

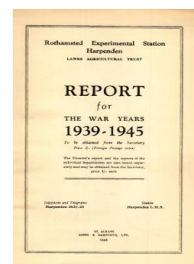
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## Report for 1939-45

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## Chemistry Department

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## DEPARTMENT OF CHEMISTRY

By E. M. CROWTHER

### GENERAL

During the years 1938 to 1941 several members of the Department were occupied with planning and equipping the new laboratories. The old ones were gutted and rebuilt to form a single compact unit with the new extension and part of the adjoining range of new glasshouses. The extra space made it possible to assign special rooms to spectroscopy, X-rays, micro-chemistry and the varied preparation and analytical work required for a rapidly expanding programme of field experiments. Some of the new laboratories could not be fully equipped during the war and were therefore used by other bodies, the A.R.C. Unit on Soil Metabolism and the British Leather Manufacturers' Research Association.

From 1940 onwards the programme of the Department was devoted increasingly to special investigations for the Agricultural Research Council, the Ministry of Agriculture and the Ministry of Supply, and to advisory work in connection with food-production problems. Much of this work was done in collaboration with other research institutes and the agricultural advisory service. It amounted in fact to what has become known as "operational research" or the collection of objective information by which current policies can be judged and improved. An early example of this type of investigation was the joint report from the Chemistry and Statistical Departments on the basis of a war-time fertiliser policy, described on page 262 of this Report. Much of this war-time work has not yet reached the stage at which results of more permanent and general interest can be worked up and published.

A great deal of the work has necessarily been carried out by teams with each individual specialising in one aspect. Those responsible for the main sections have been :—

R. G. Warren	Analyses and general supervision.
H. V. Garner	Field experiments away from Rothamsted.
G. Nagelschmidt	X-rays and mineralogy, and phosphate fertilisers in pot and laboratory experiments.
J. B. Hale	Spectrographic analyses.
G. W. Cooke	Soil phosphates and field experiments on phosphate fertilisers.
S. G. Heintze	Soil manganese and laboratory tests on availability of nitrogen fertilisers.
A. H. Bunting	Field experiments on organic manures and composts.
N. S. Bamji	Nitrification and fermentation tests.
E. H. Cooke	Pot experiments.
B. Benzian	Records and correlation of data.
H. Nixon	Phosphate and fluorine analyses.
Others who have joined the Department more recently are :	
W. E. Chambers	Spectrographic analyses of wheat straws from Broadbalk plots.
J. M. Bremner	Soil organic matter.
P. W. Arnold	Basic calcium-phosphate equilibria.

### SOIL-FERTILITY PROBLEMS

During the war the laboratory work has been very closely linked with field and pot investigations. Nearly all of the field experiments provide soil, manure and crop samples which have been examined

partly to supplement the yield data by bringing out changes in crop quality and recovery of added nutrients, and partly to test the validity of laboratory methods in advisory work against the actual behaviour of field crops under a variety of soil conditions. It is anticipated that soil samples from many of the field experiments will be used for several years to come in checking and developing improved methods for soil analyses that may emerge from future laboratory investigations on more theoretical points.

The largest series of comparable soil samples from fertiliser experiments is provided by those on sugar beet carried out annually since 1933 for the Committee on Sugar Beet Education and Research, in collaboration with the agriculturists of the 17 sugar-beet factories in Britain. Several alternative methods for estimating readily soluble phosphate and potash (and some other elements) have been compared with the actual crop responses to various fertiliser combinations over a long period and a wide variety of soil conditions. It has become possible to define more closely the range of conditions with which current methods and some alternative ones may be used with fair confidence on soils of average fertility. The following figures illustrate the value of the citric-acid method which is widely used in advisory work. They give the average responses to standard dressings of superphosphate or muriate of potash in soils grouped by the results of analyses on samples taken before the manures were applied (the tests on phosphate are limited to non-calcareous soils and the responses to potash are in the presence of basal dressings of sulphate of ammonia).

*Average responses in cwt. of extra sugar per acre in soils grouped by citric-acid analyses*

$P_2O_5$ in soil, mg. %	No. of centres	Mean response to superphosphate	$K_2O$ in soil mg. %	No. of centres	Mean response to muriate of potash
to 8	6	8.2	to 8	112	4.4
8.1—16	31	3.1	8.1—12	57	2.2
16.1—32	72	1.5	12.1—16	22	0.3
32.1—64	62	1.1	Over 16	20	0.0
Over 64	13	0.4			

There would clearly be a great economy if the practical manuring of such soils could be adjusted more closely to the indications from soil analyses. There is, however, room for great improvement in the analytical methods themselves, for discrepancies between expectation and performance are not uncommon. Some improvements are possible by recognising the limitations in the actual methods. Thus, for phosphate the citric-acid method is unsatisfactory for very acid soils and fairly good for calcareous soils, provided extra acid is used, whereas leaching with acetic acid is good for acid soils but not for highly calcareous ones. When such limitations are recognised there is a wide choice of alternative methods, some of which can be carried out quite rapidly, provided the technique of extraction and analysis is most rigidly standardised. Rough uncritical soil analyses may be most seriously misleading.

Pot cultures provide a useful but limited intermediate stage between laboratory and field experiments. Much time has been devoted to developing a rapid small-scale pot test for available nutri-

ents, especially phosphates, in soils, fertilisers and manures. This test proved particularly serviceable for various war-time fertilisers for which there was little or no previous experience in this country. One such class of materials was examined quickly and repeatedly as the scale of production expanded from the first few grams of a laboratory preparation through two stages of pilot plant to full commercial production. The problem of maintaining sufficiently constant moisture contents in small pots was solved by allowing glass pots to stand in saucers kept partly filled with water and using a mixture of soil with coarsely crushed flint to provide sufficient aeration. Radishes were generally used as the test crop, and after about four weeks were weighed, dried and ashed for phosphate analyses to determine the total uptake.

The small-pot technique has been used to estimate the available-nutrient contents of soils by measuring responses to added nutrients. It has not so far proved satisfactory for investigations on the difficult problem of phosphate fixation, perhaps because it fails to allow sufficiently intimate reactions between soil and fertiliser or because the soils remain continuously moist. Experiments on phosphate fixation are therefore being made with undiluted soil in larger pots testing a variety of phosphates in two contrasted soils by a series of successive crops of rape or cuttings of perennial ryegrass.

In laboratory studies on the availability of soil phosphates and the fate of added phosphates attempts have been made (13 to 17) to fractionate soil phosphate by successive extractions with such reagents as a weak acid, an alkali (followed by a differentiation between organic and inorganic forms) and a strong acid. This gives at least roughly a distinction between calcium phosphates, organic phosphates, iron phosphates and apatites. Extractions by dilute acids are sometimes complicated by secondary reactions by which dissolved phosphate recombines with or is absorbed by other soil constituents. Attempts have been made with some success to limit the extent of this disturbance by adding such materials as 8-hydroxyquinoline or selenious acid to the acids used in extracting the calcium phosphate.

Calcareous soils present special problems in using phosphate fertilisers and in assessing phosphate requirements. Soluble phosphates may react with calcium carbonate. Limestones or chalk may contain appreciable quantities of phosphate not immediately available to plants and yet brought into solution by excess acid in laboratory tests. It has recently been found (12) that the remaining chalk fragments in some of the Broadbalk soils receiving superphosphate contain appreciable amounts of fluorapatite, formed mainly during the last 50 years when the superphosphate has been made from mineral phosphate containing fluorine. Such a formation of fluorapatite in calcareous soils has long been expected on theoretical grounds, but this is the first direct experimental proof of its actual occurrence. One practical indication from studying the reactions likely to occur in calcareous soils is that soluble phosphates might well be given in small annual dressings, where possible close to the seed, and not in occasional heavy dressings intended to build up reserves.

At the other end of the scale of reaction attempts have been made to see what amounts of available phosphate are mobilised by liming very acid soils. These appear to be comparable with those in ordinary fertiliser dressings for land which has been manured in the past, but small for reclaimed waste land.

#### ORGANIC MANURES

The general question of the part played by soil organic matter and bulky organic manures in the maintenance of soil fertility has remained in the forefront of our programme (2, 3, 35). During the war special attention was given to using both farm and town wastes to best advantage. The results of the experiments at Rothamsted are briefly summarised in the report on "Field Experiments on the Rothamsted Farm."

The experimental batches of farmyard manure made in bullock boxes and open yards were analysed to follow the fate of the nutrients, and the crops grown with them were also analysed to determine the ultimate recovery of the plant foods from the original feeding stuffs. All the materials used in the Rothamsted potato experiments and in a number of the other field experiments were tested in pot experiments in collaboration with the Botany Department and in laboratory nitrification and fermentation tests. Apart from supplying plant foods the farmyard manures and other fibrous manures have a definite physical effect which often appears to depend on maintaining a spongy open structure in the vicinity of the potato tubers.

Throughout the war a steadily increasing number of field experiments was carried out in different parts of the country under the auspices of a Conference of the Agricultural Research Council and often in collaboration with members of the Institute of Sewage Purification (4, 5, 6, 39). Before the agricultural quality of town refuse necessarily deteriorated with improving salvage, a number of experiments had shown that its manurial value was too low to justify appreciable expenditure on transport and application. Sewage sludges used alone or in composts with straw offered much more promise, essentially as sources of slowly available nitrogen and phosphate. Their poverty in potash must be recognised if seriously unbalanced manuring is to be avoided. The availability of the phosphate varied widely, low values commonly occurring in sewage sludges with large amounts of iron and other heavy metals from industrial wastes. Digested sludges have not only better physical conditions, but more readily available nitrogen than raw sludges. Shed-dried ground sludges are naturally more convenient to handle and are richer in immediately available nitrogen. Some of them have given poor results when applied in potato bouts, and it is not yet clear whether this is due to toxic elements or to providing too high local concentrations of ammonia near the sets. In most of the experiments the amounts of available nitrogen and phosphate accounted for the observed benefit: indications of physical improvements were obtained only after repeated heavy dressings. Composts of sewage sludge and straw have shown additional advantages which must be ascribed to physical improvements in the soil and to the supply of useful amounts of potash. In 1945 this range of field

experiment was extended so as to test sludge-straw composts and straw ploughed-in with sulphate of ammonia over a wide range of soils, including some thin soils over chalk, so as to provide extreme contrasts with the older Rothamsted experiments.

Most of the experiments on bulky organic manures have been of a complex design testing the various combinations of N, P, K fertilisers in conjunction with each organic manure and also without organic manure. Many of the experiments have been on market-garden soils for which additional supplies of organic manures are so much needed.

An important section of this work has been the laboratory study of the content and availability of the nutrients, especially nitrogen, in different kinds of farmyard and other bulky manures. A large mass of analytical data has shown the average composition and range of variation of bulky organic manures produced under different conditions. The field experiments have brought out several instances in which practical judgments of the stage of rotting and manurial value of farmyard manures differed markedly from their performances in the field. The phosphate and potash contents frequently provided the clue. Some indication of the availability of nitrogen and the stage of rotting or maturity was obtained from a new fermentation test based on the effects of added cellulose or ammonium nitrate on the carbon-dioxide production under controlled conditions.

#### FERTILISERS

Slowly acting nitrogen fertilisers such as hoof-and-horn were difficult to obtain during the war and various alternatives were investigated. One very slowly acting but none the less active material was found in formalised casein, a plastic waste. This decomposes in the soil much more slowly than hoof-and-horn of normal grist, and for this reason its value was not at first realised in practice. It should have merit as a basal dressing where a succession of crops is to be taken. It serves to provide an interesting contrast with the more rapidly available forms, such as dried blood and fine hoof meal, in trials on rate of action of nitrogenous fertilisers.

Ground leather waste has negligible value as a fertiliser. Our experiments have shown that some of the commercially treated leather wastes prepared by acid digestion have only a modest fraction of their nitrogen in a rapidly available form and very little in slowly available forms. In collaboration with the British Leather Manufacturers' Research Association attempts were therefore made to find other ways of converting waste leather into a useful fertiliser by detanning vegetable-tanned leather to free the hide protein. It was shown by laboratory tests, by pot-culture experiments in collaboration with the Botany Department and by field experiments on market-garden crops that the product was comparable in activity with dried blood or crushed hoof. Unfortunately it did not prove possible during the war to put the process into production on the commercial scale.

The choice of phosphate fertilisers is rapidly being widened as the result of promising new developments in chemical engineering. New concentrated soluble phosphates, triple superphosphate and a

granulated form of ammonium phosphate proved popular during the war. An entirely new range of insoluble but highly available phosphates is steadily being developed, especially in the United States. Under some conditions considerable economy can be effected by using relatively cheap mineral phosphates in place of more elaborately treated products. There are still great possibilities in improving the present low efficiency of phosphate fertilisers by adjusting the amounts, forms and methods of application more closely to the actual conditions of crop, soil, climate and farming system.

Some of these problems became urgent several times during the war when it seemed possible that one or both of the two imported ingredients of superphosphate—mineral phosphate and pyrites—might become scarce. Had sulphuric acid been short, it would have been necessary either to use mineral phosphates directly on suitable land or to convert them into more readily available fertilisers in some other way. Good results were obtained in experiments with Curacao mineral phosphate, a very hard material containing unusually little fluorine. Fortunately it was found unnecessary to grind it very finely. Both Curacao and Gafsa mineral phosphates gave excellent results for swedes on acid land in wet districts, especially in soils with very little phosphate soluble in dilute acetic acid. A novel American phosphate—calcium metaphosphate—with the equivalent of over 60 per cent.  $P_2O_5$  acted very slowly but appeared to be well suited for leys and other crops occupying the land for long periods.

In collaboration with the Building Research Station of the D.S.I.R., investigations were carried out for the Ministry of Supply on preparing a readily available phosphate by sintering mineral phosphate with soda ash and sand in the presence of steam. Materials with high citric solubilities were shown to give good results in pot and field experiments. The product, which has been produced successfully on the full technical scale under the name "Silico-phosphate," compared very favourably with superphosphate containing equal amounts of phosphate for swedes and re-seeding in the wetter areas, but not for potatoes and cereals in drier ones.

Several series of laboratory, pot and field experiments showed that there was no reason under wartime conditions to vary our earlier conclusion that the citric-acid test provides a sound indication of the amounts of available phosphate in different kinds of basic slag. Those with little citric-soluble phosphoric acid should not be regarded as phosphate fertilisers, but as liming materials containing a little available phosphate. They may prove useful in heavy dressings for re-seeding on very acid soils, but should not be used far from the works.

In a few field and pot experiments self-slaking or falling slags from blast furnaces and certain special steel furnaces proved almost as effective as limestone, and in addition increased the availability to crops of phosphates in the soil or added fertilisers.

Reverted mixtures of superphosphate and such basic materials as lime, basic slag and serpentine gave results closely similar to those from superphosphate supplying the same amount of phosphate. This result suggests that all the merits claimed for serpentine-superphosphate in New Zealand are not likely to be generally

obtained in Britain. It also adds one more piece of evidence for the view that the time has arrived for revising the methods adopted in the Fertiliser and Feeding Stuffs Act Regulations for expressing the solubility of phosphate fertilisers. The water-solubility test originally designed for distinguishing between good and bad samples of superphosphate can have little general validity when all superphosphates and ammonium phosphates are well made and when many other phosphate fertilisers containing little or no water-soluble phosphate are known to be as effective as superphosphate.

Combined fertiliser-grain drills imported during the war have demonstrated their value on many farms. Field trials on cereals carried out by Technical Development Sub-committees for an A.R.C. Conference showed that on the average  $1\frac{1}{2}$  cwt. of superphosphate per acre drilled with the seed were as good as 3 cwt. broadcast (10). Trials were made at Rothamsted and a few other Hertfordshire farms during 1945 on four different methods of applying a mixed fertiliser for potatoes, using a special experimental drill built by the National Institute of Agricultural Engineering. Apart from a slight and doubtful superiority at low rates of dressing of fertilisers placed in contact with the sets, there were negligible differences between the methods used.

#### FERTILISER REQUIREMENTS OF CROPS

##### *Sugar beet*

The standard series of sugar-beet experiments carried out since 1934 has served to establish the general requirements of this crop for average soils and seasons and to show how special soil needs can be estimated by soil analyses (33). The average responses in 268 experiments on commercial farms, averaging over all combinations of other fertilisers were :—

	Washed roots Tons per acre	Tops Tons per acre	Sugar %	Cwt. sugar per acre
0.8 cwt. N per acre as sulphate of ammonia ..	1.32	2.86	-0.40	3.8
1.0 cwt. P <sub>2</sub> O <sub>5</sub> per acre as superphosphate ..	0.45	0.32	0.01	1.6
1.2 cwt. K <sub>2</sub> O per acre as muriate of potash ..	0.44	0.26	0.22	2.0

The main need is for ample nitrogen with only moderate amounts of phosphate and potash.

The responses to nitrogen and potash together were greater than the sum of the separate responses, but those to superphosphate did not depend on the presence or absence of other fertilisers.

	Cwt. sugar per acre
Response to 4 cwt. sulphate of ammonia per acre ..	3.1
Response to 2 cwt. muriate of potash per acre ..	1.2
Response to both together .. .. .	5.8



The standard series of experiments brought out marked seasonal differences in the behaviour and composition of the sugar-beet crop. Thus, the dry summer and wet autumn of 1944 were particularly unfavourable for sulphate of ammonia, whereas in the wet summers and dry autumns of 1936 and 1941 sulphate of ammonia gave particularly large average responses.

From 1940 onwards the design was modified to test in addition the effect of 5 cwt. of agricultural salt per acre (8, 9, 36, 43). The average results for the individual years and for the main classes of soils are summarised below for plots receiving nitrogen at the rate of 4 cwt. of sulphate of ammonia per acre. On the average of over 100 experiments 5 cwt. of salt gave 5.2 cwt. of additional sugar, whereas 2 cwt. of muriate of potash gave only 2.8 cwt. of additional sugar. Even where 2 cwt. of muriate of potash were given there was a further response of 2.6 cwt. of sugar for 5 cwt. of salt, but where salt was given there was a negligible additional response to potash.

*Cwt. additional sugar per acre on sugar-beet plots receiving sulphate of ammonia*

<i>Average for all soils (except fens)</i>	No. of trials	From muriate of potash alone	From salt alone	From muriate of potash and salt together
1940	24	3.5	5.4	6.1
1941	22	3.2	4.8	5.2
1942	21	3.4	5.0	5.8
1943	18	1.1	4.4	4.4
1944	18	2.4	6.4	5.2
Five years	103	2.8	5.2	5.4
<i>Average for five years</i>				
Coarse sands	20	2.8	5.4	6.4
Fine sands	19	4.7	5.7	6.5
Light loams	37	2.3	4.7	4.4
Heavy soils	27	2.2	5.3	5.2
Fen soils	9	2.4	2.3	3.8

Systematic analyses of samples of sugar-beet tops and roots showed that the larger crops grown with salt contained no more potash than those without salt. Considerable amounts of sodium are taken up from the salt, but only a very small amount of the sodium reaches the roots. This work has shown that sodium must be regarded as a true plant food for sugar beet (and mangolds), and that salt does not act by drawing on reserves of potash in the soil. Sugar-beet growers should use salt much more generally, even when supplies of potash are easier to obtain.

Special series of experiments have shown that the salt may be applied at any convenient time up to a week or two before sowing, even before the winter ploughing. Although the soil structure may be temporarily damaged by heavy rain after applying salt, there has been no evidence of any bad effect on the crop or any cumulative effect on the soil, even where, as in one Rothamsted experiment, salt is applied every year.

Borax was included in the series of experiments from 1945. In that season it had negligible effects.

#### *Flax*

In collaboration with H.M. Flax Establishment, Flitcham Abbey, Norfolk, and Advisory Chemists, a series of field experiments was carried out each year from 1940 to 1945 to test the effect of nitrogen, phosphate and potash fertilisers on the yield of processed flax. On the whole the crop proved relatively unresponsive. Even where the field weights of the crop showed responses to fertilisers the benefit often disappeared during processing. There is no justification for manuring flax differently from barley. Phosphate and potash are needed only on soils deficient in these plant foods.

#### *Rubber*

Some of the results of manurial experiments on young rubber plantations conducted since 1933 by Messrs. Dunlop Plantations in Malaya, in collaboration with Rothamsted, have been published (11). Mineral phosphate had spectacular effects on early growth, the trees reaching the tapping stage from six months to two years earlier, with a corresponding saving in the initial costs of getting an estate into bearing.

#### *Oil palm*

A somewhat similar series of manurial experiments was laid out on the oil-palm estates of Messrs. Unilever in Nigeria, and the Belgian Congo, from 1940 onwards, the plans being outlined and some of the sites selected during Dr. E. M. Crowther's tour in West Africa in 1938-39 (1). So far the trees already in bearing have given only modest responses, though this may have been due in part to war-time interruption in the manuring programme. Results for the young trees are not yet available.

#### *Forest trees in nurseries*

Under a programme of work drawn up by a Forestry Commission Committee on Nutritional Problems in Forest Nurseries, manurial experiments were laid out in 1945 on four nurseries and in boxes at Rothamsted. All the nursery experiments included Sitka spruce and ash, with Scots pine and birch in addition at one nursery. The preliminary indications from shoot growth in the first season are that much of the benefit from composts, especially in wasteland or woodland nurseries, can be ascribed to the plant nutrients they supply, and that some of the differences between nurseries can be interpreted in terms of the nutrient status of their soils as shown by soil analysis and by the responses to fertilisers. The indications are necessarily tentative as the experiments are to be continued through the lining-out stage and new experiments started in seedbeds in 1946. In box experiments with a slightly calcareous old nursery soil, on which Sitka spruce had consistently failed, large plants were grown on soil which had been acidified, the heights regularly following the amounts of acid added.

#### SOIL MANGANESE

Manganese deficiencies in such crops as oats, potatoes and sugar beet are fairly easily recognised by leaf symptoms and by the response of the crop to sprays containing manganese sulphate.

The deficiencies are known to be associated with certain types of soil, especially heavily limed soils rich in organic matter, but there is no satisfactory method of estimating available manganese in the soil. A variety of different soils including a series of pairs of samples representing contrasted crops in the same field was examined for different forms of manganese. The exchangeable manganese generally increased with soil acidity, but, except in some very acid soils, formed only a small fraction of the readily reducible manganese. The exchangeable manganese tended to be low where sensitive crops showed deficiency symptoms, but it was sometimes low where the crops were healthy. There was no evidence to support the view that the readily reducible manganese was a better guide than the exchangeable manganese. In some contrasted pairs of soils, mainly fens, with similar reactions and contents of exchangeable and total manganese, deficiency symptoms were shown on the soil with the higher content of nitrifiable nitrogen. Other evidence from field and pot experiments supported the view that high available nitrogen favours manganese-deficiency symptoms.

Manganese salts added to neutral or alkaline soils rapidly pass over from the exchangeable to other forms which are readily reducible and soluble in solutions of sodium hexametaphosphate. In the course of an attempt to fractionate organically bound manganese it was found that a large proportion of the soil manganese dissolves as a complex ion in sodium hexametaphosphate. When the staff of the A.R.C. Unit of Soil Metabolism had established the importance of trivalent manganese in the soil, a critical examination was undertaken in collaboration with them on methods for determining the various forms of manganese in soils. Improved methods for determining di-, tri- and tetra-valent manganese were applied to contrasted soils without, however, finding any infallible criterion for manganese deficiency. In most deficient soils a high proportion of the total reducible manganese was in the manganic form.

Pot and field experiments on "marsh spot" in peas showed that direct application of manganese materials to the soil had erratic effects, owing to rapid oxidation. The critical period for the uptake of manganese proved to be from the flowering stage onwards. By injecting various inorganic nitrogen compounds and amino acids into plants it was possible to induce "marsh spot" in peas grown on soils amply supplied with available manganese. It was also found that oats in pot experiments on manganese-deficient soils developed severe "grey speck" when treated with sulphate of ammonia.

#### SPECTROGRAPHIC ANALYSES

With the completion of the new extension in the autumn of 1939 it was possible to equip a basement room as a spectrographic laboratory complete with dark room. The installation consisted of a Hilger medium quartz spectrograph (fd 60 cm.) and non-recording microphotometer, with the necessary equipment for arc and spark methods and for the Lundegårdh air-acetylene flame method for analysing solutions.

In analysing plant material for the common elements by the flame method the preparation of hydrochloric-acid extracts of ash

was found to be too laborious, and a more convenient method avoiding ashing was developed from a brief statement by Terlikowski and Sozunski that hydrochloric acid extracts all the potassium, sodium, calcium and magnesium from dried ground plant material. The extraction of calcium was not quite complete, but all the manganese was extractable. In the standardised method 1.25 g. of dried ground material are extracted by 50 ml. of N.HCl for 24 hours with occasional stirring. Tenfold dilution of this extract is needed for potassium and the higher ranges of sodium and magnesium, and further dilution for calcium.

Boron is determined by the copper arc, the plant ash being mixed with an equal weight of a mixture of sodium chloride and ammonium sulphate containing 1 per cent. of tin as an internal standard. A plate-blackening trace is applied to every plate and a correction made for blackening of the spectral background. Other undetected sources of error may still remain. The standard error of the mean of two determinations on the same ash sample is about 9 per cent.

The Lundegårdh method as adopted for the analysis of plant material has been found extremely useful in diagnosing mineral deficiencies in crops and in following the uptake of nutrients in field experiments. Since 1940 a considerable amount of work has been done in helping Advisory Chemists in the diagnosis of plant diseases. In cooperation with Dr. R. Hull of the Midland Agricultural College and Dr. Marion Watson of the Rothamsted Plant Pathology Department, sugar-beet diseases have been studied both by means of field experiments and by analysing samples from many different localities. Deficiencies of magnesium, manganese and boron and ones due to potassium and/or sodium can now be recognised and distinguished from each other and from chlorosis of the foliage due to virus and fungus diseases. On some acid soils chlorosis is accompanied by excess manganese in the leaves.

The technique of sampling field experiments during the growing season to obtain representative samples was studied in 1940 and 1941 for potatoes and sugar beet. Every year since then samples of leaves and roots from nearly all the main series of sugar-beet manurial experiments have been analysed to study the function of salt in the manuring of sugar beet and the role of sodium in the plant. To obtain more information on these questions series of whole-plant samples were taken in collaboration with Dr. D. J. Watson at three-weekly intervals from all plots in the Rothamsted two-course rotation experiment during 1942 and 1943. Plant weights and leaf areas were taken on each occasion and the samples analysed for nitrogen, potassium, sodium, calcium, magnesium and manganese.

A considerable number of analyses have been made on leaves from Nigerian oil palms showing various "yellowing" diseases, and some evidence obtained of potassium and magnesium deficiencies.

#### SOIL MINERALOGY

The chief object of this section of the work was to develop methods for a quantitative determination of the various minerals in the clay fractions of soils, and to use them for the study of

typical soil profiles. In a critical review (19) of the results obtained by 1939, it was shown that it was essential in determining clay minerals to use combinations of X-ray, optical and chemical methods. One example of this approach to problems of soil formation was given in a detailed study (21) of two Indian soil profiles. It was shown that contrasted clays can be produced from the same parent material under different topographical conditions, with kaolin as the chief constituent under eluvial and montmorillonite under illuvial conditions. Work on other tropical soils from the Sudan, India and West Africa was interrupted by the war. As a preparation for further work in this field, existing information on the mineralogy of soil colloids was reviewed in 1944 in a Technical Communication of the Imperial Bureau of Soil Science (20).

The same technique for studying clay minerals was used in work conducted for the Medical Research Council's Enquiry into Pulmonary Disease in South Wales Coalminers (24-30). The mineralogical composition and sources of airborne dusts and composition of lung residues of miners who had suffered from pneumokoniosis were studied, and the results correlated with medical, environmental and pathological work.

#### ANALYTICAL METHODS

The large number of soil, fertiliser and crop samples taken in connection with the field and pot experiments in the work described above made heavy demands on the analytical staff of the Department. Whenever moderate accuracy will suffice rapid colorimetric or turbidimetric methods have been developed. Thus, in the small pot experiments the radishes from single pots or from bulked replicate pots are burnt with alcoholic magnesium nitrate and ashed, and the phosphoric acid determined colorimetrically by the Hilger Absorptiometer. Other crops and bulky organic manures are digested with sulphuric and nitric acids, and the phosphate determined colorimetrically. Approximate determinations of potash in crops, manures and extracts of soils are made turbidimetrically by the cobaltinitrite method.

Where less accuracy will suffice, colorimetric and turbidimetric determinations are made visually for phosphate, potassium and magnesium. In plant samples from field plots and for checking visual diagnoses of deficiency symptoms, comparisons have been made between the total contents of these nutrients and the amounts brought out in "tissue-testing." With Morgan's reagent (sodium acetate and acetic acid) the amounts of phosphate and potash extracted increased steadily up to two hours. Normal hydrochloric acid extracted the potash more rapidly and completely.

Nitrates and ammonia in soils are determined by extracting with an acid salt solution and determining ammonia before and after reduction with reduced iron using either the Conway aeration unit or the micro-Kjeldahl method for the ammonia.

Methods have been developed for determining fluorine in fertilisers and some progress made with a micro-method for use on soils.

## PUBLICATIONS

### SOIL FERTILITY AND MANURING

1. SAMPSON, H. C. and CROWTHER, E. M. 1943. *Crop production and soil fertility problems*. The West Africa Commission 1938-1939 Technical Reports, Leverhulme Trust, 8-58.

This report on one section of the agricultural problems of the British West African colonies was prepared by two of the technical experts who accompanied a Commission of four members of Parliament on a tour of all the British and French colonies in West Africa during the winter of 1938-9. It describes the main climatic, geographical and soil conditions, the characters and origins of the principal crops and the main types of West African agriculture, including the special problem of plantations in West Africa. It discusses the administrative and investigational work of the several Departments of Agriculture and the agricultural problems arising in the activities of other Departments. Proposals are advanced for a post-war reorganisation of scientific research and survey, with special emphasis on ecological and soil surveys, meteorology, field experiments on crop rotations and the maintenance and improvement of soil fertility.

2. CROWTHER, E. M. 1943. *The maintenance of soil fertility*. J. Roy. Soc. Arts., **91**, 431-437. Reprinted in *The American Fertilizer*, **99**, No. 12; and *Agriculture—Today and Tomorrow*, edited by Sir John Russell; and in abridged form in *Agriculture*, **50**, 262-264.

A general discussion of the scientific and practical problems involved in the maintenance of soil fertility, with special reference to the role of soil organic matter and plant roots in the formation and conservation of soils, the rotation of crops, and the supply of available nutrients.

3. CROWTHER, E. M. 1942. *Straw on the land*. J. Min. Agric., **49**, 146-150.

The practical problems involved in utilising local war-time surpluses of straw are discussed in relation to the nutrient contents of straw, the physical effects of straw manures, the preparation of composts and direct ploughing-in. On the average the straw crop from one acre contains about as much potash as 1 cwt. of muriate of potash, but on potash-deficient soils the straw supplies much less potash. Results of a Rothamsted rotation experiment since 1934 showed that straw and a fixed quantity of fertilisers gave the best results when the straw was ploughed-in during the autumn and fertilisers applied directly to the following potato crop. The immediate effect from composting the straw and fertilisers was less than using the fertilisers alone. In the second year there was moderate residual effect from each method of using straw. There was little or no immediate or residual effect of straw in either form on sugar beet or barley.

4. CROWTHER, E. M. 1943. *Composts, their preparation and value*. Ann. Appl. Biol., **30**, 392-395.

A general review.

5. CROWTHER, E. M. and BUNTING, A. H. 1942. *The manurial value of sewage sludges*. Annual Summer Meeting, July 9th and 10th, 1942, J. Inst. Sewage Purif. 13-25.

The history of investigations into the agricultural use of sewage and sewage sludges is summarised in relation to modern views on the part of soil organic matter and plant foods in the maintenance of soil fertility. The principal types of current sewage sludges are described. The results of preliminary pot and field experiments on representative sewage sludges are briefly presented.

6. CROWTHER, E. M. and BUNTING, A. H. 1944. *The manurial value of sewage sludges*. Part 2. Presented at Annual General Meeting, Midland Institute, Birmingham. 6th and 7th December, 1944. J. Inst. Sewage Purif., 46-52.

A review of the results of recent field and pot experiments on different kinds of sewage sludge, with analytical data for the average chemical com-

positions of sewage sludges grouped according to the principal processes used at the sewage works. Although some sewage sludges supplied considerable amounts of readily available nitrogen and phosphate, a few others were singularly inactive in field experiments on potatoes. Digested sewage sludges have not only better physical properties but supply more available nitrogen than raw sludges. Shed-dried sludges hold out considerable promise, especially where they can be prepared from sewage without unduly high contents of heavy metals and other industrial wastes.

7. RUSSELL, E. J. and GARNER, H. V. 1941. *Rothamsted experiments on the manuring of potatoes*. Emp. J. Exp. Agric., 9, Part I: 195-215. Parts II and III: 217-235.

The results of experiments carried out at Rothamsted and its outside centres from 1921-1940 are discussed. Sulphate of ammonia was usually very effective on a wide range of soils. The mean increase was at the rate of 0.92 tons of potatoes for 0.25 cwt. of N per acre. Nitrogen gave practically the same increase in the presence as in the absence of dung, and the nitrogen response was usually greater in the presence of phosphate and also in the presence of potash. None of the other sources of nitrogen included in the experiments, calcium cyanamide, ammonium bicarbonate, ammonium chloride was as effective as sulphate of ammonia. Superphosphate was less consistent in its action than sulphate of ammonia, the increases ranging from 0.4 to 1.9 tons for 0.5 cwt. of  $P_2O_5$  per acre according to soil type. It did best on the fenland soils and the heavier type of mineral soils. The phosphate responses in presence of dung were on the average little more than half those observed in the absence of dung. Gafsa phosphate was ineffective on potatoes at Rothamsted.

Potash responses depended on the state of fertility of the soils, rich well-farmed soils giving high basal yields and low responses to potash, poor soils giving as a rule handsome responses. Light fenland soils, and the heavy loam at Rothamsted were highly responsive to potash dressings. Dung reduced the potash responses by approximately 80 per cent.

Magnesium sulphate was ineffective at practically all centres at which it was tried. Chloride, applied as ammonium, sodium or potassium chloride had a slight and rather consistent depressing effect on yield amounting on the average to a loss of 5 cwt. of potatoes per 1 cwt. of Cl supplied in the manure.

Farmyard manure and potassic fertilisers markedly increased the percentage of saleable tubers.

8. CROWTHER, E. M. and GARNER, H. V. 1940. *Potash and potash substitutes for sugar beet*. Sugar Beet Rev., 13, 248-250.

A summary of the results of pre-war field experiments in which promising results had been obtained from agricultural salt.

9. CROWTHER, E. M. 1943. *Worth its weight in sugar*. Farmers Weekly, 18, No. 15, 29.

A summary of the results of field experiments over several seasons in each sugar beet factory area of Great Britain in which it was found that agricultural salt at the rate of 5 cwt. per acre gave on the average about its own weight of additional sugar. This extra crop is obtained without any increased drain on the potash reserves of the soil. The increased crops given by salt contain no more potassium but much more sodium than those grown without salt. Sugar beet and mangolds are among the few cultivated crops for which sodium is a true plant food.

10. CROWTHER, E. M. 1945. *Combine-drilling of phosphate fertilisers for cereals*. Agriculture, 52, 170-173.

The average results of 46 trials by Technical Development Sub-Committees during 1943 and 1944 showed that  $1\frac{1}{2}$  cwt. superphosphate per acre drilled with the seed was as effective as 3 cwt. broadcast.

11. HAINES, W. B. and CROWTHER, E. M. 1940. *Manuring Hevea*. III. *Results on young buddings in British Malaya*. Emp. J. Exp. Agric., 8, 170-184.

The results of fourteen manuring experiments with young budded rubber laid down in 1934 on the Dunlop rubber estates in Malaya are described.

The experiments tested sulphate of ammonia, mineral phosphate and muriate of potash each at three levels in a 3 x 3 x 3 design, with individual plots of 3 experimental rows half a mile long with a pair of similarly treated guard rows. Each set of five fertilised plots was separated from the next set by an unmanured row. The set of experiments occupied 1,260 acres, exclusive of guard rows.

Phosphate proved to be the outstanding stimulant of growth, earlier maturity to the extent of between 6 months and 2 years being gained where the manuring was started early in the life of the trees. Nitrogen gave much smaller effects, and potash normally gave nothing measurable. Interactions between fertilisers were not of great importance. The yields of latex per tree in early tapping years gave very small differences in comparison with the great influence of phosphate on vigour and growth as shown in the number of trees fit for tapping.

#### SOIL PHOSPHATES

12. NAGELSCHMIDT, G. and NIXON, H. L. 1944. *Formation of apatite from superphosphate in the soil*. *Nature*, **154**, 428.

Chalk fragments from Broadbalk plots which had received superphosphate for a century contain up to 3 per cent.  $P_2O_5$ . X-ray diffraction data before and after heating suggest that the phosphorus is present as apatite. Examination of old soil samples showed that both phosphorus and fluorine contents increased steadily with time, at first slowly when the superphosphate was made from bone ash, and much more rapidly since 1888 when the superphosphate has been made from mineral phosphate, relatively rich in fluorine.

13. GHANI, M. O. 1941. *Fractionation of phosphoric acid in organic manures*. *Indian J. Agric. Sci.*, **11**, 954-958.

The distribution of the principal forms of phosphorus compounds were determined in poultry manure, farmyard manure and straw compost, prepared by the Adco process. A large part of the phosphorus in poultry manure was present as phytin, but three-quarters of the total in farmyard manure was inorganic, mostly in an easily soluble form. Classical plots at Rothamsted and Woburn gave high values for both acetic acid-soluble and organic phosphorus where farmyard manure had been applied repeatedly.

14. GHANI, M. O. 1942. *Determination of organic phosphorus in alkali extracts of soils*. *Indian J. Agric. Sci.*, **12**, 336-339.

In colorimetric determinations of inorganic and organic phosphorus in alkali extracts of soils, oxidation of organic matter by bromine at 100° breaks down a part of the organic phosphorus compounds, but oxidation at room temperature does not.

15. GHANI, M. O. 1943. *Fractionation of soil phosphorus. I. Method of extraction*. *Indian J. Agric. Sci.*, **13**, 29-45.

Earlier work by L. A. Dean (*J. Agric. Sci.* 1938, **28**, 234) on extracting phosphorus by successive treatments with a weak acid, an alkali and a strong acid was critically examined and extended. N/2 acetic acid was used for the preliminary removal of exchangeable bases and as a solvent for readily soluble phosphorus. Sodium hydroxide extracted more phosphorus than sodium carbonate at equivalent concentrations. Alkali extraction at 100° dissolved more phosphorus than at 20° or 40° but organic phosphorus compounds were partly decomposed. Several extractions were necessary to dissolve the whole of the soluble phosphorus at room temperature, and a suitable porous candle filtering apparatus was therefore devised for repeated extractions. 2N sulphuric acid after the alkali treatment was suggested for removing the apatite fraction. See also Part II. *Chemical nature of the phosphorus fractions* by M. O. GHANI and S. A. ALEEM, 1943, *ibid*, **13**, 142-147.

16. GHANI, M. O. 1944. *Fractionation of soil phosphorus. III. The organic phosphorus fraction*. *Indian J. Agric. Sci.*, **14**, 261-267.

An alkali extraction method and an ignition method for determining organic phosphorus gave fairly concordant results, except in fen soils. With suitable modifications to meet the special conditions of these soils the two methods gave comparable results. The hydrogen peroxide method for determining organic phosphorus was found to have serious limitations.



17. GHANI, M. O. 1943. *The use of 8-hydroxyquinoline as a means of blocking active iron and aluminium in the determination of available phosphoric acid of soils by dilute acid extractions.* Indian J. Agric. Sci., **13**, 564-565.

8-hydroxyquinoline was used to deactivate free sesquioxides during acid extraction of soils. It prevented re-fixation of phosphorus dissolved by acetic acid from a mixture of basic iron phosphate and mineral phosphate, and increased the amount of phosphorus extracted by acetic acid, especially from acid lateritic soils.

#### MINERALOGY OF SOIL COLLOIDS

18. RAYCHAUDHURI, S. P. 1941. *Studies of the physico-chemical properties of associated black and red soils of Nyasaland Protectorate.* Indian J. Agric. Sci., **11**, 100-109.

Detailed physico-chemical studies were made on a typical pair of closely adjacent red and black soil profiles from Domira Bay, Nyasaland. The silica-alumina and silica-sesquioxides in the clay fractions were only slightly lower in the red soils. The free sesquioxides by Truog's method were higher in the clay fractions from the red soils. The black soils had only a little more total carbon but appreciably higher C/N ratios than the red soils at similar depths. The black soils had much higher exchangeable bases, buffer capacities, moisture equivalents and imbibitional moisture capacities than the red soils, although the two types had nearly the same clay contents.

19. NAGELSCHMIDT, G. 1939. *The identification of minerals in soil colloids.* J. Agric. Sci., **29**, 477-501.

X-ray, optical, dehydration and chemical methods in use for the identification of minerals in soil colloids are discussed with special regard to their limitations. These are mainly due to uncertainties about the variation of physical properties of standard minerals with decreasing grain size and the possible existence and importance of amorphous material. The aggregate method in X-ray analysis, which is specially important for soil colloids, is described. Various techniques for dehydration and optical analysis are discussed, and it is concluded that they are useful only when used in combination with X-ray data. The advantages of combining various chemical methods with X-ray analysis are pointed out.

Standard data are given for a number of minerals, and the evidence for their occurrence in soil colloids is reviewed. These minerals are quartz, cristobalite, oxides and hydroxides of iron and aluminium, minerals of the kaolinite, montmorillonite and mica groups.

20. NAGELSCHMIDT, G. 1944. *The mineralogy of soil colloids.* Imp. Bur. Soil Sci. Tech. Commun., **42**, pp. 43.

A critical review of the literature with bibliography.

21. NAGELSCHMIDT, G., DESAI, A. D. and MUIR, A. 1940. *The minerals in the clay fractions of a black cotton soil and a red earth from Hyderabad, Deccan State, India.* J. Agric. Sci., **30**, 639-653.

The mineral compositions of the clays from a red earth and a black cotton soil from Hyderabad, Deccan State, India, occurring in close proximity in the field are determined. Both soils are derived from the same or from very similar parent rocks, a coarsely crystalline granite or gneiss.

For both soils there is practically no variation in the mineralogical composition of the clay throughout the profile, but for any given clay there is some variation with grain size. The main contrast between the two is that the red clay contains predominantly kaolinite or halloysite whereas the black clay contains mainly beidellite, a member of the montmorillonite group. The topography appears to be the principal factor associated with this difference in minerals, and the processes of weathering believed to have produced the contrasted clays are discussed with reference to experiments on the leaching of felspar in the laboratory and on hydrothermal synthesis.

22. NAGELSCHMIDT, G. 1941. *The identification of clay minerals by means of aggregate X-ray diffraction diagrams.* J. Sci. Inst., **18**, 100-101.

23. NAGELSCHMIDT, G. 1944. *X-ray diffraction experiments on illite and bravaisite*. *Min. Mag.*, **27**, 59-61.

One sample of illite and a fresh glauconite showed no change in basal spacing on saturation with calcium. Two illites from Illinois showed a small but distinct broadening of the basal reflection towards larger spacings on saturation with calcium. Although illite may be assumed to have a basal spacing of 10 Å, there may be intermediate products of weathering in which 10 and 15 Å packets are interstratified in various ways.

#### MINERALOGY OF AIR-BORNE DUSTS AND COALS

24. NAGELSCHMIDT, G. and KING, E. J. 1941. *The biochemistry of silicic acid. 9. Isolation and identification of minerals in lung residues and air-borne dusts from coal mines*. *Biochem. J.*, **35**, 152-158.
25. NAGELSCHMIDT, G. and HICKS, D. 1943. *The mica of certain coal-measure shales in South Wales*. *Min. Mag.*, **26**, 297-303.
26. NAGELSCHMIDT, G. and HICKS, D. 1943. *X-ray diffraction technique for the identification of non-coal minerals in coal*. Proceedings of a Conference on the ultra-fine structure of coals and cokes, B.C.U.R.A. London, June 1943.
27. NAGELSCHMIDT, G. 1943. *Chronic pulmonary disease in South Wales coalminers. II. Environmental studies. D. The composition of the air-borne dusts at the coal-face in certain mines*. Special Report Series No. 244 (Medical Research Council) published by H.M. Stationery Office, 95-124.
28. HICKS, D. and NAGELSCHMIDT, G. 1943. *Chronic pulmonary disease in South Wales coalminers. II. Environmental studies. F. The chemical and X-ray diffraction analysis of the roof and clod of some South Wales seams, and of the mineral matter in the coal*. Special Report Series No. 244 (Medical Research Council) published by H.M. Stationery Office, 153-186.
29. HICKS, D. and NAGELSCHMIDT, G. 1943. *Chronic pulmonary disease in South Wales coalminers. Appendix II. The composition of the dust in the run-of-mine coal*. Special Report Series No. 244 (Medical Research Council) published by H.M. Stationery Office, 218-222.
30. KING, E. J. and NAGELSCHMIDT, G. 1945. *Chronic pulmonary disease in South Wales coalminers. III. Experimental studies. B. The mineral content of the lungs of workers from the South Wales coalfield*. Special Report Series, No. 250. Chronic Pulmonary Diseases in South Wales Coalminers, Part III (Medical Research Council), published by H.M. Stationery Office, 1-20.

#### GENERAL

31. OLDERSHAW, A. W. and GARNER, H. V. 1942. *Tunstall experiments with carbonate of lime*. *Agriculture*, **49**, 37-39.

A summary of the first 10 years' results on a replicated experiment on the acid soil (pH 4.9) at Tunstall, Suffolk, showing the exhaustion of ground chalk applied at 4 levels in 1932. Excellent yields were obtained, but definite deterioration of the plots receiving only 1 ton of carbonate per acre were apparent towards the end of the period.

32. OLDERSHAW, A. W. and GARNER, H. V. 1944. *Light land experiments at Tunstall, Suffolk*. *J.R.A.S.E.*, **105**, 98-114.

An account of the Tunstall experiments since their commencement in 1925. The results include replicate experiments on potatoes and carrots, testing dung and its interactions with mineral fertilisers.

33. GARNER, H. V. 1944. *The war-time manuring of sugar beet*. *Farmers Weekly*, August 1944.
34. GARNER, H. V. 1943. *The production of malting barley*. *J. Incorp. Brewers Guild*, November 1943.

35. GARNER, H. V. 1943. *Straw composting*. Farmer & Stockbreeder, December 1943.
36. GARNER, H. V. 1941. *Agricultural salt*. Agriculture, **48**, 183-185.
37. NORMAN, A. G. 1939. *The composition of forage crops. III. Cocksfoot. Changes in the herbage during growth, with and without additions of nitrogenous fertiliser*. Biochem. J., **33**, 1201-1208.
38. CROWTHER, E. M. and WEIL, J. W. 1939. *Report on the analyses of barleys valued for the Rothamsted Conferences of 1935, 1936 and 1937*. J. Inst. Brewing, **45**, No. 12.
39. BUNTING, A. H. 1945. *Sewage sludge and the market gardener*. Agriculture, **52**, 123-126.

A general discussion of modern sewage sludges, their manurial value and use in composting straw.

40. CROWTHER, E. M. 1945. *Russians overcome late frosts in potatoes*. Farmer & Stockbreeder, July 17, 1129.

In an account of recent Russian developments in agricultural science, mention was made of methods of overcoming late frost damage to potatoes in the extreme north by planting in wide high ridges across the direction of the winds prevailing in the dangerous period of August and September.

41. CROWTHER, E. M. 1945. *Agrochemistry*. Nature, **156**, 227.

A review of some of the outstanding achievements of Russian agricultural chemistry, as demonstrated during the celebrations of the 220th Anniversary of the Academy of Sciences of U.S.S.R. in June, 1945.

42. CROWTHER, E. M. 1945. *Muck, magic and composts*. Farmer & Stockbreeder, **59**, 1960.

A review of Sir Albert Howard's "Farming and Gardening for Health and Disease."

43. CROWTHER, E. M. 1941. *5 cwt. of salt : 5 cwt. extra sugar*. Farmer & Stockbreeder, **55**, 620.