also received from National Defence a large number of Army specifications, such as those for paints and chemicals. Efforts to augment this collection were not immediately satisfactory, for at that time no direct liaison had been established in London; however, this condition soon improved. Air Raid Precautions literature was also acquired and indexed.

At the start of the war, offers of assistance poured in from all parts of Canada - in particular, from the universities. As needs became clearer, it was possible to utilize the services so offered. In the chemical warfare work, some fifty to sixty projects were initiated in the first two years of the war. Out of this work came valuable assistance on subjects of immediate interest and also a supply of well-trained scientists, who subsequently joined the staff of the chemical warfare organization.

After Dunkirk, the growth of the Chemical Warfare Section of the Division of Chemistry accelerated, and so did its activities. A number of the staff members went into uniform and were seconded back to the National Research Council to carry on with their work. Many new aspects of chemical defence were considered; and knowledge of the work was spread by bringing into the Associate Committee representatives of the Army, of the Navy, and of the Air Force. A number of staff members were sent to the United Kingdom at various times to become familiar with the work being carried on there, and to inform workers there of Canadian plans and investigations.

Early in 1941, the Associate Committee was dissolved and the Laboratory staff became the Research Establishment (C.W.) under the Army's Directorate of Technical Research. This may be considered the formal separation of the chemical warfare group from the organization of the

Division of Chemistry. However, some civilian members of the establishment remained on the Research Council payroll for some time, and other members of the Divisional staff continued to give considerable assistance in the laboratory work. Actually, the co-operation between the Division of Chemistry and Chemical Warfare Laboratories was so close at all times that for many of the projects it is difficult to say whether they were carried out by the Chemical Warfare Laboratories or by the Division of Chemistry. In the account which follows, investigations have been included in the Divisional history for which considerable scientific direction was provided by the staff of the Division of Chemistry - funds and laboratory assistants were frequently provided by the Chemical Warfare Laboratories and there was, of course, a continual exchange of scientific information between the officers of the Chemical Warfare Laboratories and Research Council staff engaged on related projects.

After the fall of France, a need arose for a site for large-scale chemical warfare experiments, since the United Kingdom had previously carried out such work in French Algeria. A Field Experimental Station was eventually established at Suffield, Alberta, as a joint United Kingdom-Canadian enterprise. By May of 1941, the staff of the establishment had increased to about twenty, and very considerable growth was in prospect, in particular, with respect to the Field Experimental Station at Suffield. The establishment was then reorganized, becoming the Chemical Warfare Laboratories of the Directorate of Chemical Warfare and Smoke. Change-over was completed by December 1st of that year.

One member of the Division of Chemistry staff continued to be engaged part time for about two years in checking production of asbestos-wool pads and in development of improved ones. Most of this work was done in the plants of the companies producing these pads; it was a matter of obtaining the asbestos in a state of subdivision such that it gave the desired filter properties when mixed with the wool.

The effect of mustard gas on concrete was studied on behalf of the Chemical Warfare Laboratories, and extensive tests were made of a number of products recommended as linings for concrete mustard gas storage tanks. The Divisional Workshops made up special pieces of equipment, as required, and the Divisional Stores supplied the numerous requirements of laboratory apparatus and chemicals not sufficient in quantity or value to warrant special outside purchase.

At various times during the war, requests were made for the investigation of carbon monoxide in army workshops, tank hangars, and aircraft, and for the determination of carbon monoxide in the blood of men and animals exposed under various conditions. Visits were made to a number of research centres in the United States and reports were prepared on developments in carbon monoxide detectors, instruments, and related problems. Other projects carried out by the Chemistry Division for the Chemical Warfare Establishment are described in the sections following.

While chemical warfare was not resorted to during the war, except in the use of smoke screens, it is possible that this omission was due wholly to the enemy's knowledge that we were well prepared and in a position to retaliate. Since it was not the policy of the United Nations to initiate chemical warfare, Canada's expenditures on this work were really a very cheap form of insurance. As the record shows, the Division of Chemistry provided the scientific nucleus from which the Chemical Warfare Laboratories evolved, supplied the laboratory space, obtained equipment and, when the Army finally took over the section, continued to afford it space, equipment loans, and considerable specialized scientific direction.

RESPIRATORS

In the preceding section it was shown how the Chemical Warfare Laboratories grew from the pre-war planning for respirator production. At the opening of hostilities, a small proofing laboratory was in existence at the National Research Council and mass production problems had been largely solved, in which latter connection the Rubber Laboratory had made a considerable contribution.

The outbreak of war in 1939 caused an immediate re-survey of plans, in order to produce in Canada such components as the active charcoal and filter pads, which it had previously been planned to import from the United Kingdom.

The filter pads were first investigated. These pads, consisting of a mixture of wool and asbestos fibre, are used in respirator containers to prevent the passage of particulate smokes, which would penetrate a container filled with charcoal only. Fortunately, the Division of Chemistry had been working on both wool and asbestos, so

that staff members were immediately available to start preliminary investigations. Within a few days contacts had been made with industrial companies equipped to produce this type of material and in the next few weeks experimental filters were produced at the National Research Laboratories, while one company prepared to go into production. Production began early in 1940 and by the spring of that year a second company was producing experimental batches. When work was undertaken to develop an improved wool-asbestos filter, the penetrometer used to test the filtering efficiency of the pads proved to be unsatisfactory. The Division of Physics was requested to design a more sensitive type and this new instrument became available in the spring of 1940.

Active carbon had not been made in Canada prior to 1939 because there was not enough demand for it to warrant starting a plant here. The field had been thoroughly investigated by at least one company and found unattractive. Once war started, however, interest in producing revived. While decisions regarding its manufacture rested with the Department of National Defence, the National Research Council was concerned in a consulting capacity and obtained and tested several brands of American manufacture.

Late in 1939, while in the United Kingdom regarding medical research, Sir Frederick Banting learned that the authorities there had become alarmed at the possibility of the enemy using arsine gas. In discussing gas defence generally, it was learned that, since about 1938, several European neutrals had been exploring the possibility of placing orders for respirators in the United States and that, among other things, they had asked concerning a charcoal which would be effective against arsine. A copper impregnated charcoal, which would be useful for this purpose, had been developed in the United States during the First World War and could, it was learned, be obtained through regular commercial channels. A trial lot was ordered at once and a request was sent to Ottawa to place a confirming order.

Meanwhile, the laboratory staff in Ottawa had been preparing to run proofing tests against arsine, in addition to the gases already used in proofing. The copper impregnated charcoal gave excellent results when received and, in general, behaved better and more uniformly than the silver treated charcoal.

This work was very timely for a final decision had been made to manufacture charcoal in Canada and the chemists in charge of this work were sent to the National Research Laboratories for preliminary training.

CARBON MONOXIDE CONTAINER DEVELOPMENT

Protective Container for Carbon Monoxide in Air at High Humidity

In order to protect personnel against carbon monoxide in tank landing craft and other situations where a considerable hazard from this gas exists, it was considered highly desirable to investigate the minimum requirements of drier and hopcalite (an efficient oxidation catalyst for carbon monoxide) in a small contained which could be attached to the face piece of an assault respirator and would thus afford good protection under the prevailing atmospheric conditions.

Hopcalite is very readily poisoned by water vapour, which is strongly adsorbed. Therefore, in order to avoid the use of excessive amounts of catalyst and to maintain the dimensions of the protective container within practical limits, it is necessary to have an efficient drying agent in the system.

The geometry of a catalytic bed consisting of layers of various drying agents and hopcalite, to provide adequate protection against high concentrations of carbon monoxide in air of high relative humidity, was investigated under air flow conditions sufficient to meet the requirements for the maximum rate of ventilation of the lungs in men at work.

The best results were obtained with magnesium perchlorate as the drying agent. For a container four inches in diameter, a layer of 2.0 inches of magnesium perchlorate with 0.5 inch of hopcalite will provide protection for over thirty minutes against toxic carbon monoxide concentrations, at a flow rate of 64 litres per minute, in air at 80% relative humidity.

The Oxidation of Carbon Monoxide by Silver Permanganate on Various Metallic Oxide Carriers

Although hopcalite, a mixture of manganese dioxide and copper oxide, is used widely in conjunction with various protective drying agents in carbon monoxide respirators and

detector instruments, it has many disadvantages. The life of the catalyst is roughly equivalent to the life of the drying agent, as hopcalite is rapidly poisoned by water vapour. Furthermore, the heat liberated in the presence of relatively high carbon monoxide concentrations in a respirator container of this type may raise the temperature of the effluent air to the point where some cooling device is necessary to protect the wearer. Thus, at a concentration of 0.35% carbon monoxide, the heat developed in a small container of the assault respirator type, during oxidation at a flow rate of 32 litres per minute, is sufficient to raise the temperature of the effluent air to 75°C, and at concentrations in excess of 0.50%, the temperature may rise to over 100°C.

The activities of a considerable number of preparations were therefore investigated in order to develop a compound which either would serve as a catalyst or would react stoichiometrically with carbon monoxide, in moist as well as dry air, and which, when incorporated in a protective container, would afford adequate protection and greater comfort to the wearer than present hopcalite containers of standard design.

It was found in preliminary studies that, of the various compounds investigated, silver permanganate showed the highest activity towards carbon monoxide in moist air. The activity increased when this compound was distributed on an oxide carrier. Consequently, a thorough study was made of various metallic oxides as carriers for silver permanganate, with special reference to the following factors in relation to activity and useful life:-

(a) Carbon Monoxide concentration
(b) Height of catalytic bed of material
(c) Flow rate (i.e. space velocity)
(d) Moisture content of granules
(e) Size of granules
(f) Temperature and relative humidity of influent air
(g) Induction period
(h) Aging at various temperatures
(i) Ratio of catalyst to carrier

Among the more efficient carriers investigated were diatomite silica, kaolin, chrysotile asbestos, and the oxides of copper, zinc, lead, antimony, molybdenum, tin, titanium, iron, cobalt, zirconium, and cerium. These silver permanganate preparations may be subdivided according to their carriers into two braod classes as follows:-

(a) mixtures which require about 8 to 10% of adsorbed water for optimum active life, such as those containing diatomite silica, kaolin, or finely ground asbestos.

(b) mixtures which are most active when little or no adsorbed water is present in the granules.

The reaction between silver permanganate and carbon monoxide is stoichiometric but the velocity of this chemical reaction may be increased greatly by the presence of a carrier, which, in this respect, acts catalytically. With the proper type of carrier, relatively high space velocities may be employed with complete oxidation of carbon monoxide to the dioxide, until about 80% of the permanganate has been converted to the manganite stage. Thereafter, the efficiency falls off owing to a sintering of the active surface.

The effective life of the catalyst (breakdown time) is inversely proportional to the carbon monoxide concentration and space velocity and is directly proportional to the height of catalytic material through which the gas stream is passed. There is an increase in active life with decrease in average particle size down to about 0.9 mm. diameter, below which there is no further improvement in activity. Although the optimum range of temperature for longest life would appear to lie between 24° and 40°C., the catalyst is still active enough at 0°C. for respirator requirements.

Special attention has been directed towards those metallic oxide preparations which showed sufficient stability, after prolonged aging at elevated temperatures, to warrant consideration for practical purposes such as use in respirator containers. Many catalysts which showed a high initial activity towards carbon monoxide, when freshly prepared, were eliminated from further consideration because of a slow decomposition reaction, resulting in the liberation of oxygen, the rate of which increased at higher temperatures. In this category may be placed those catalysts containing the oxides of aluminium, silicon, titanium, cadmium, cerium, vanadium, nickel, and manganese.

The most efficient catalyst discovered thus far is one containing 69 mole percent zinc oxide and 31 percent silver permanganate, dried to less than 0.5% adsorbed water. After prolonged aging at 60°C. this catalyst showed a life corresponding to several hours for efficient removal of 0.5% carbon monoxide in moist air under standard test conditions

in the assault respirator container. This performance is considerably better than that shown by equivalent hopcalite containers. The investigation is being continued.

The work was then transferred to the Chemical Warfare Pilot Plant to prepare this zinc oxide catalyst on a pilot plant scale and to fill a considerable number of respirator containers for physiological and service tests.

TOXIC SUBSTANCES

Oxido-bis-β-chlorodiethylsulphide

The first problem on toxic substances which was undertaken by the Organic Laboratory at the request of the Chemical Warfare Laboratories consisted in the preparation of oxide-bis-β-chlorodiethylsulphide. This substance is a by-product which accompanies mustard gas prepared by the action of hydrogen chloride on thiodiglycol. It is also a vesicant and a better knowledge of its properties was desirable. Although it is relatively easy to separate the mustard gas from the reaction mixture, it is extremely difficult to purify the oxido-compound which is the main by-product. Several attempts were made, therefore, to synthesize the substance by other methods, but all of these proved fruitless; since the main phase of the study had for its object the preparation of the disulphone of oxido-bis-β-chlorodiethylsulphide, a direct synthesis of this compound was attempted from mustard sulphone but no success had been achieved when a more pressing problem caused the work to be abandoned.

Nitrogen Mustards

It was known that before the collapse of their country the French chemists had devoted considerable effort to the study of tri-(β-chlorethyl)-amine and its vesicant properties. The French were also known to have had the large stores of this vesicant, which was commonly called "French mustard", and the Germans were in possession of these stores. It also became known that the Germans were manufacturing large quantities of a nitrogen mustard which was apparently not French mustard. This was suspected to be bis-(β-chlorethyl)-methylamine and chemists in England, the United States, and Canada immediately turned their attention to this substance. Part of this work was done in the National Research Laboratories. Bis-(β-chlorethyl)-methylamine can be prepared in the laboratory by the action of ethylene oxide

on methylamine and subsequent chlorination of the product. It has to be handled with extreme care because of its action on the skin and on the eyes. Several kilos were prepared in the Laboratory to supply various workers at the Research Council, and at Canadian universities, who had volunteered to study the substance. Only one mishap occurred during this work and it, fortunately, was without serious results.

Its rate of hydrolysis in water, its rate of reaction with itself, and its chemical properties were studied. Means were sought to stabilize it, for at room temperature it reacts with itself to form an innocuous compound. The search for substances capable of reducing the rate at which the nitrogen mustard decomposed was only partially successful. It was eventually abandoned because of the discovery that a homologue of this nitrogen mustard, which was just as toxic, was itself sufficiently stable to render the use of stabilizers unnecessary.

Various compounds partially similar in structure to the two nitrogen mustards referred to above were synthesized in the Laboratory and submitted to the Physiological Section of the Chemical Warfare Laboratories for toxicological assay. For instance, such substances as the following were prepared in the Laboratory:-

1. Ethyl N-$\beta\beta'$-dichloroethyl carbamate
2. $\beta\beta'$-dichloroethyl-nitroso-amine
3. β-chlorethyl N-β-chloroethyl-N-nitroso carbamate

A short investigation into the preparation and properties of tri-(β-chlorethyl)-amine, the so-called French mustard, was also carried out.

An extremely toxic gas was discovered in the United States by Dr. Kharasch but, unfortunately, this gas decomposed so readily on standing that it could not be stored with safety. However, the intermediate compound obtained just before the final step in the synthesis is quite stable. Several attempts were, therefore, made in this Laboratory to design a means whereby the last step in the synthesis, which is a nitrosation reaction, could be carried out in the shell just before use. The nitrosation reagents are so toxic to mice that it was not possible to observe the extent of the reaction nor its rate, because the animals died as readily from exposure to the vapor of the reagent used as they did after exposure to the gas.

VENTILATION AND ANTI-GAS PROTECTION IN ARMOURED TANKS

Carbon Monoxide and Gun Fumes

At the request of the Directorate of Chemical Warfare, an investigation was undertaken in December 1941 at Camp Borden on the carbon monoxide content of the atmosphere in various types of armoured tanks under general operating conditions and of carbon monoxide, nitrous, and ammonia fumes, generated during the firing of the guns under battle conditions. The concentration of these gases at the breathing level of the personnel in the tank during firing of the Besa, Browning, two-pounder, and six-pounder guns was studied under various rates of fire in order to evaluate the potential hazard to human life from this source. Whenever, it was demonstrated that, in the type of tank under examination, the gas conditions with the existing ventilation were dangerous at the maximum rate of fire likely to prevail in battle, an auxiliary ventilating system was installed and its effectiveness tested.

Under normal operating conditions, without the firing of the guns, the carbon monoxide concentrations attained from seepage of exhaust fumes in the various types of tanks, travelling alone or in convoy, were generally below 100 parts per million and did not constitute a danger to human health.

The concentration of gases discharged inside a tank during the firing of the armament depends upon the calibre and type of gun, rate and direction of air movement in the vicinity, rate of fire, and total number of rounds of ammunition expended. Higher carbon monoxide concentrations were found during the firing of the Besa machine gun (7.92 mm.) than during that of the Browning gun (0.30 in.) under similar conditions. If the ventilation rate inside the turret is appreciable, the gas concentration tends to drop quite rapidly in the reloading intervals between firing, but under other conditions dangerous amounts of carbon monoxide may accumulate.

Under similar weather and other operating conditions, the carbon monoxide hazard was found to be appreciably greater in the Ram I tank than in the Valentine VII for equivalent firing rates. Thus, in firing six rounds of two-pounder (A.P. Shot, Cordite) and 750 rounds of Browning accumition (S.A. Ball 30 06) in ten minutes there was recorded an average carbon monoxide concentration of 0.85% and a maximum of 0.102% in the Ram I tank compared with 0.017% and 0.023%, respectively, in the Valentine VII.

As the above and other data indicated that the Ram I tank was inadequately ventilated, a modified ventilating system was installed. This system resulted in a considerable improvement in carbon monoxide conditions, but it was deemed advisable, for adequate removal of fumes, to provide ventilation at a rate of at least 200 cu.ft. per minute in the main turret. It was found that the nitrous fumes released during the firing of the guns were not in sufficiently high concentration to constitute a hazard to the health of the crew.

Further tests disclosed that the ventilation system of the Ram II tank was also inadequate to reduce the fumes to a safe level during the firing of the armament, including the six-pounder gun, from a stationary position. The carbon monoxide concentrations exceeded the danger limit of 0.05% in all tests and frequently rose to values above 0.10%. Recommendations to correct these conditions were made.

Anti-gas Protection of Personnel

The object of this work was to develop a method of either collective or individual protection for the crew members of tanks against gun fumes or outside gas attacks. The investigation was carried out mainly along the following lines: (a) the development of a gas filtering unit with individual ducts leading to each crew member; (b) the degree of protection afforded by air at various flow rates delivered at a slight positive pressure to a loose-fitting transparent plastic mask covering a portion of the operator's face. Experiments were carried out in a gas chamber with various chloroacetophenone vapour and lethal concentrations of sulphur dioxide. With a volume of 3 to 4 cu.ft. of filtered air per minute for each operator, complete protection was afforded by drafts of air from various types of orifices, without the use of a mask, was also investigated. It was found that the effective zone of protection in this case was extremely limited, even at flow rates as high as 20 cu.ft. per minute for each operator.

Further studies of gun fumes and anti-gas protection were carried out at the Chemical Warfare Field Experimental Station, Suffield, Alberta. The project consisted not only of chemical tests to determine the concentration of gases released under various operating conditions but also of clinical and physiological tests to determine the effect of the gases upon the tank personnel. The results indicated that dangerously high concentrations of carbon monoxide and ammonia were released during the firing of the armament of the Ram II tank and the average carbon monoxide content of

the main turret could not be reduced to the safe level of less than 0.05% by volume without the use of an auxiliary exhaust fan ventilation system. In the absence of forced ventilation, rapid and prolonged firing of the six-pounder gun impaired the health and efficiency of the crew by causing a marked increase in the carbonmonoxyhaemoglobin content of the blood and by other physiological effects.

Complete protection was afforded against both gun fumes and non-persistent gas attack by use of an "anti-gas protective unit", which supplies four cu.ft. of filtered air per minute to each crew member.

COMBUSTION PRODUCTS OF FLAME-THROWERS

In connection with the physiological effects of flame-thrower attack on animals in various types of bunkers (as reported by the Experimental Station, Suffield, Alberta), it was considered important to study the combustion products of various types or compounds having widely different ignition temperatures, since the fuel ignition temperature for long range flame-throwers appears to have some bearing on the ability to maintain a burning rod in lengths of about 150 yards or more.

A measured quantity of the charge was injected under pressure into a specially constructed combustion chamber of about eight cu.ft. capacity and the mixture ignited simultaneously by a small hydrogen flame. Gas samples were collected at definite time intervals and the concentrations of carbon monoxide, carbon dioxide, and oxygen in the combustion products were determined.

The combustion of acetone, methyl ethyl ketone, dioxane, phenetole, and ethyl formate was found to yield considerably higher percentages of carbon monoxide than the combustion of gasoline, the standard flame-thrower fuel, under similar conditions. The addition of iron pentacarbonyl to gasoline also resulted in an increase in the carbon monoxide content of the combustion products. As there is a correlation between the carbon monoxide formed and the fuel ignition temperature, it was suggested that liquid fuels having a high ignition temperature be substituted for gasoline in flame-thrower tests.

ANALYSIS OF AIR FOR TOXIC FUMES

The Estimation of Low Concentrations of Nitric Oxide and Nitrogen Dioxide

The accurate determination of low concentrations of "nitrous fumes" in air is of importance in gun fume problems, testing of respirators, physiological experiments on toxicity, and in many other fields. As little as 40 parts per million by volume may be decidedly toxic to human beings. As it has been well established that the oxidation of nitric oxide in air or oxygen follows a termolecular law, and as it can be assumed that in very dilute gaseous mixtures all the nitric oxide molecules which have undergone oxidation are in the disocciated form of nitrogen dioxide, the problem resolved itself into finding a method for determining the relative proportions of nitric oxide and nitrogen dioxide in an air stream.

The methods finally adopted were the determination of total oxides (nitric oxide plus nitrogen dioxide) and of nitrogen dioxide simultaneously. The gas sample for total oxides was collected at a measured flow rate by a water displacement method and the oxides in both the gas and water phases were converted to nitrate with acid hydrogen peroxide solution. The nitrate was then reacted with phenol disulphonic acid and estimated quantitatively in a photoelectric colorimeter. The nitrogen dioxide was determined by passage of the gas sample at a measured flow rate through an absorption train of high efficiency, containing 10% potassium iodide solution. The iodine liberated in this reaction was estimated by titration with standard sodium thiosulphate, 0.002 N, using starch indicator.

As a test of these methods, a study was made of the rate of oxidation of nitric oxide in an air stream in the concentration range of about 0.75% to less than 0.10% by volume. The values for the absolute reaction velocity constant K, calculated on the basis of a third order reaction, were found to be in good agreement with those reported in the literature.

Rapid Determination of Low Concentrations of Ammonia in Air

Field trials of the Light Type respirator with a new model container disclosed that ammonia could be detected in the inspired air and in a number of cases the concentration of ammonia proved high enough to be objectionable to

personnel. This seemed to be true particularly of charcoal respirators in which the nitrogen content of the charcoal was relatively high.

As the problem involved the testing of a considerable number of charcoal containers to determine the effect of various treatments in eliminating this objectionable feature in the charcoal, a rapid and accurate method capable of detecting as little as one milligram of ammonia per cubic metre of air was desirable.

After some unsuccessful trials with potentiometric methods, a suitable and rapid procedure was evolved by measuring the change in conductivity of a definite volume of 0.0016 N sulphuric acid solution, maintained at constant temperature, in an absorber containing a sintered glass disc of fine porosity. A measured volume of air from the charcoal under examination was passed through this solution and the change in conductivity was determined by a Wheatstone bridge galvanometer, using platinized platinum electrodes having a known cell constant. Tests on the charcoal in these special Light Type containers showed ammonia concentrations as high as 25 to 35 milligrams per cubic meter in the effluent air under high humidity conditions. Treatment of the charcoal with water followed by heating in a stream of air at 170-180°C. was found to be effective in removing nearly all of the volatile ammonia.

MINIATURE INCENDIARY BOMBS

This investigation was initiated to determine the feasibility of a proposal submitted to the Inventions Board of Canada for the production of small incendiary bombs which would ignite by gentle percussion or shock when sensitized. In view of the fact that millions of these pellets could be broadcast by plane over enemy territory, numerous small fires would result and would devastate effectively the forests and agricultural crops. The idea seemed to be a distinct improvement on the incendiary leaflets used early in the war and, for the purpose in hand, considerably better than large thermite or magnesium bombs. Although an effective miniature bomb was developed quite rapidly, its use against the enemy was rejected, at first, on humanitarian grounds. Later, however, after field trials at the Experimental Station, Suffield, Alberta, a modified version of this pellet was adopted.

The bombs consisted of small nitrocellulose plastic rods, spheres, and cubes, coated with a red phosphorus emulsion in acetone, and sensitized by an immersion method in a mixture of sodium chlorate, acetone, and water. Extensive tests were carried out to determine the best means of coating the nitrocellulose plastic in order to obtain pellets which would ignite readily when thrown on surfaces such as grass and soil and yet which would not be too dangerous to handle. Various concentration factors were studied in order to evaluate the limits of sensitivity and the question of rapid drying while falling through the air.

In the method of preparation finally adopted, the pellets, although safe to handle in the moist condition, become sensitive in less than one minute when falling through the air or when dried in an air stream. The bombs then ignite even when thrown into a bed of soil from a height of only five feet. One million of these, in the form of cylinders 1/4 inch in diameter and 1/4 inch in length, weigh about 650 pounds and cost about $450 to $500 to manufacture.

ANTI-GAS FABRICS AND CLOTHING

In September 1939, the Department of National Defence asked the Textile Laboratory of the Division of Chemistry to co-operate in work on anti-gas protective clothing. There was, therefore, a very close association between the Textile Laboratory and the Chemical Warfare Laboratories all through the war.

Good facilities for the examination of textiles were available in the laboratories of the National Research Council but it was necessary to extend these greatly to take care of the varied types of work required in connection with protective clothing. It was necessary, first, to provide additional equipment for laboratory studies of permeable and impermeable protective clothing. It was later necessary to organize and supervise pilot plant and full-scale plant investigations, chiefly in the field of permeable clothing. A considerable amount of co-operative work was carried on with outside firms in the field of impermeable clothing. The term "impermeable" is here used to designate fabrics carrying a continuous coating of some vesicant-resistant material; such fabrics were used in the manufacture of anti-gas capes, boots, etc. The term "permeable" is used to designate fabrics impregnated with some chemical substance which, by reacting chemically with vesicants, prevents their passage through the fabric; such fabrics retain their porosity to air, whereas

the impermeable type do not.

It was finally decided by the Chemical Warfare Laboratories that a small pilot plant for processing fabrics and garments should be set up; the Textile Laboratory was responsible for designing and equipping the plant, and also for its operation in the early stages. Later the plant was turned over to the Development Section of the Chemical Warfare Laboratories.

The Textile Laboratory has carried out investigations and tests on many types of equipment. These include: battle dress serge; protective wrapping material; butyl coated fabrics; neoprene; capes; gloves; webbing; adhesive tape; decontamination mitts; anti-gas suits; eyeshields; rayon fabric for ground sheets; anti-dimming cloth; moistureproof cellophane; oilskin jackets; stockinettes; anti-gas respirator fabric; parchment paper; elastic webbing; rubber sponge; fearnought flannel; detector gas sleeves; helmet curtains; leather samples; pliofilm; aprons; suits, including trousers, jackets and hoods; naval coats; zippers for anti-gas valises; haversack duck; capes; and bags for contaminated clothing.

Work on Impermeable Type Fabrics

Methods for Measuring Penetration Times: The method first used for measuring penetration times of liquid mustard and Lewisite was that developed by the Chemical Defence Experimental Station, Porton, England, involving the use of a metal diffusion cup and using a dilute solution of potassium permanganate as indicator. This method was found to be unsatisfactory. The indicator gave inconsistent results and was replaced by a solution of alizarin red adjusted to a pH of 6.1. The brass cups used in the Porton method were replaced by flanged glass cups mounted in batteries of twelve in a thermostatically controlled water bath, all connections on the off side of the cups being glass to glass. This method was similar to that finally adopted by the United States Chemical Warfare Service.

Penetration Time Limits: The problem of setting up permissible penetration time limits for impermeable type fabrics was a difficult one, since it involved an attempt to correlate laboratory penetration times with those which would be expected to cause injury under field conditions. It was therefore necessary to work in close co-operation

with the Physiological Section of the Chemical Warfare Laboratories in order to choose probable safe laboratory penetration times for the various types of impermeable fabrics used in the manufacture of anti-gas capes.

Co-operation with Canadian Manufacturers of Anti-gas Fabrics: When the manufacture of anti-gas fabrics was begun in Canada in 1940, the manufacturers possessed little or no experience in this field. This difficulty was complicated by the fact that high initial production was required. The Council co-operated with four firms but only one of these appeared to possess the necessary knowledge and technical control to produce a satisfactory range of anti-gas fabrics. A fair amount of this type of fabric was processed by a second firm but great difficulty was encountered owing to their lack of proper technical control. Co-operation with these firms involved the carrying out of hundreds of tests on experimental production runs, together with numerous consultations and plant visits. One troublesome property of oil-dressed fabrics was their tendency to undergo spontaneous combustion where the aging of the polymerized oil coating was incomplete. Two plant fires from this cause were investigated and recommendations were made to prevent their re-occurrence.

The Textile Laboratory was requested by the Inspection Board to undertake the setting up of an adequate system of inspection for these manufactured fabrics and to perform all the necessary testing. In the course of this work the Laboratory issued approximately 2500 routine inspection reports covering the manufacture of sixteen million yards of anti-gas fabric.

Investigation of other types of Impermeable Fabric: Considerable work was carried out on the investigation of other types of impermeable fabrics, namely, those having various types of synthetic resin and synthetic rubber coatings. The inability of the oil-dressed coatings to withstand low temperatures made it desirable to investigate coatings which would remain flexible at temperatures of 0°F, and lower. In this connection the outstanding properties of butyl rubber, both with regard to flexibility of low temperatures and to high protection against mustard gas and Lewisite, were early recognised. Unfortunately, adequate supplies of butyl rubber became available only at the end of the production of anti-gas fabrics in Canada, but there is little doubt that any future production would make use of butyl rubber.

Work on Permeable Type Fabrics

Laboratory Investigations: The various methods for measuring penetration times of permeable fabrics, as developed at Porton, were tried out and it was finally agreed to adopt the standard Congo red penetration method at 30°C. The laboratory facilities were therefore extended to carry out these tests on a large scale.

Owing to the fact that the impregnants used are useful by virtue of their content of active chlorine, it was obvious that there was a danger of damage occurring, especially in the case of cotton fabrics. A considerable amount of work was therefore carried out on the effects of various impregnants on wool and cotton under conditions of accelerated aging. At 65% relative humidity impregnated cotton cloth showed high rates of loss of impregnant and of breaking strength after twelve weeks' storage, whilst in the case of wool the loss was considerably less.

An investigation was also undertaken of the effect of other types of impregnating chemicals, with particular reference to their stability and to probable damage to the fabric. Included in this investigation was the effect of different types of dyes and cloth finishes; in general, sulphur dyes had an adverse effect on the stability of the impregnants. Associated with this problem was the effect of washing and dry cleaning on impregnated garments; experiments were conducted to determine whether or not they could be cleaned without reducing the impregnant content below a satisfactory level.

Work on the laundering of one type of impregnant treated cotton fabrics was extended to include laundering with various types of detergents, including soap, soap plus alkali, and sulphated fatty alcohol. The results indicated that the sulphated fatty alcohol caused less reduction in mustard penetration time than was the case with soap. The use of soap plus alkali appeared to offer little advantage.

Development of Plant Impregnating Processes: (a) Solvent Suspension Process: Work on impregnation was commenced on a laboratory scale in 1941. In 1942 substantial contract demands were raised by the Department of Munitions and Supply for impregnated battle dress for use by the Canadian Army overseas. The laboratory operations were therefore extended to pilot plant scale and one thousand

suits of Army battle dress and a thousand pair of socks were impregnated. The finished garments were tested and found quite satisfactory.

Some of the factors which influence the degree of impregnation were investigated. The speed of centrifuging, the concentration of the impregnating solution, and the type of material had the greatest effect. The duration of centrifuging had little influence. The omission of the centrifuging process, together with a suitable reduction in the concentration of the impregnating solution, gave a uniform, readily-controlled product. It was found that the degree of impregnation can be calculated from the increase in weight of the treated material with an accuracy sufficient to serve as a processing check.

Full commercial production was then commenced in the dry cleaning plants of Langleys Ltd., Toronto, and Vails Ltd., Ottawa. Some 400,000 suits of battle dress were processed throughout the duration of these contracts. Reports have been issued giving details of layout and operations at these plants. It was found that pinned-on pieces offered a satisfactory method of sampling the product for inspection.

(b) Solvent Processes:- Investigations were also made of trichlorethylene, perchlorethylene, tetrachloroethane and pentachloroethane with regard to their possible use as solvents in the impregnation process. Tetrachloroethane appeared to be the most suitable solvent investigated. The Laboratory also looked into the effect of steam pressing on the impregnant content of the treated materials, and into the effect of increased chlorinated hydrocarbon content on the properties of impregnated material.

(c) Aqueous Processes:- During 1942 and 1943 the trend in anti-gas impregnation of clothing was in the direction of aqueous processes rather than processes involving solvent solutions or solvent suspensions of the impregnants. Work on aqueous processes was begun at the fabric processing pilot plant late in 1943. Owing to the unexpected deterioration of a batch of brown-sulphur-dyed drill impregnated by the British aqueous process, a study was made of the causes of tendering. This was found to be due to the effect of the impregnant on the sulphur dye in the fabric.

Storage and Wearing Trials: Representative samples of battle dress treated with one type of impregnant were examined after six and twelve months' storage for appearance, impregnant content, and chemical damage to the cotton sewing thread. The garments were found to be quite satisfactory.

Samples of impregnated battle dress were subjected to wearing trials and garments were withdrawn for examination at intervals of one, two, three, and four months. There was a gradual loss of protective value caused by mechanical removal of impregnant; higher chlorinated-paraffin content tended to reduce this loss.

Miscellaneous Investigations on Impregnated Clothing: Samples of four fabrics, exposed to the vapour of methyl-dichlorosilicane for thirty minutes, attained varying degrees of water repellancy, depending on the type of fabric. Light-weight cotton materials having a fine weave, such as mercerized cotton or tarantule, gave the best results, followed closely by wool serge. The results with cotton denim were decidedly inferior. Laundering destroyed much of the water repellent quality of wool serge and of cotton denim but did not appear to affect mercerized cotton or tarantule. Silicane treatment followed by impregnation is applicable only to wool serge material and even here it is doubtful if the silicane is of any value.

Battle dress serge impregnated with charcoal was tested for mustard and Lewisite vapour penetration times and for the effect of laundering and dry cleaning upon the subsequent penetration time. It was found that a negligible amount of carbon was removed by the aqueous detergent solutions. A much smaller amount of carbon was removed by Stoddard solvent than by trichlorethylene or perchlorethylene, but the cloth retained its original vapour resistance best in the case of cleaning with trichlorethylene.

Velanized battle dress serge, impregnated by two different processes, showed a slightly greater overall protective value against mustard vapour as compared to the non-velanized impregnated fabric. The water repellancy of velanized battle dress serge was slightly reduced by impregnation, somewhat more so than in the case of non-velanized serge. The results of accelerated aging test indicated that the stability of the impregnants was somewhat greater in the case of the velanized serge.

PROTECTIVE COATINGS

A number of investigations in connection with chemical warfare were carried out by the Protective Coatings Laboratory of the Division of Chemistry:

Gas Detection

In the month of October, 1939, an urgent request was received from the Royal Canadian Navy for the preparation of a pilot batch of gas detector paint for immediate use on H.M.C. ships. A specification for this material was obtained and arrangements were made with a local paint company to manufacture the varnish vehicle. As soon as the materials were received, they were processed on the laboratory paint mills, mixed according to the specification with the active agent and solvents, packaged in one-quarter pint cans, and labelled. This is believed to be the first batch of gas detector paint manufactured in Canada.

Development work in connection with detector paint was then continued in co-operation with the Chemical Warfare Laboratories, and specifications were prepared covering detector paint in various colours and compositions.

Another of the early projects was the development and testing of detector powders for application to uniforms and other fabrics. Their use was later discontinued but not before satisfactory formulae had been developed and commercial production had commenced.

A brief study was conducted to determine the deleterious action, if any, of chromium compounds in detector paints.

The Laboratory assisted in the development of a suitable detector paint for application to detachable paper "sleeves" and a highly satisfactory product was put into production by the Canada Paint Company as the result of the combined effort. Some 30,000 gallons of this particular paint were manufactured and millions of the sleeve protectors were made up by contractors and distributed to the Armed Forces. In connection with the contracts for this item, the Laboratory assisted further by conducting approval tests on many batches of the paint and by investigating occasional difficulties which arose from time to time. Approval tests were also carried out on the active ingredient for incorporation into the paint. Colour standards were prepared for both the yellow and khaki types of paint.

It is essential that detector paint contain no turpentine or aromatic solvents. The Laboratory developed test procedures for detecting the presence of small amounts of such liquids. Early in 1943, the use of detector paint on steel plates was begun, and the Laboratory co-operated in preparing the specification for the priming paint, the detector paint, and the method of application.

Chemicals and Smokes

Assistance was given to the Royal Canadian Navy in the procurement and inspection of the special paint remover required for the removal of paint from ships in case of poison gas contamination. Instructions for its use, including the necessary precautions, were supplied to Naval Service.

Inspection tests were also made of many samples of various chemicals and compositions for smokes including zinc oxide, aluminum powder, and calcium silicide.

Interior Coating for Gas Shells

The interior of gas shells must be coated to prevent interaction between the metal and the chemical. This is highly important from the standpoint of safety. Manufacture of an enamel for this purpose was commenced by a Canadian paint company in 1943, following instructions supplied from Great Britain. The Protective Coatings Laboratory was assigned the duty of carrying out the rigid inspection tests and found that serious problems were occurring in its manufacture. Responsibility for correcting these difficulties was then assumed by the Inspection Board.

A similar project had been undertaken previously, namely, production of a lining for storage tanks for war gases. A promising material for this purpose was developed in conjunction with one of the leading resin manufacturers and samples were submitted to the Chemical Warfare Laboratories. It was found, however, that rather than run the risk of imperfection in the coating, due to improper application, it was preferable to use chemically resistant containers which required no coating.

F. APPENDIX

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