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A STATE EXPERIMENT IN CHEMICAL RESEARCH¹

By Professor G. T. MORGAN, F.R.S.

SUPERINTENDENT OF THE CHEMICAL RESEARCH LABORATORY, DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH

AT the Bristol meeting of 1875 my predecessor, Professor A. G. Vernon Harcourt, spoke to this section on the teaching of chemistry, and in the course of his very inspiring address he remarked that "the science of chemistry would advance more rapidly if it were possible to organize chemists into working parties having each a definite region to explore," and he went on to inquire, "Is such an organization in any degree possible?"

I propose this morning to describe the attempt recently made by a department of state, namely, the Department of Scientific and Industrial Research, to give effect to Professor Vernon Harcourt's prophetic vision. The answer to his question is in the affirmative. Such an organization is in some degree possible, and has actually become an accomplished fact. I must, however, leave for one of my successors in this

chair the further inquiry, "Can such an organization become permanent and still retain its primary and paramount function of chemical exploration?"

ORIGIN OF THE CHEMICAL RESEARCH LABORATORY

The work of the Department of Scientific and Industrial Research began in 1915, and during the ensuing ten years the department had at various times become interested in investigations of a chemical nature, such, for example, as (1) large-scale researches on the chlorination of methane; (2) large-scale researches on the production of formaldehyde, (3) investigations on the production of glycerine, (4) investigations on the manufacture of chemical products from fish residues, (5) general researches on the corrosion of metals, (6) general researches on high-pressure reactions, including the reactions between carbon monoxide and hydrogen.

These investigations, which were undertaken mainly

¹ Address of the president of Section B—Chemistry, British Association for the Advancement of Science, Bristol, September, 1930.

under the auspices of the Chemistry Coordinating Research Board, were carried out by isolated groups of workers, who were often located in widely separated laboratories. One group studied the corrosion of metals at the Royal School of Mines, another examined fish products in the Imperial College of Science and Technology, whereas a third experimented on the chlorination of methane and on the recovery of formaldehyde from waste liquors of wool-scouring at the Royal Naval Cordite Factory in Dorsetshire.

It soon became evident that some increase in economy and efficiency could be attained by bringing together under one roof these scattered groups of workers who would receive encouragement and stimulus by becoming part of a more centralized scientific organization.

A suitable site was chosen on the Bushy Park Estate in close proximity to the National Physical Laboratory and the Admiralty Research Laboratory, and here in 1924 the building of a chemical laboratory was commenced on a plot of land allowing ample scope for future expansion.

The original plans drawn out by the architects of H.M. Office of Works made provision for three laboratory units each of rectangular shape and built round four sides of a central courtyard. The front and back of the hollow rectangle consist of two two-story blocks; the front block designed for general and special small scale laboratories with the necessary offices, the back block arranged to accommodate workshops, service rooms and heating plant. The two sides of the rectangle, which consist of two single-story blocks with saw-toothed roofs, north lighted and with a clear head room of about sixteen feet, give adequate space for large-scale laboratories.

These buildings are constructed in steel and brick and so arranged that partitions can be readily removed for alterations or extensions. In the two-story blocks the floors and roofs are formed of hollow concrete tubes, but in the engineering section of the building, where heavy superloading had to be considered, a more rigid type of construction in steel and concrete floors was adopted. In the interest of economy, plaster and other relatively expensive internal finishings were omitted wherever possible, any distemper or paint being applied to flush-pointed brickwork. The floors were covered with stout cork carpet, laid directly on the cement rendering.

The laboratories are equipped with specially designed fittings, the framing and fronts are of stained British Columbia pine, whereas the bench tops and other portions subjected to hard wear are in teak or Iroko wood. The internal drainage to laboratory sinks is effected by open stone-ware three quarter circular channels finished with acid-resisting glaze.

Wherever exposed internally, structural steel and joiners' work are coated with acid-resisting paint. The benches of small scale and special laboratories carry five services—gas, water, steam, vacuum and compressed air. Each room is amply supplied with electric current (D.C.).

In conformity with the neighboring buildings of the National Physical Laboratory, a simple modern Georgian style was adopted in the design of the elevations of the new laboratory. The buildings are faced externally with multi-stone sand face bricks, reconstructed Portland stone being used sparingly in cornices, string courses and entrance doorways.

The construction of one of these units was started towards the end of 1924, and when scientific work was commenced in the autumn of 1925 about one third of the first unit had been built, although actually only one room was ready for occupation. The fitting of the remaining laboratories and workshop was, however, rapidly effected, and by the end of 1926 the whole of the available space was fully occupied, the staff then consisting of the superintendent and ten chemists, with one engineering assistant and ten members of the artisan, clerical and general staff.

The frontage to the half unit was commenced in November, 1927, and completed for occupation by Easter, 1928, and the staff was then increased gradually to its present total strength of about sixty.

Beyond a small addition for stores and workrooms completed in 1929 there has been no further extension of the building, so that after five years rather more than half of the first unit has been erected and put into commission. There has been no attempt to force the growth of this state laboratory, which is still to be regarded as being at an experimental stage.

ADMINISTRATION AND CONTROL

The work of the laboratory is conducted under the guidance of a Chemistry Research Board, which has taken over certain functions of the older Chemistry Coordinating Research Board. This board is charged with the duty of advising the department on the program of work to be undertaken at the laboratory and of exercising general supervision over its execution.

At the outset executive control was exercised by a part-time director of chemical research and a whole-time superintendent, but from 1927 to the present this responsibility has been vested in a whole-time director.

PROGRAM OF RESEARCH

At the present time the scientific and technical staffs are occupied on six specific items of research prescribed on the advice of the Chemistry Research Board, and "working parties of exploration" are detailed to these mandated researches by the director.

Now since these explorations were started at different times and in various circumstances, I propose to describe them simply in the order in which they have come under my notice. This arrangement is purely chronological, and has no bearing whatsoever on any order of merit or importance. Moreover it is essential to success in any research laboratory that each researcher should regard his own investigations as the most interesting and important in the world.

When thus arranged, the six mandated researches are as follows: synthetic resins, low temperature tar, high-pressure chemistry, corrosion of metals, chemotherapy and research on water pollution. In addition to these prescribed investigations a certain amount of general research is carried out at the discretion of the director.

SYNTHETIC RESINS

The growing importance of synthetic resins in chemical industry is gauged by the fact that the world's production of formaldehyde resins which was of the order of 9,000 tons in 1921 had increased to 13,000 tons in 1926, of which Great Britain was responsible for 16 per cent. as against 40, 24 and 8 per cent. derived respectively from the United States, Germany and France, other countries accounting for the remaining 12 per cent. Such resins are employed in the manufacture of moulding powders and electrical components. The production by industrially available means of resins of high dielectric capacity is a matter of national importance, and it was with this objective that an investigation of phenol-formaldehyde resins was begun even before the central laboratory was ready for occupation.

In May, 1925, a chemist was appointed to work at this problem in the University of Birmingham, and attention was directed to formaldehyde condensations with homologues of phenol, namely, the cresols and xyenols. Experience soon showed that *m*-cresol and 1:3:5-xylenol were especially suitable for such condensations which in the case of the former phenol were extended to a semi-works scale.

According to the nature of the catalyst employed, phenol-formaldehyde condensations yield, in general, one or other of two distinct types of resin. Alkaline catalysts lead to the production of resins of "bakelite" type, which, although originally soluble and fusible, yet possess the property of moulding under the combined effect of heat and pressure into hard insoluble and infusible products constituting by far the more important group of phenol-formaldehyde resins.

Acid catalysts favor the production of resins of "novolak" type, which, being permanently soluble and fusible, are utilized principally as shellac substitutes in lacquers and varnishes.

Alkaline Condensations: After successful small-

scale tests, alkaline condensations of formaldehyde were performed on twenty-four pounds of *m*-cresol, carried out under factory conditions in a plant of semi-works scale comprising a jacketed reaction vessel, reflux condensers, washing and storage tanks, drying and incorporating vessels and a hydraulic press with heated platens.

A systematic study of this alkaline condensation revealed the presence of several crystalline intermediates which precede the formation of resin. The latter was employed in the production of moulded articles and of laminated boards for electrical testing.

Acidic Condensations: The chemical nature of formaldehyde-phenolic resins is still a matter of speculation, but the appearance of crystalline intermediates in the early stages of acidic condensations is of interest as denoting the course of these reactions. During these researches several crystalline intermediate products were isolated for the first time.

FORMALDEHYDE-KETONE RESINS

In the foregoing formaldehyde-phenol condensations, acetone is sometimes used as a medium, but since in the presence of alkalis this solvent condenses with formaldehyde to yield resins, the chemistry of the process has been elucidated by a study of the interaction of formaldehyde and the ketones under alkaline conditions. As the homologous series is ascended the formation of resin decreases. Acetone yields mainly resin and small proportions of γ -ketobutanol, $\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{OH}$ and of the tetrahydropyrone formed by dehydration of the tetrahydric alcohol $\text{HO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CO} \cdot \text{C}(\text{CH}_2 \cdot \text{OH})_3$. Methyl ethyl ketone gives considerable proportions of the following mono- and di-hydric alcohols, $\text{CH}_3 \cdot \text{CO} \cdot \text{CH}(\text{CH}_2 \cdot \text{OH}) \cdot \text{CH}_3$ and $\text{CH}_3 \cdot \text{CO} \cdot \text{C}(\text{CH}_2 \cdot \text{OH})_2 \cdot \text{CH}_3$ with but little resin. Diethyl ketone furnishes no resin but leads to similar mono-, di- and tri-hydric alcohols.

LOW TEMPERATURE TAR

There is at the present time in this country no process of chemical industry which is more in the public eye than low temperature carbonization of coal. The matter is of supreme national importance, for the larger problems facing this mode of utilizing coal are both economic and technical and turn on the exploitation to the best advantage of the resulting products: smokeless fuel, gas, aqueous liquor and tar. Now since any marked appreciation can be expected only in the case of the last of these products it follows that processes tending to an increase in the value of the tar are of fundamental interest.

During the last five years a systematic study of the chemical constituents of low temperature tar has been in progress in the Teddington laboratory and, in our

experiments on this material, quantities of the order of 40 gallons have been handled in the semi-scale plant. The starting materials, supplied by H.M. Fuel Research Station as part of the government's scheme of scientific investigation into the utilization of our national resources of coal, consist of pedigree tars derived from coals of definite origin carbonized under carefully controlled and reproducible conditions.

It was soon found that although low temperature tar had been produced at carbonizing temperatures of about 600°, yet it could not again be heated even to comparatively low temperatures—round about 150°—without undergoing considerable alterations of a chemical nature. Accordingly, distillation processes were replaced by milder methods of extraction, and the tar was not heated above 120° until its more decomposable constituents had been removed.

A representative tar from a typical bituminous coal (Kinneil coal) was heated to 120° to remove light oils and adhering aqueous liquor, and the residue extracted by systematic use of solvents to separate it into its major constituents: neutral oils and waxes, aromatic hydrocarbons, bases, phenols and carboxylic acids. It was then noticed that each of these five main groups of products could be separated into two fractions, one portion consisting of crystallizable substances conveniently termed "crystalloids," the other portion composed of amorphous resinous materials to which the name "resinoids" was applied.

THE CRYSTALLOIDS OF TAR

Waxes and Neutral Oils: From the least volatile fractions of neutral oils, waxes are obtained melting over a considerable range of temperature, and X-ray analysis of the less fusible of these waxes has revealed the presence of hydrocarbon chains containing 26, 27 and 29 carbon atoms.

The neutral oils contain both saturated and unsaturated hydrocarbons and also oxygenated substances reacting with ferrichloric acid, HFeCl_4 .

Aromatic Hydrocarbons: Naphthalene, a characteristic major constituent of high temperature tar, is present in low temperature tar, together with β -methyl-naphthalene, but only in such small proportions that they have to be separated through their picrates.

The least volatile tar oils after removal of waxes and resins deposit on cooling a material analogous to the green grease of high temperature tar. This product consists principally of the methyl derivatives of anthracene, although a small proportion of this hydrocarbon itself may possibly be present. Oxidation of various fractions from this product leads to 2-methyl-anthraquinone, 2:6- and 2:7-dimethylantraquinone and 2:3:6-trimethylantraquinone. The proof of the

orientation of methyl groups in these anthracene derivatives has involved the synthesis of the hydrocarbons and of their quinones.²

Bases: The volatile bases of low temperature tar are mainly tertiary amines although a small amount of aniline was detected. The following bases were isolated and purified through their crystalline mercurichlorides: pyridine, *a*-picoline, 2:4- and 2:6-lutidines and symmetrical collidine; quinoline and quinaldine were isolated as picrates.

Phenols: Low temperature tars contain a high proportion of material extractable with aqueous caustic soda, but only a portion of this soluble extract consists of true phenols, the remainder is composed of non-phenolic substances which, however, dissolve in solutions of the alkaline phenolates. These non-phenolic materials are recovered from a caustic soda extract of the tar either by agitation with an organic solvent such as chloroform or more simply by saturating the alkaline extract with salt. The true phenols remaining in the alkaline solution are separated into crystalline and resinous portions by solution of the former in light petroleum. Further fractionation of the petroleum soluble phenols has led to the isolation of the following compounds: phenol, the three cresols and five of the six possible xylenols. Bacteriological examination of the phenols of low temperature tar has shown that their germicidal value increases with rise of boiling point to an optimum fraction boiling at 140–170° under 5 mm pressure. Moreover, it has been found that direct chlorination of these higher phenols of low temperature tar increases considerably their germicidal potency.

THE RESINOIDS OF TAR

With each class of crystalloid in the low temperature tar there is present a corresponding resinoid which constitutes the least volatile portion of each major fraction. These products, which are termed respectively resinenes (neutral resins), resinamines (basic resins), resinols (phenolic resins) and resinoic acids (acidic resins), are obtained as amorphous powders after extraction of the corresponding crystalloids with petroleum or other suitable solvent. These resinous tar products are promising materials for further research from both scientific and industrial view-points.

An extension of the solvent method of extraction to other varieties of tar from wood, peat, lignite and bituminous coal has revealed the presence in each tar of the four classes of resins, although in wood tars the amount of resinamines was very small. Coal tars produced by carbonization at high and at intermediate

² *Journ. Chem. Soc.*, 1929, 2203 and 2551.

temperatures show considerable variations in their resin contents.

AQUEOUS LIQUORS OF COAL CARBONIZATION

The aqueous liquors which accompany low temperature tars have been extracted systematically with organic solvents in quantities of 30 gallons at a time, and in this way phenol, *o*-cresol, catechol and its two methyl derivatives, resorcinol and quinol, have been isolated, together with a new type of resins to which the name resinolic acids has been given, as they are intermediate in chemical properties between resinols and resinic acids. Resinolic acids in the presence of ammonia are largely responsible for the dark red color of the aqueous effluents from gasworks. These aqueous liquors have also furnished on systematic extraction aniline, pyridine and α -picoline, and the series of fatty acids ranging from formic to *n*-valeric acids.

HIGH PRESSURE CHEMISTRY

During the past ten years increasing attention has been directed to the use of pressure as a means of facilitating the course of chemical reactions, and research on high pressure syntheses was started at the laboratory in 1926 on the recommendation of the Chemistry Coordinating Research Board, whose members were impressed by the possibilities revealed by the work of Patart in France and of the Badische Anilin und Soda Fabrik in Germany.

The plant required for this investigation was designed and built in the laboratory workshop, and the earliest experiments were carried out with hand compressors. Subsequently motor-driven compressors and circulators were added to the equipment. This plant was first tried out with catalysts of the Patart type (normal or basic zinc chromate) in order to gain skill and confidence in the process. It was thus found that on passing the mixed gases (1 vol. CO, 2 vols. H₂) at the rate of 30,000 vols. per hour, measured at N.T.P. over unit volume of such a catalyst at 380° and under 200 atmospheres' pressure the hourly production of methyl alcohol was about twice the volume of catalyst space.

The addition of cobalt chromate or nitrate to the foregoing zinc chromate catalyst led to an interesting development, since with the more complex catalyst ethyl alcohol and other higher alcohols made their appearance, although methyl alcohol remained the predominant product. Small amounts of aldehydes and acids were also detected. By the use of mixed cobalt catalysts containing zinc, together with chromium or manganese, the following alcohols have been obtained in addition to methyl and ethyl alcohols: *n*-propyl, *n*-butyl, *iso*-butyl and *n*-amyl alcohols and racemoid 1-

methyl-propylcarbinol CH₃·CH₂·CH(CH₃)·CH₂·OH. So far only primary alcohols have been detected. Aldehydic products have been identified as follows: formaldehyde, acetaldehyde, propaldehyde, *n*-butaldehyde and also certain aldehydals arising from the condensation of the foregoing aldehydes and alcohols. Moreover, the synthetic products contain formic, acetic, propionic and *n*-butyric acids.

The addition of even small proportions of cobalt to copper-manganese oxide catalysts (Audibert type) has a marked effect on the production of ethyl alcohol and its homologues, and a similar result is noticed on replacing the cobalt in these catalysts by iron. Traces of alkali hydroxide promote the formation of higher alcohols, and in this respect potash is more efficacious than lithia.

HELIUM FROM MONAZITE SAND

In addition to their synthetic experiments, the staff engaged on high pressure chemistry have brought to completion a research on the extraction of helium from the monazite sand conveyed to this country from Travancore for the manufacture of thorium and ceria required in the incandescent mantle industry. During this manufacture each gram of sand evolves 1 cc of helium at N.T.P., so that 100 tons of sand would discharge into the atmosphere approximately 100,000 liters of the gas. Our requirements of this raw material were entirely met through the kind assistance of the late Mr. Edmund White, formerly managing director of Messrs. Thorium, Limited.

The gas was liberated by heating the monazite at 1000° in heat-resisting steel pots in a stream of carbon dioxide, and the issuing gas was passed over cupric oxide at 500° to oxidize hydrogen and carbon monoxide. Carbon dioxide was then removed by aqueous caustic soda and the residual gas passed over metallic magnesium at 600° in order to remove nitrogen and over metallic calcium at 580° to eliminate the remaining impurities. Several hundredweights of sand were thus treated and returned to Messrs. Thorium, Limited, who found that they could still employ the heated material in their process providing that it was mixed with a certain proportion of raw sand. The purified gas containing 99.5 per cent. of helium was compressed into storage cylinders.

CORROSION RESEARCH

The researches on corrosion were originally started by a committee of the Institute of Metals in 1916, and after eight years the more scientific developments of these problems were undertaken by the Corrosion Research Committee of the Department of Scientific and Industrial Research, this work being pursued in the metallurgical department of the Royal School of

Mines until the workers concerned were transferred to Teddington at Easter, 1928.

CORROSION OF IMMERSSED METALS

Research on the corrosion of immersed metals has been concentrated on an attempt to put the theory of this phenomenon on a secure quantitative foundation. For although earlier work in this country and in the United States had furnished a qualitative explanation of corrosive action in water or in salt solutions, yet this description of the process postulated the influence of more than a dozen factors on the corrosive rates of immersed metals. Accordingly, one aim of the present research is to acquire precise information as to the interaction of these factors, and another objective is to ascertain whether the lack of reproducibility in corrosion experiments is inherent in the corrosion process itself or whether it is due to imperfect regulation of all variables. Among these factors are purity of materials: metal, water, salt and atmosphere, constancy of temperature and pressure, and freedom from mechanical agitation. Zinc of a purity of 99.99 per cent., distilled water with an electrical conductivity of 0.058 gemmhos at 20° and purified oxygen were employed, and all experiments were carried out at 25° within a temperature range of $\pm 0.02^\circ$ over long periods of time, sometimes for upwards of six months.

Measurements of oxygen absorbed, corrected for any hydrogen evolved, made at frequent intervals during the course of such experiments have enabled one to plot continuous corrosion time curves which are often sufficiently smooth and regular to be investigated mathematically.

The apparatus employed for this purpose is shown among the exhibits from the laboratory. Originally designed for zinc it is now being used extensively for work on iron and steel.

Oxygen passes through water or salt solutions to the immersed metal either by diffusion or convection, but the latter mode of transference is by far the more effective at more than very shallow depths. Convection currents may arise in a salt solution owing to four different causes: (1) temperature changes, (2) density changes produced by evaporation at the surface layer, (3) density changes produced by differences of oxygen concentration, (4) mechanical agitation. The apparatus employed for these quantitative experiments is immersed in a thermostat and corrosion occurs in a closed space within it so that the effects of temperature changes (1) and evaporation (2) are practically negligible, and special precautions are taken to prevent agitation (4). Accordingly, by removing oxygen from the neighborhood of the metal, the corrosion process produces convection currents of the third category due to changes in concentration of

oxygen. The velocity of these convection currents depends on the difference in density between the solution saturated with oxygen at the liquid surface and the solution next to the metal. Assuming that the latter solution contains very little oxygen, the velocity of convection will probably be proportional to the solubility of oxygen in the liquid, but the amount of oxygen carried by the current is also proportional to its solubility. Hence, the rate of corrosion (y) should be proportional to the square of the oxygen solubility (x), a relation which is expressible by the equation $y = kx^2$. This assumption has been verified for on plotting the observed rates of corrosion against oxygen solubility one obtains curves of parabolic form.

Hydrogen evolution due to the interaction of water or salt solution with metals such as zinc or steel is of greater importance than is generally supposed. Determinations of the hydrogen liberated during zinc corrosion have shown that a very small amount of impurity has a considerable influence on the amount of gas evolved. In N/10,000 potassium chloride measurable quantities of hydrogen are obtained from 99.99 per cent. zinc, whereas no hydrogen was detected from zinc of spectroscopic purity. The proportion of zinc corrosion due to evolution of hydrogen increases with concentration of potassium chloride, and with 2N-solutions it amounts to 17.4 per cent. of the total corrosion.

When all the foregoing factors are taken into account, successive corrosion experiments exhibit a high degree of reproducibility and the curves indicate that duplicates differ from their mean value by 1 per cent. or even less. This constancy indicates that the corrosion of zinc and allied metals is not inherently erratic, but is quite a suitable subject for physico-chemical investigations.

ATMOSPHERIC CORROSION

Investigations of various types of indoor and open-air corrosion and of protective oxide films, previously conducted under the auspices of the Atmospheric Corrosion Research Committee of the British Non-ferrous Metals Research Association, were taken over by the Department of Scientific and Industrial Research in July, 1927. This work was continued at the Royal School of Mines until April, 1928, when the corrosion section was transferred to the Chemical Research Laboratory. The more outstanding results since obtained are as follows:

Composition of Green Patina on Copper Structures: Samples of the familiar green patina on exposed copper surfaces, obtained from typical localities, town, country, marine and urban-marine, were analyzed completely. Contrary to the general belief, basic copper carbonate was found to be not the prin-

cipal but only a minor constituent of the green patina. With the exception of the product from a purely marine atmosphere in which basic copper chloride predominated, the major constituent throughout was basic copper sulphate, and excess of basic sulphate over basic carbonate was greater in the rural than in the urban samples. Where urban and marine conditions coincided, basic sulphate predominated greatly over both basic chloride and basic carbonate.

It has recently been found that these constituents of the green patina tend to assume the chemical composition of the corresponding green copper minerals. In the limits, the basic copper sulphate of corrosion coincides in composition with brochantite of which the coordination formula is $[\text{Cu}\{(\text{HO})_2\text{Cu}\}_3]\text{SO}_4$, and the basic copper chloride of corrosion with atacamite $[\text{Cu}\{(\text{HO})_2\text{Cu}\}_3]\text{Cl}_2$. Basic copper carbonate on the other hand tends to assume the composition of malachite $[\text{Cu}\{(\text{HO})_2\text{Cu}\}]\text{CO}_3$. Complete agreement with the composition required by the coordination theory has been realized in corrosion products after 70 years' exposure and upwards. After shorter periods of exposure the basicity of the product is in a lower ratio than that of the corresponding minerals.

The complete analysis of these corrosion products entailed special precautions. The carbonates were decomposed by phosphoric acid instead of hydrochloric or sulphuric acid, and any hydrogen chloride and hydrogen sulphide simultaneously set free were eliminated by *p*-nitrosodimethylaniline and copper powder respectively.

Corrosion of Magnesium Alloys: The growing use of light magnesium alloys for motor-car and aircraft work has necessitated increased attention to the corrosive properties of these metals. In 1929 a research was begun with the object of discovering improved methods of protection and of learning more about the nature of the corrosion. More than 500 different protective coatings have been produced by chemical means and tested for resistance to sea-water sprays. Of these coatings a few are sufficiently promising to warrant further study.

CHEMOTHERAPY

In 1927 a joint exploratory committee of the Department of Scientific and Industrial Research and of the Medical Research Council decided that there was need for organized research in chemotherapy, and accordingly the Medical Research Council set up a permanent committee to advise them on investigations in this field. To this committee the department has nominated three chemical members, including the director of chemical research, and facilities have been afforded by the department for a staff of three chemists to work on problems based on an agreed program.

These chemists have already prepared a considerable number of organic compounds of possible utility in chemotherapy and these are being tested systematically under arrangements made by the permanent committee. This work of national importance is a joint effort of several groups of chemists working in different laboratories, so that a wide and thorough search for greatly needed drugs and therapeutic agents is in progress.

The Teddington contribution to these researches may be classified under the two following main headings:

1. *Analogues of Bayer 205 or Fourneau 309:* Last year in his presidential address to the physiology section of the British Association in South Africa, Professor W. E. Dixon referred to the serious ravages produced in that continent by sleeping sickness (trypanosomiasis), and his admirable survey of the position from the view-point of chemotherapy renders unnecessary any further elaboration of that aspect of the problem in the present summary.

The activity of medicaments of the Bayer 205 or Fourneau 309 type may depend more on the aggregate effect of the whole molecule rather than on the presence in the molecule of any particular group or arrangement. In this, as in other cases, there are no definite laws connecting therapeutic activity and chemical structure.

Compounds have been prepared in which the terminal aminonaphthalenedisulphonic radicals have been replaced by analogous complexes derived from aminocarbazole di- and tri-sulphonic acids or from the disulphonic acids of aminofluorene and of aminofluorenone, but so far the effect of this substitution has not been encouraging. The possibility of a beneficial introduction of arsenic into the fluorene nucleus has, however, been considered, and experiment has shown that trypanocidal activity is manifested when an arsinic acid radical is present in a fluorene molecule in conjunction with an amino-group.

2. *Organic Derivatives of Arsenic and Antimony:* During many years organic arsenicals have received much attention, whereas organic antimonials have not been subjected to the same careful scrutiny, partly owing to the fact that they are more difficult to prepare in a state of purity and partly because the curative results have been less promising.

Nevertheless, since antimony in organic combination appears to possess specific trypanocidal activity and some curative action in kala-azar, experiments have been made in the Teddington laboratory on the preparation of antimony analogues of the more successful arsenicals. Tryparsamide (phenylglycine-amido-*p*-arsinic acid) is used extensively in treating trypanosomiasis, and its antimony analogue has been under

examination. In the more stable meta series, phenylglycine-amido-*m*-stibinic acid and certain allied compounds show a slight trypanocidal effect. The antimony analogue of stovarsol (3-acetyl-amino-4-hydroxy-phenyl arsinic acid) or more probably its internal dehydration product has also exhibited some therapeutic activity.

Concurrently with this study of organic antimonials further experiments have been made on organic arsenicals produced by condensing atoxyl successively with succinic anhydride, and with a base such as ammonia, methylamine, dimethylamine, piperidine or aniline. Certain of these derivatives have also exhibited a definite action on trypanosomes.

In addition to the preparation of antimonials directly applicable to therapeutic tests, our knowledge of the organic chemistry of antimony has been extended among aliphatic derivatives by the production of antimony analogues of the cacodyl group and in the aromatic series by the synthesis of cyclic antimonials analogous to the alkyl- and aryl-carbazoles.

WATER POLLUTION RESEARCH

This research originated from a joint request made to the Department of Scientific and Industrial Research by the Ministry of Health and the Ministry of Agriculture and Fisheries.

During the past two years, experiments have been in progress under the auspices of the Water Pollution Research Board on the base-exchange method of water softening. One of the objects of this work has been to determine the most satisfactory way of carrying out the process, such points having been examined as the effect of varying the rate of flow of water through the bed of base-exchange material and the quantity, concentration and time of contact of the salt solution used in regenerating this material. There are two types of base-exchange material in actual industrial use, treated minerals and synthetic products prepared by interaction of solutions of sodium aluminate and sodium silicate. It appears from the result of the Teddington experiments that with treated minerals the exchange of bases is confined to the outer surface of the particles whereas with the synthetic materials diffusion to the inner surfaces or into the mass of the gel is an important factor. This study of the base-exchange process has also been extended to the case of waters rich in magnesium.

Disintegration of the base-exchange materials and contamination of the softened waters by silica and alumina have been investigated. At the rate of flow employed normally in water softening, the silica content of the water is not increased seriously and is certainly not greater than that often encountered in untreated waters.

In addition to this practical work a report summarizing existing knowledge of the base-exchange or zeolite process for water softening has been compiled and published.

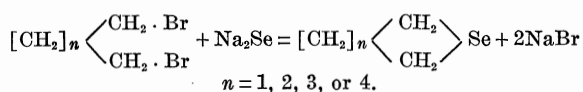
GENERAL RESEARCH

Investigation of complex aromatic hydrocarbons:

In 1926 the dyestuffs industry development committee of the Board of Trade suggested that further fundamental research was desirable on the following coal tar products: acenaphthene, carbazole, fluorene, perylene and phenanthrene. Two of these suggestions were adopted and, with the assistance of two chemists, the director, who is also a member of this statutory committee, undertook a study of acenaphthene and perylene, the work being continued until 1928. During this period considerable progress was made with the former hydrocarbon, the nitration of which was studied under anhydrous and hydrous conditions. For nitrations, in the absence of water, diacetylorthonitric acid and benzoyl nitrate were employed, the latter being a reagent discovered originally in 1906 by Professor Francis, of this university. Several new nitro derivatives were identified, and 2-aminoacenaphthene and 2-acenaphthenol (2-hydroxyacenaphthene) were prepared for the first time.

Higher Fatty Acids: In order to identify the waxes isolated from low temperature tar a parallel research was made on the synthesis of individual waxes from the higher fatty acids. Those waxes containing an even number of carbon atoms were produced electrolytically by Kolbe's classical synthesis (1849), whereas their homologues containing odd numbers of carbon atoms were prepared by a more modern process due to Grün, Ulbrich and Krezil (1926). By these complementary processes individual waxes containing 27, 30 and 34 carbon atoms were prepared for comparative purposes. This inquiry necessitated the study of several higher fatty acids, including arachidic acid, and in such cases analytical data were confirmed by X-ray analyses carried out in the National Physical Laboratory.

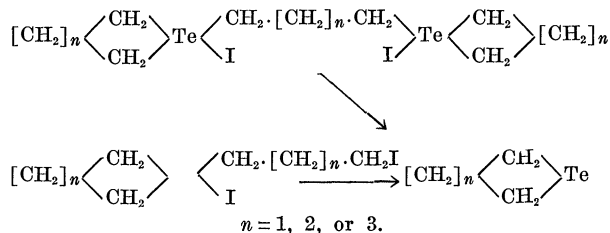
Cyclic systems containing selenium and tellurium: Considerable progress has been made in the study of heterocyclic systems containing selenium or tellurium atoms. The selenium series has been prepared by a general method, the interaction of alkylene bromides and sodium selenide in an inert atmosphere.



In this way the cyclic selenohydrocarbons with $n = 1, 2, 3$ or 4 have been obtained for the first time. The five-membered ring, cycloselenobutane (tetrahydro-

selenophen) and its next homologue, *cycloselenopentane*, are formed by the foregoing reaction with considerable facility, but the four- and seven-membered rings show signs of instability, and in their production complex solid polymerides make their appearance.

In the tellurium series the corresponding cyclic derivatives are conveniently prepared by the action of aluminium telluride on alkylene halides. This process leads to the production of cyclic systems containing quadrivalent tellurium.



From the foregoing complex telluronium iodides the cyclic tellurohydrocarbon is obtained by thermal dissociation under reduced pressure. By such means *cyclotellurobutane* and *cyclotelluropentane* have been isolated and some evidence was obtained of the existence of a four-membered ring.

Aromatic selenium and tellurium compounds: Phenol and the cresols have been condensed with selenium oxychloride when two types of seleniferous products have been distinguished, polar salt-like substances (Formula I) and non-polar selenides (Formula II).



When the cresols were condensed with basic tellurium chloride the following types were distinguished, all containing quadrivalent tellurium: $\text{HO} \cdot \text{C}_7\text{H}_6 \cdot \text{TeCl}_3$, $(\text{HOC}_7\text{H}_6)_2\text{TeCl}_2$, $(\text{HOC}_7\text{H}_6)_3\text{TeCl}$. The more soluble of such selenium and tellurium compounds have been tested on trypanosomes, but so far no evidence of activity has been discerned.

Studies in the Diphenyl Series: The *o*-xenylamine required in the synthesis of cyclic antimonials was formerly obtained in a somewhat tedious manner by the pyrolysis of diazoaminobenzene. This base has now been prepared by a method practicable on a large scale from commercially obtainable diphenyl.

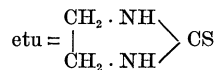
o-Xenylamine and its homologues, for example, 4':5-dimethyl-*o*-xenylamine, are convenient starting-points for the synthesis of carbazole and phenanthridine derivatives.

RESIDUAL AFFINITY AND COORDINATION

An experimental study of the effect of various coordinated addenda on the valencies of copper, silver and gold has been pursued during the past five years with the following results.

Stabilization of the cupric condition: In the absence of suitable addenda the cupric ion is unstable when in combination with less electronegative anions such as iodide, sulphite, thiosulphate, thiocyanate, selenocyanate and hypophosphite, but by coordinating this metallic ion with ethylenediamine ($\text{en} = \text{NH}_2 \cdot [\text{CH}_2]_2 \cdot \text{NH}_2$) stability is thereby conferred on the bivalent condition, and well-defined complex salts of the following types are obtained: $[\text{Cu}, 2\text{en}, 2\text{H}_2\text{O}]\text{I}_2$, $[\text{Cu}, 2\text{en}, \text{R} \cdot \text{OH}]\text{I}_2$ ($\text{R} = \text{CH}_3$ or C_2H_5), $[\text{Cu}, 2\text{en}]\text{X}$ where $\text{X} = \text{SO}_3$, S_2O_3 , S_2O_6 , S_3O_6 or S_4O_6 and $[\text{Cu}, 2\text{en}]\text{Y}_2$ where $\text{Y} = \text{CNS}$, CNSe or H_2PO_2 . Moreover, the following stable normal salts $[\text{Cu}, 2\text{en}]\text{CO}_3$, $2\text{H}_2\text{O}$, and $[\text{Cu}, 2\text{en}](\text{NO}_2)_2$ are obtainable with carbonate and nitrite radicals respectively.

Stabilization of the cuprous condition: Even more noteworthy than the preceding effect of ethylenediamine is the influence of addenda containing sulphur on the stability of the cuprous ion. Cuprous sulphate, an endothermic compound, decomposes in water with generation of heat and loss of half its copper, $\text{Cu}_2\text{SO}_4 + 5\text{Aq} = \text{CuSO}_4 + 5\text{Aq} + \text{Cu}$, but by coordinating the cuprous ion with ethylenethiocarbamide,



the univalent condition becomes stabilized even in combination with nitrate, sulphate and acetate radicals. The following colorless water-soluble salts have been identified: $[\text{Cu}, 4\text{etu}]\text{NO}_3$, $[\text{Cu}, 3\text{etu}]_2\text{SO}_4$ and $[\text{Cu}, 3\text{etu}]\text{CH}_3\text{CO}_2$.

Coordination compounds of silver: Since the silver ion is generally univalent, its coordination with ethylenethiocarbamide or other sulphur containing addenda does not involve any change of valency. It is, however, significant that $[\text{Ag}, 3\text{etu}]\text{Cl}$ is a water-soluble salt which remains colorless even after prolonged exposure to light.

A contribution to the chemistry of bivalent silver has been made by coordinating its ion with α - α' -dipyridyl (dipy), and the following colored salts have been isolated $[\text{Ag}, 2\text{dipy}]\text{S}_2\text{O}_8$ (chocolate brown), $[\text{Ag}, 2\text{dipy}](\text{HSO}_4)_2$ (dark brown plates) and $[\text{Ag}, 3\text{dipy}](\text{ClO}_3)_2$ crystallizing in well-defined, lustrous, black, acicular prisms.

Stabilization of the aurous condition: Coordination of gold salts with ethylenethiocarbamide has the same effect as with copper compounds. The fundamental univalency of the metallic ion becomes stabilized so that the following complex aurous salts have been identified: $[\text{Au}, 2\text{etu}]_2\text{SO}_4$, $2\text{H}_2\text{O}$, $[\text{Au}, 2\text{etu}]\text{NO}_3$, $[\text{Au}, 2\text{etu}]\text{Cl}$, H_2O and $[\text{Au}, 2\text{etu}]\text{Br}$, H_2O . These compounds are colorless and dissolve in water to prac-

tically neutral solutions (P_{α} value about 6.2). Conductivity experiments indicate that in dilute aqueous solutions these complex salts are highly ionized so that the complex radical $[\text{Au}, 2 \text{ etu}]^1$ plays the part of a compound alkali ion. The bromide of this series was mentioned last year by Professor W. E. Dixon (*loc. cit.*) as being a compound which had the effect of delaying death when administered to animals infected with bovine tuberculosis.

CHEMICAL ENGINEERING

The mainstay of the foregoing investigations are the well-equipped workshops manned by five skilled artisans who are engaged on the production and maintenance of the appliances and plant required in the various research programs. Appliances for high-pressure chemistry are a speciality of the laboratory workshops, and such plant includes bombs and pre-heaters for flow-through experiments with gaseous reagents, and autoclaves of various types for reactions with gases, liquids and solids. The researches on tar products call for automatic extractors, filter plant and stills operating under either ordinary or diminished pressures.

THE STATE LABORATORY AND THE SCIENTIFIC PUBLIC

The twofold primary aim of any state research laboratory should be the collection and dispersal of scientific knowledge and information. For the former function of collection and discovery of new knowledge the exploring parties foreseen by Professor Vernon Harcourt should supply an adequate means providing that each group proceeds under enlightened and inspired leadership. But for the complementary function of dispersal of information a chemical laboratory must depend largely on such well-established media of publication as the journals of the leading chemical societies. The greater part of the published research of the Teddington laboratory has appeared in the journals of the Chemical Society and of the Society of Chemical Industry, although a certain proportion has been published in the Proceedings of the Royal Society, *Journal of the Institute of Metals*, and Proceedings of the Institution of Chemical Engineers. Grateful recognition should be recorded for the generous aid afforded by all these learned societies, and special thanks are due to the first two mentioned. It is my personal opinion that this mode of dispersing chemical knowledge should have priority over its publication in official governmental reports. First, because in this way the information radiates more rapidly to a wider public; thus each of the two chemical journals just mentioned has more than 5,000 registered readers. Secondly, because this form of publication is frequently preceded by a reading and

discussion of the subject-matter at a scientific meeting, and lastly because the financial circumstances of the learned societies compel them to impose a limit on the length of communications which is conducive to brevity and conciseness.

RELATIONS WITH OTHER SCIENTIFIC INSTITUTIONS

Apart from substances of therapeutic interest prepared for the committee on chemotherapy, numerous other research materials have been distributed to colleagues in the universities and research institutions. Compressed helium and carbon monoxide have been rendered available for scientific workers requiring these gases. Organic derivatives of tellurium have been lent to the Cambridge University Chemical Laboratory for the purpose of physico-chemical measurements, and to the Birkbeck College for the demonstration of the parachor of this element. Compounds of special chemical interest have been supplied to the Davy Faraday Laboratory and to the National Physical Laboratory for the X-ray study of their crystal structure. It is a pleasant duty to refer to the aid received from the Government Laboratory in respect of microanalyses and in connection with the work on synthetic resins.

Reference has already been made to the close collaboration of the laboratory with H.M. Fuel Research Station in regard to the products of coal carbonization. Certain preparations from low temperature tar have been submitted to the Cotton and Woollen Research Associations, for examination in connection with the chemical treatment of textile fibers.

RELATIONS WITH CHEMICAL INDUSTRY

The associations of the laboratory with chemical industry have always been cordial and are daily becoming increasingly intimate. Prominent industrialists either individually or in their corporate capacity as members of the Association of British Chemical Manufacturers and allied organizations have visited the laboratory and sometimes repeatedly.

Arising out of these visits and informal conferences, more than a hundred samples of the research products of the laboratory have been distributed to interested enquirers.

Members of the scientific staff participate in the work of the Committee for the Standardization of Tar Products Tests, the Bureau of Chemical Abstracts, the Corrosion Committee of the Iron and Steel Institute, and the Council and various Committees of the Society of Chemical Industry.

Although the laboratory is not a teaching institution in the academic sense of the term, yet facilities have been afforded for collaboration in research to chemists in training of approved qualifications. The

two leading metropolitan gas companies have seconded to the laboratory for this purpose junior members of their scientific staffs who have worked at Teddington for periods ranging from six to eighteen months. The subjects so far selected for this collaboration have been high-pressure chemistry and low temperature tar.

In the foregoing description of the activities of the new laboratory I have endeavored to speak as historian rather than as advocate, but if any justification is to be included I would take as the two leading points of my case: First, the scientific and industrial importance of the researches completed and in progress; secondly, the significant fact that of the sixteen members of the laboratory staff who have resigned during the five years, fourteen have gone into chemical industry to occupy positions of considerable importance and responsibility. The appreciation of chemical talent is a valuable function of this state laboratory.

ANTICIPATIONS AND CURRENT TENDENCIES

Those who feel sufficiently interested in the realization of Professor Vernon Harcourt's vision should not fail to visit the exhibit of laboratory products now on view in an adjacent room, for these specimens, diagrams, models and photographs furnish a record of the researches of this youthful organization which is far more realistic and appealing than any words of mine can be.

Certain of these investigations have an immediate practical objective; others represent the long view. It is, however, impossible to draw a definite distinction between these contrasted types. The aim of a state laboratory should rather be to encourage a judicious blend of the two.

The chemical preparations now selected for exhibition as representing the work of the first five years are only the more distinctive specimens of a much larger collection which is continually being accumulated and classified. In a similar orderly manner chemical knowledge is being collected and systematized in the files and card-indexes compiled by members of each exploring party. So soon as any particular research is sufficiently complete it is contributed to the appropriate learned society. Occasionally publication takes the form of patent specifications. By such concerted efforts the laboratory must come to be recognized as a storehouse of chemical information at least for those branches of the science which are included in the scope of its researches.

Is it desirable that this scope should be extended, and if so in what directions? This is not the occasion to discuss matters of departmental policy, but, in my present capacity, I may, like my predecessor of fifty-five years ago, indulge in anticipations of how

future developments might be of advantage to chemical science in general and to British chemistry in particular.

INORGANIC AND MINERAL CHEMISTRY

An eminent authority has recently enquired what has become of inorganic chemistry and this question is frequently repeated. The present answer is that, so far as this country is concerned, the subject is no longer investigated systematically. British chemists are now for the most part content to leave this work of exploration to their contemporaries in other lands. Yet the British Empire is endowed with mineral resources to an extent unsurpassed by any other nation or empire under the sun. It can scarcely be contended that in this respect we are rendering an adequate account of our stewardship. Although there are a few meritorious exceptions, one may say broadly that there is no sustained British attempt to study the rare earths, the less common alkalis, or the metals of the platinum group. Such chemical curiosities as beryllium, gallium, germanium, indium and thallium rarely excite the scientific interest of our investigators. Yet the chemical study of the less common elements, and especially of those grouped under the disparaging term of "minor metals," is a matter of considerable scientific importance and one which sooner rather than later is likely to yield results of industrial value. If proof of this statement is needed, reference may be made to the inert gases which were first noticed in 1894 and subsequently found by Ramsay and Travers to be five in number. To-day three of these gases are employed industrially.

I have already mentioned low temperature tar which is literally a burning question. The great German combination of chemical factories—the Interessen Gemeinschaft—have recently filed patents describing the catalytic effect of molybdic acid on the hydrogenation under pressure of this intractable material. They claim a clear volatile product obtainable in good yield and suitable for motor fuel. Further investigation shows that this beneficial catalytic influence is peculiar to molybdenum compounds and is not possessed by analogous compounds of the other metals of the sixth periodic family. It certainly pays to study chemically the idiosyncrasies of the rarer elements and their derivatives.

THE ORGANIC CHEMISTRY OF VITAL PRODUCTS

At the Bristol meeting of 1898, Professor F. R. Japp's presidential address to this section dealt with the subjects of stereochemistry and vitalism. He called attention to nature's method of preparing single optically active substances, and referred to the insufficiency of the mechanical explanation of vitalistic phenomena.

Considerable advances have since been made in our knowledge of the fundamental process of photosynthesis, notably as the result of suggestive discoveries by Professor Baly and his collaborators, but nevertheless we still have much to learn from nature in regard to the synthesis of carbon compounds. This study of the products of the vital activities of animal and vegetable organisms was the original province of organic chemistry, and to this circumstance the science owes its distinctive name. During the last eighty years, however, organic chemists have extended the scope of enquiry to many substances which are produced not as the result of vital forces, but through the agency of the laboratory arts.

For instance, the organometallic compounds, which have no counterparts in nature, have received intensive study because of their influence on the development of modern chemical theory, their practical application in many operations of organic synthesis and their utilization as drugs, weapons of chemical warfare and antidetonants. No objection can be urged against the continued investigation of such important artificial products providing that naturally occurring organic materials are not overlooked.

Professor Japp's address supplies the philosophic reason for a closer study of the products of vital activity, and at present other more mundane considerations may be adduced in support of such researches.

Political and economic forces are bringing into prominence the urgency for a mutually advantageous interchange of commodities between the constituent nations and colonies of the British Empire, and in this pooling of natural resources organic chemistry must play an essential part. Many of the natural products of the dominions and dependencies are in need of systematic chemical study.

Animal and vegetable fats have been mentioned by an investigator in that field as constituting a neglected chapter of organic chemistry, but the phrase is at least as applicable to many other groups of organic substances, for example: the essential oils, the natural gums and resins, and the numerous products of fermentation processes.

By catalytic reductions, involving high temperatures and pressures, one obtains from the oxides of carbon many members of the homologous series of alcohols, aldehydes, fatty acids and esters. Plant life accomplishes similar results under ordinary atmospheric conditions. A comparative study of these two dissimilar sets of processes is clearly demanded.

The importance of imparting to organic chemistry an increasingly biological bias has been illustrated in a convincing manner by my immediate predecessor, Professor Barger, so that anything more than a passing reference to this desirable tendency is hardly required of me. Perhaps, however, I should add that in stressing the need of more systematic research in inorganic and mineral chemistry and in the organic chemistry of vital products, I am convinced that the best results will only be attained if the problems are attacked with the newest weapons which the armory of modern physics can provide.

The primary object of such investigations is the collection of accurate chemical information, but the workers in these two great fields should be stimulated in every possible way to keep a shrewd lookout for any practical applications of their scientific knowledge. When viewed from this standpoint it will be realized that a state experiment in chemical research such as I have described provides competent and enterprising investigators with favorable opportunities for developing their inventive talent in fundamental work of national value and importance.

OBITUARY

RECENT DEATHS

THE death is announced of Dr. W. Howard For-syth, assistant professor of dairy husbandry at the Connecticut Agricultural College.

DR. EDWARD L. SPITZER, an attending physician and former president of the Jewish Memorial Hospital in New York, died on September 18 at the age of sixty years.

FREDERICK D. PRATT, engineer of the General Electric Company, who on March 8 sailed from New York with a group of other engineers to supervise the establishing of electrical plants for the Soviet Government in Russia, died on September 20 at Moscow.

CARL A. MEISSNER, metallurgical engineer, who has for twenty-five years been connected with the U. S. Steel Corporation, died on October 13, at the age of seventy-one years.

DR. LEWIS EVANS, collector of and writer on old scientific instruments, founder of the Lewis Evans Collection of Scientific Instruments in the Old Ashmolean Building at the University of Oxford, died on September 25, aged seventy-seven years.

HERBERT E. SOPER, statistician of the School of Hygiene and Tropical Medicine, London, died on September 10.

M. JEAN BRUNHES, who was for sixteen years pro-