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Chemistry Department

G. W. Cooke

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CHEMISTRY DEPARTMENT

G. W. COOKE

I. K. Otter left in March and R. K. Cunningham joined the staff in June. P. W. Arnold and D. S. Jenkinson attended the Seventh Congress of the International Society of Soil Science at Madison, Wisconsin. G. W. Cooke visited Israel in March as a guest of Dead Sea Works Ltd. and of the Agricultural Research Station of the Israel Ministry of Agriculture; he gave a series of lectures and visited research institutes and experimental farms. In October Cooke attended a Conference at Seville on "Nitrogen in Agriculture" organised by the journal *Agrochimica*.

R. G. Heathcote of the Colonial Research Service left in September to go to Nigeria. Y. C. Lin (from Hong Kong) joined the Department for training in soil-fertility work. N. J. Barrow (Australia), M. Birecki (Poland), S. Oliver (Spain), R. C. Salmon (Rhodesia), K. Singh (India), L. V. Vaidyanathan (India) and Y. Yamada (Japan) all worked in the Department during the year.

SOIL ORGANIC MATTER AND ORGANIC MANURES

Soil organic matter and freshly added organic materials have quite different effects on crops. Decomposed organic matter in soil contains reserves of plant nutrients (mainly N, P and S) that are released slowly; it also modifies permanently some of the physico-chemical properties of soil, but often it has relatively little effect on gross physical characters. Freshly added plant residues and organic manures usually improve physical properties and also provide a "flush" of N and P when they decompose; both effects may increase crop yields but are short-lived. In laboratory work we are attempting to distinguish between "old" and "new" organic matter and to measure rates of decay. In field experiments we are examining the effects of farmyard manure, and of crop residues to try to separate nutrient and physical actions. We are also developing simple methods for measuring physical improvements in soils produced by organic matter, and for characterising "difficult" soils where crops benefit from improving physical properties. Such work is essential for interpreting experiments on organic manures and for assessing practices that increase soil organic matter.

Decomposition of plant material in soil

Ryegrass roots and tops uniformly labelled with ^{14}C were incubated with soil from two Broadbalk plots (Plot 2B has farmyard manure (FYM) each year and the soil has 2.4% of organic C, Plot 3 has no manure or fertiliser and the soil has only 0.97% of organic C). With both soils the plant carbon lost as CO_2 was directly proportional to the amount added when N supply was enough for microbial activity. About one-third of the plant carbon

(from either roots or tops) was left in the soil after laboratory incubations lasting 156 days, and after field incubations lasting from April to October; there was little further loss of carbon in the field during the next winter. Adding fresh ryegrass tops increased rates of decomposition of "native" soil organic matter, in soil from Plot 2B by 50% and in soil from Plot 3 by 100%. Some of the CO₂ evolved during incubation was fixed (presumably by micro-organisms) and returned to the soil organic matter. The same proportion of CO₂ was fixed in the laboratory experiments on both soils (1½% of that evolved), and was not affected by adding plant material. (Jenkinson.)

After ryegrass uniformly labelled with ¹⁴C had decomposed in soil for a year in the field, residual activity (31% of that added) was determined in fractions of soil organic matter separated by J. M. Bremner and T. Harada's system (*J. agric. Sci.* (1959), **52**, 137). The results given are from an experiment where 0.3% of labelled plant material was added to soil from Broadbalk Plot 2B:

| | Total carbon (Percentages of carbon in each fraction) | Added plant carbon |
|--------------------------------|--|-----------------------|
| Inorganic | 5 | 1 |
| Soluble in 0.1N-HCl | 1 | 4 |
| Soluble in 0.5N-NaOH | 48 | 60 |
| Soluble in N-HCl : N-HF | 4 | 4 |
| Residual | 41 | 32 |

The distribution of added plant carbon was not very different from that of carbon in the whole soil organic matter. "New" soil organic matter did not occur predominantly in one fraction of the whole organic matter, as would happen if this method separated "labile" and "stable" fractions of organic carbon in soils. The distribution of "new" organic matter in the two Broadbalk soils was similar. (Jenkinson.)

Oxidation of soil organic matter by dichromate

In titrimetric methods for determining carbon in soil, the oxidising mixtures must be at a high temperature to oxidise organic matter completely, but this also tends to decompose the dichromate. By omitting H₂SO₄ and using only H₃PO₄ and K₂Cr₂O₇ in the mixture, oxidation may be done at 175° without decomposing dichromate rapidly. The organic carbon of a number of soils was oxidised quantitatively by this method, the CO₂ being collected gravimetrically and the O₂ used determined titrimetrically on the same sample.

$$\text{The ratio } \frac{\% \text{ C calculated from O}_2 \text{ used}}{\% \text{ C calculated from CO}_2 \text{ evolved}}$$

ranges from 2 for CH₄ to 0 for CO₂, with soils about 1. Soils differed significantly in this ratio: values for Broadbalk FYM-treated plot (2B) and the unmanured plot (3) were 1.05 and 0.97 respectively, implying that carbon in the FYM plot is less oxidised. The oxidation ratio of a low-moor-alkaline peat was 1.00, that of a high-moor sedge peat 1.16. For this ratio to be determined accurately, samples must contain no Fe⁺⁺, Cl⁻ or charcoal. (Jenkinson.)

Farmyard manure

Elaborate field experiments are needed to measure the separate contributions towards crop yields made by the nutrients in FYM and by any other direct or indirect effects it may have. Besides N, P and K, other elements supplied, such as Ca, Mg and Na, may increase yields. Neither the chemical forms of these elements in manure, nor their availabilities to plants, are known precisely, which complicates even further any experiment on FYM that attempts to interpret its value.

Chemical composition. The total contents of N, P and K differ in different samples of farmyard manure, depending on origin and on storage methods. Three samples used in experiments at Rothamsted and Woburn had the following contents:

| Sample | Dry matter (%) | Total (% in dry matter) | | |
|--------|----------------|-------------------------|-----|-----|
| | | N | P | K |
| 1 | 20.0 | 2.0 | 0.3 | 1.5 |
| 2 | 18.0 | 3.0 | 0.7 | 2.0 |
| 3 | 25.5 | 3.5 | 1.2 | 4.4 |

The differences illustrate the difficulties of comparing similar amounts of nutrients as FYM and as fertilisers in field experiments of simple design. Thus if FYM at 15 tons/acre were compared with 0.9 cwt. N, 0.75 cwt. P_2O_5 and 1.5 cwt. K_2O /acre; the comparisons that were satisfactory with either sample 1 or 2 might fail with sample 3 (this was so high in K that 15 tons/acre supplied 4 cwt. K_2O /acre in a readily soluble form).

The solubilities of Ca, Mg, K and Na of FYM in several solvents were determined for a few samples (Smith). Amounts of elements dissolved in boiling concentrated HCl were used as "total" contents, and the average values for the manures tested were Ca 1.7%, Mg 0.4%, K 3.6% and Na 0.3%. Of these amounts water dissolved 5% of Ca, 10% of Mg, 75% of K and 80% of Na. Cold N-HCl and N-ammonium acetate both dissolved 95% of the total K and 100% of the Na. In contrast, these two solvents extracted different proportions of Ca and Mg. Ammonium acetate extracted 15% more of the Ca and Mg than did water, and dilute HCl dissolved 85% of the Ca but only 40% of the Mg. Approximately all the K and Na in FYM can be rated as available to crops, but more than half of the Mg is not even soluble in dilute acid. Some at least of the Ca that is insoluble in a neutral salt solution is present as carbonate, which would serve as a Ca reserve.

Position effects. Rothamsted experiments summarised by H. D. Patterson and D. J. Watson (*Rep. Rothamst. exp. Sta.* for 1959, pp. 164-168) showed that FYM was more effective relative to fertilisers for sugar beet than for potatoes, perhaps because the N, P and K equivalents of FYM are greater for sugar beet than potatoes. Besides the possibility that nutrients in FYM and in fertilisers have not the same relative efficiencies for varied crops, the positions in the soil of the nutrients supplied by the two kinds of materials differ, especially where fertilisers are broadcast on the surface of a seedbed for crops like sugar beet. At sowing, and for at least

a month afterwards, nearly all broadcast P and K fertilisers are confined to the top 2 or 3 inches of soil, and dry weather after sowing prevents much of these nutrients reaching plant roots. In contrast, nutrients supplied by FYM are deeper and in moister soil.

Differences in both quantities and position of nutrients may explain the higher yields from FYM than from fertilisers obtained in both the Market-garden and the Ley-Arable Experiments at Woburn. In the Market-garden Experiment FYM supplied much more total N, P and K than were given as fertilisers, or were needed by the red beet grown. Both P and K have accumulated in the soil from FYM dressings, and analyses below show the two nutrients have moved into the subsoil at least 24 inches deep.

Readily-soluble P and K (mg./100 g. of soil)

| Depth (in.) | Fertiliser plots | | Farmyard manure plots | |
|----------------|---------------------|---|--------------------------|----|
| | P | K | P | K |
| 0-9 | 31 | 8 | 85 | 35 |
| 12-18 | 12 | 6 | 21 | 33 |
| 18-24 | 9 | 5 | 17 | 32 |

P and K levels are much higher in the FYM plots; the nearly constant high level of K for all three sampling depths in FYM plots indicates that much added K has passed below 24 inches and is now out of range of globe beet roots. The 30 tons/acre average annual dressings of FYM used are maintaining at each depth a "saturation value" of 30-35 mg. K/100 g. of soil. On the plots of the Rothamsted Classical Experiments "saturation" level has not been reached, even in the surface soil, for, although the readily soluble K is much higher on Barnfield than the "saturation value" at Woburn, K levels in Rothamsted subsoils (9-18 inches) are only two-thirds of those in surface soils.

In the Market-garden Experiment at Woburn much P has accumulated in soils where FYM and sewage sludge have been applied for the last 18 years. Table I shows the gains, relative to soil that has had P only as fertilisers.

Most of the gains in total P are as increases of inorganic P soluble in acetic acid and in NaOH; organic P increases are surprisingly small. Apart from the increase in alkali-soluble inorganic P in the second depth of the FYM plots, most of the P has accumulated in the 0-9-inch layer; but gains of P, expressed as percentages of the total gain for each profile (Table I), show that much more inorganic P had moved into the second soil depth with FYM than with sludge, even though much more total P was applied as sludge than as FYM. The Market-garden Experiment has been redesigned to test higher rates of fertilisers incorporated at two depths, and magnesium. The results should indicate whether factors other than the nutrients in FYM improve the crops.

In the Woburn Ley-Arable Experiment from 1956 to 1959 FYM applied to sugar beet gave 10 cwt. more sugar/acre each year when this crop was grown after 3 years of arable cropping or 3 years of lucerne, whereas NK fertilisers had only small effects on yields. Where the test crop of beet was preceded by a 3-year grazed ley,

however, there was no response to manure, and yields without FYM were as good as in the other rotations where FYM was given.

Where no FYM was given the soil had little readily soluble K (3.5 mg. K/100 g. soil) after all rotations except the grazed ley, where it was high (7 mg. K). Where FYM was given to beet, soluble K values were high immediately after the crop was taken, but at the end of 3 years of arable cropping, or 3 years of lucerne, values (ca 4 mg. K) were only a little above those of plots receiving fertilisers only. Soil under grazed ley still had 10 mg. of soluble K/100 g. when the plots were due for the next dressing of FYM.

TABLE I

Extra P in soils of the Woburn Market-garden Experiment receiving farmyard manure and sewage sludge, as compared with soils having fertilisers

| Depth (in.) | Farmyard manure plots | | | | Sewage sludge plots | | | |
|----------------|---|----------------------|-----------|---------|---------------------|----------------------|-----------|---------|
| | Total | Soluble | | | Total | Soluble | | |
| | | In acetic acid | In NaOH | | | In acetic acid | In NaOH | |
| | | | Inorganic | Organic | | | Inorganic | Organic |
| | <i>mg. of P per 100 g. soil</i> | | | | | | | |
| 0-9 | 45 | 34 | 9 | 7 | 145 | 79 | 36 | 18 |
| 12-18 | 25 | 8 | 19 | 3 | 10 | 3 | 5 | 1 |
| 18-24 | 15 | 4 | 10 | 2 | 10 | 3 | 2 | 0 |
| | <i>percentages of gains for the whole profile</i> | | | | | | | |
| 0-9 | — | 74 | 24 | 58 | — | 93 | 84 | 95 |
| 12-18 | — | 17 | 50 | 25 | — | 4 | 12 | 5 |
| 18-24 | — | 9 | 26 | 17 | — | 4 | 4 | 0 |

Soluble K in the soils of the arable rotations without FYM is low because more K is withdrawn by the crops than is added by fertilisers, but in the grazed ley rotation 0.7 cwt. K₂O/acre was gained in the 5-year rotation of test and treatment crops. Where FYM was applied, the K added exceeded the K withdrawn in all rotations; the accumulation was greatest with the grazed ley (3.6 cwt. K₂O/acre per rotation).

In this experiment the K fertiliser broadcast on the surface before sowing supplies 0.9 cwt. K₂O/acre; the 15 tons/acre dressing of FYM, which is ploughed in, supplies 3.9 cwt. K₂O/acre. The fertiliser K has had little effect on the yield or on K uptake by sugar beet; in contrast, both K accumulated in the soil during the grazing period and K supplied by FYM have greatly increased percentage K in beet tops:

Percentage K in dry matter of sugar-beet tops

| | Without manure | Increase by | |
|-------------------------|-------------------|-------------|-----|
| | | Fertiliser | FYM |
| Arable rotation ... | 2.2 | 0.2 | 1.1 |
| Grazed ley rotation ... | 3.6 | 0.05 | 0.6 |

The Market-garden and the Ley-Arable Experiments at Woburn both suggest that differences in the amounts of K supplied by

fertilisers and by FYM, and also differences in the positions of fertiliser-K and of K from FYM within the soil, may account for much of the greater yields produced by FYM than by fertilisers. A preliminary experiment tested these two factors in 1960 on Stackyard Field at Woburn on land that had grown sugar beet in 1959 and where the soluble soil K was higher than in the plots of the arable rotations of the Ley-Arable Experiment. The fertilisers were either broadcast on the seedbed or dug in and were compared with FYM (15 tons/acre) and with peat saturated with solutions of NH_4NO_3 and KCl at rates so adjusted that the test dressing provided as much organic matter as the FYM treatment, and as much N and K as the NK fertiliser treatment. The whole experimental site received the same basal dressing/acre as the Ley-Arable Experiment (0.7 cwt. N, 0.7 cwt. P_2O_5 and 0.9 cwt. K_2O). Table 2 gives weights of sugar-beet seedlings at singling and tops and roots at harvest. There were clear gains from using extra NK fertilisers and also from digging the dressings in. In the Market-garden Experiment FYM gave greater yields of red beet than fertilisers, and the beet developed earlier; in this experiment sugar-beet seedlings with NK fertilisers dug in were even earlier than those with FYM. At harvest FYM gave nearly 1.5 tons more roots per acre, but slightly less tops, than dug-in NK fertilisers; both treatments increased yields by about 50% above the "untreated" plots (which had received the standard dressings of the Ley-Arable Experiment).

TABLE 2
Weights of sugar beet at singling time and harvest

| Treatment/acre (addition to basal NPK) | | | | | At singling (g. dry matter/ microplot) | At harvest (tons/acre) | |
|---|-----|-----------|-----|-----|--|---------------------------|-------|
| | | | | | | Tops | Roots |
| None | ... | ... | ... | ... | 20 | 10.3 | 9.3 |
| 0.7 cwt. N | { | Broadcast | ... | ... | 21 | 16.2 | 11.3 |
| | | Dug in | ... | ... | 33 | 15.6 | 11.9 |
| 3.6 cwt. K_2O | { | Broadcast | ... | ... | 11 | 10.7 | 9.7 |
| | | Dug in | ... | ... | 24 | 11.2 | 12.9 |
| 0.7 cwt. N + 3.6 cwt. K_2O | { | Broadcast | ... | ... | 12 | 15.1 | 12.4 |
| | | Dug in | ... | ... | 48 | 17.2 | 14.6 |
| FYM | ... | ... | ... | ... | 25 | 16.5 | 16.0 |
| Enriched peat | ... | ... | ... | ... | 38 | 16.1 | 15.3 |

Broadcast NK fertilisers checked germination and retarded growth of the seedlings; at harvest yields of tops and roots were each 2 tons/acre lower than from the same fertilisers dug in. Putting the K fertiliser lower in the soil increased its effectiveness, but much of the difference in yields of roots between fertilisers and FYM was eliminated by equalising the amounts of K applied. (Warren and Johnston.)

Organic matter and soil structure

On many soils, Rothamsted clay is an example, crops grown in continued arable systems yield well for many years without needing more organic materials to improve structure than the residues of crops grown on the land; with other soils, this treatment degrades

structure because organic matter is lost, and yields fall. At present there is no better basis for advising farmers on the need to improve soil structure than local knowledge, and there is no suitable routine laboratory or field procedure for identifying "difficult" soils. Poor structure that interferes with root growth is probably associated with proportions of sand and silt that lead to close packing of soil, and with too little clay or organic matter of the right kind to bind aggregates. Close packing leads to shortages of water and air, and to pores that are too small for roots to grow through when the soil mass dries and becomes rigid.

Clay and sandy soils, mainly from Rothamsted and Woburn, which have had contrasted but continuous treatments, were used in laboratory experiments. Thin sections made by D. A. Osmond (Soil Survey) showed close packing in natural aggregates of sandy soils under continuous arable cultivation. Pore-size measurements showed that checks to root growth were not likely in the clays, but were possible in the sands. The effects of grass grown for many years, and of regular annual dressings of FYM, on several physical properties were measured in both sands and clays. Continuous grass was much more effective than regular dressings of FYM in improving the soils by making them more permeable and giving them a structure stable to water and resistant to effects of mechanical ill-treatment. In both sands and clays, aggregates formed under permanent grass were quite stable to water, and the soils were very permeable. Annual dressings of FYM prevented the crumbs of clay soil slaking, but did little to stabilise the sand. Clay aggregates from land cultivated continuously without FYM slaked, but not as seriously as sands having the same treatment (these were quite unstable). Sandy soils in continuous arable cultivation had low water permeabilities after slaking, but where FYM had been given each year the slaked soils were more permeable. Clay soil continuously cultivated had low permeability after slaking, but where FYM had been applied each year slaking was prevented and permeability remained high.

A practical test was developed. The percentage loss in pore space, after wetting and draining an aggregated soil sample twice, measured *structural instability*; passing water through the sample remaining from the slaking test measured *permeability*. The two parts of this test indicate whether soil structure will deteriorate in wet weather and whether trouble may be expected from impermeability when slaking occurs. Samples from the Woburn Ley-Arable Experiment showed that continuous arable cultivation without FYM gave very unstable aggregates and the soil packed badly after slaking. Where FYM was given to root crops in the rotation, soil structure was a little more stable and permeability was higher, but neither improvement from FYM was great. Six months after undersowing cereals with a ley, the soil was much more stable, but it still packed badly. Three years under ley stabilised structure completely, but most of the improvement was lost a year after ploughing.

The test devised needs no elaborate apparatus, is simple and quick to do, and can be used on soils taken during routine sampling for fertility measurements. In this work the test showed the

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essential differences between sands and clays, and distinguished the different effects of FYM and of grass on soil structure. But its value for farmers cannot be assessed until many soils have been examined. (Williams and Cooke.)

FIELD AND LABORATORY EXPERIMENTS ON N, P AND K FERTILISERS

"Micro-plot" experiments

Much of the Department's field work on soil and crop nutrition problems is with "micro" plots (4–12 sq. yd.). Experiments with such small plots are used when land is limited—several new experiments have been superimposed on the large plots of some of the old Classical Experiments. They are also essential when many materials or treatments to be tested make "farm-scale" experiments too unwieldy. Micro-plot techniques suffice where yields need not be related closely to agricultural practice and where crop composition is important. The work is exacting, and much must be done by hand; all operations must be more careful and uniform than is necessary when larger plots are used. Errors with small plots and larger plots have been similar, and micro-plot technique is now one of our standard working methods. Much more field experimentation of this kind has been done by the Department recently by limiting ordinary experimentation on commercial farms. A skilled team under Penny (and in association with Widdowson) now provides this service to the Department. Most of the field work reported here has been made possible by this change.

Timing of nitrogen dressings for winter wheat

Seventeen experiments done from 1958 to 1960 measured the worth of autumn seedbed-dressings of N for winter wheat, in terms of equivalent spring top-dressings applied either early (in March) or late (in May). Combinations of these three times of manuring were also tested. Table 3 summarises fourteen experiments on medium-heavy loams giving satisfactory results at harvest. Autumn N increased yields at thirteen of the fourteen centres, but usually by less than equivalent amounts of N given as spring top-dressings. March top-dressings produced the strongest growth, but this was often reflected adversely in severe lodging at harvest. May top-dressings caused very little lodging and usually gave the highest yields. Dividing the lower dressings had no advantage over single May top-dressings, but with the higher level of N, applying half in autumn and half in May, or half in March and half in May, was better than a single dressing. Twelve of the wheat crops followed roots or a legume, and for these May was the best time to apply N. At two centres where the wheat followed a cereal, March N was better than May N, perhaps because it helped to limit damage from "take-all" disease.

Farmers growing wheat on heavy land (and most "wheat-land" is heavy) may apply part of their total N dressing in autumn without losing much fertiliser efficiency, provided the remainder is applied as a "late" (May) top-dressing. This makes "early" (March) spring top-dressing unnecessary, an operation which is often delayed, or made impossible, on heavy land by wet weather. This

work does not apply to wheat grown on light land, where autumn dressings are likely to be leached from light soils; also light land usually provides less N from soil reserves and crops may need early dressings in spring. (Widdowson and Penny.)

TABLE 3

Mean yields of winter wheat grown without nitrogen and the increases given by single and double doses of nitrogen

(14 experiments in 1958-60)

(Yields of grain in cwt./acre at 15% moisture content)

Yield without N fertiliser, 33.2

| | Increase in yield from N applied | |
|--|----------------------------------|----------------|
| | At single rate | At double rate |
| All in autumn | 5.3 | 8.4 |
| All in March | 7.1 | 8.6 |
| All in May | 7.4 | 9.8 |
| $\frac{1}{2}$ autumn and $\frac{1}{2}$ March | 6.3 | 8.4 |
| $\frac{1}{2}$ autumn and $\frac{1}{2}$ May | 6.1 | 10.2 |
| $\frac{1}{2}$ March and $\frac{1}{2}$ May | 6.0 | 10.1 |
| $\frac{1}{3}$ autumn, $\frac{1}{3}$ March and $\frac{1}{3}$ May | 6.4 | 9.3 |

(The rates given were: 0.6 and 1.2 cwt. N/acre in 1958, 0.5 and 1.0 cwt. N/acre in 1959 and 1960.)

Urea for barley on chalk soils

Table 4 gives mean yields from three experiments on Chalk soils in which urea was compared with ammonium sulphate. Each fertiliser was tested when broadcast, combine-drilled (in contact with seed) or placed 1 inch to the side of the seed; the rates used supplied 0.35 and 0.70 cwt. N/acre. Broadcast urea was consistently superior to broadcast ammonium sulphate at each centre at each rate. Combine-drilled urea checked growth slightly at two centres, but did not affect yield; at the third centre drilling urea checked growth severely and gave lower yields than broadcasting.

TABLE 4

Mean yields of grain in three 1960 barley experiments

(Cwt. of grain/acre, at 15% moisture content)

Yield without N, 20.4

| | Yield with N applied at | | | | | |
|--------------------------|-------------------------|--------------------|-------------------------|------------------|--------------------|------|
| | 0.35 cwt. N/acre | | | 0.70 cwt. N/acre | | |
| | Broad- cast | Placed | | Broad- cast | Placed | |
| In contact with seed | | At side of seed | In contact with seed | | At side of seed | |
| Ammonium sulphate ... | 27.2 | 28.8 | 29.5 | 31.9 | 33.2 | 34.5 |
| Urea ... | 28.1 | 28.3 | 29.0 | 32.5 | 31.7 | 34.0 |

Slight checks to early growth from combine-drilling ammonium sulphate did not affect yields, and drilling was better than broadcasting. Urea placed 1 inch to the side of the seed did no damage and gave better yields than broadcast urea. Side-placing

was slightly better than combine-drilling ammonium sulphate. (Widdowson and Penny.)

Ammonium sulphate, "Nitro-Chalk", urea and calcium nitrate for spring wheat and barley

Each fertiliser was compared broadcast, combine-drilled in contact with the seed, placed 1 inch to the side of the seed or top-dressed in early May. At Rothamsted spring wheat was checked by 0.8 cwt. N/acre broadcast as calcium nitrate, but not by broadcasting the other N fertilisers. Combine-drilling 0.4 cwt. N/acre as calcium nitrate checked growth, and this amount as urea caused a slight check. Combine-drilling 0.8 cwt. N/acre checked growth with all the fertilisers, most with calcium nitrate. Placing these fertilisers 1 inch to the side of the seed caused no damage. Yields at harvest showed no consistent differences between methods of application. Ammonium sulphate gave lowest yields and calcium nitrate the highest, perhaps because the wheat was grown on a freshly limed but previously acid clay-loam. At Woburn similar comparisons at 0.3 and 0.6 cwt. N/acre were made on barley. Initially there were slight checks from broadcasting 0.6 cwt. N/acre as calcium nitrate and from combine-drilling 0.6 cwt. N as urea or calcium nitrate, but these effects were less than those on wheat at Rothamsted. Drilling ammonium sulphate gave better early growth than broadcasting, but there was no visual benefit from combine-drilling or side-placing the other forms of N. At harvest, May top-dressings gave the highest yields with each fertiliser. (Widdowson and Penny.)

Tests of concentrated fertilisers and forms of nitrogen

A concentrated fertiliser (20% N, 10% P₂O₅, 10% K₂O) gave slightly higher yields of kale and Italian ryegrass than an equivalent mixture of ammonium sulphate, superphosphate and muriate of potash. The fertilisers supplied 1.0 and 2.0 cwt. N/acre for these two crops. In a test on barley 0.3 and 0.6 cwt. N/acre was applied, and 20 : 10 : 10 was the better fertiliser at the double rate. The experiment also compared ammonium sulphate, ammonium nitrate, calcium nitrate and urea for the same crops and levels of N. 2.0 cwt. N/acre as calcium nitrate or urea checked early growth of grass and kale; there were slight checks from ammonium sulphate and ammonium nitrate; 2.0 cwt. N/acre as calcium nitrate reduced yields of kale and the first cutting of grass. There was no consistent difference between yields from the other forms of N. Calcium nitrate was safe when only 1.0 cwt. N/acre was applied, and this dressing gave the highest yields of kale at this level of manuring. Ammonium nitrate gave the highest yields of grass (at 1.0 cwt. N/acre). At the second cutting of grass (grown on the residues from spring dressings of N) urea gave lower yields than the other forms at each level of manuring. For barley, ammonium sulphate and ammonium nitrate were best. (Widdowson and Penny.)

Effects of preceding one-year leys on nitrogen manuring of wheat

The Ley-Arable Experiments indicate roughly the adjustments that are needed in nitrogen manuring of arable crops that follow

3-year leys; but there is little modern information on the contribution made by 1-year leys to the N needed by following cereals. Pure stands of late flowering red clover (S.151) and of Italian ryegrass (S.22) were cut once and twice respectively for silage and then ploughed before sowing winter wheat. Three levels of N were tested on the 1959 ryegrass and four levels on the 1960 wheat. Table 5 gives yields of the 1959 forage crops and the 1960 wheat: Yields of wheat shown were obtained without N and with the highest dressing of N (0.75 cwt./acre); the response curves showed that at least this amount was needed on all plots. When the 1960 wheat

TABLE 5
Yields of forage crops in 1959 and of the following wheat crops

| | | Forage crops grown in 1959 | | | |
|----------------------------|--|----------------------------|----------|------|------|
| | | Clover | Ryegrass | | |
| Cwt. N/acre in 1959 | | 0 | 0 | 0.75 | 1.5 |
| 1959 | Yields (cwt. dry matter/acre) | 49.1 | 30.6 | 68.5 | 76.6 |
| 1960 | Wheat yields (cwt./acre with 15% moisture) | | | | |
| Grain | { without N | 34.3 | 29.7 | 31.4 | 32.7 |
| | { with N | 45.3 | 47.9 | 44.5 | 43.5 |
| Straw | { without N | 27.9 | 20.7 | 25.9 | 23.0 |
| | { with N | 35.2 | 31.1 | 31.3 | 27.7 |

was grown without N fertiliser clover was the best preparation, but N given to preceding ryegrass gave small increases in yield. When 0.75 cwt. N/acre was applied the highest yields were after ryegrass that received no nitrogen, with clover the next best, but yields following ryegrass dressed with N were less. This may be because the heavy crops of ryegrass grown with N removed much K from a soil containing little available K. Further, potash was not given to the leys, although the wheat had 0.4 cwt./acre K_2O by combine-drill. Results discussed on p. 46 show that high levels of soil K can give yields that may not be attained by fresh fertiliser dressings. (Widdowson and Penny.)

Nitrogen manuring of different grass species

In planning experiments with grass as a test crop the relative responses to N of different species of grasses should be known. In spring 1958 pure swards of cocksfoot (S.37), meadow fescue (S.215), perennial ryegrass (S.24) and timothy (Scotia) were sown direct. The leys were cut twice in 1958, three times in 1959 and twice in 1960; each was grown without N and with 0.3 or 0.6 cwt. N/acre as "Nitro-Chalk" for each cut, 0.5 cwt. P_2O_5 and 1.0 cwt. K_2O /acre was given to all plots annually. Table 6 shows total dry matter produced from each ley over the three years and the effect of this cropping on the level of dilute acid-soluble K in the surface soil immediately before ploughing in August 1960. Without N, cocksfoot was less productive than the other species; but with N, cocksfoot gave most dry matter at each level of manuring; timothy was almost equally productive when N was used, but ryegrass and

meadow fescue were inferior. The tests for available K show heavy N manuring of grass decreases reserves of soil K; the basal dressing of K used would often be considered adequate for practice, but, clearly, it was far too little. (Widdowson and Penny.)

TABLE 6

Total yields of grass species manured with three levels of nitrogen for three years and the soluble potassium in the soils at the end of the experiment

| Cwt. N/acre/cut ... | Dry matter (cwt./acre) | | | Soluble * K (mg./100 g. of soil) | | |
|---------------------|------------------------|-------|-------|-------------------------------------|-----|-----|
| | 0 | 0.3 | 0.6 | 0 | 0.3 | 0.6 |
| Cocksfoot ... | 45.3 | 162.5 | 237.0 | 15 | 9 | 5 |
| Meadow fescue ... | 56.6 | 144.3 | 191.1 | 20 | 13 | 7 |
| Ryegrass ... | 57.9 | 134.4 | 187.2 | 16 | 10 | 7 |
| Timothy ... | 56.8 | 158.1 | 211.7 | 16 | 8 | 7 |

* K soluble in 0.3N-HCl.

Autumn nitrogen for Italian ryegrass

Autumn N, applied on 14 November as ammonium sulphate or calcium nitrate, was much inferior to equivalent spring dressings applied on 5 February for Italian ryegrass grown on a light sandy loam and for a similar ley on a clay-loam soil. Ammonium sulphate and calcium nitrate behaved similarly. Yields of dry grass harvested on 5 April were: 2.1 cwt./acre without N, 4.1 cwt./acre from the November dressing and 8.8 cwt./acre from the February dressings. (Widdowson and Penny.)

Comparisons of solid nitrogen fertilisers with solutions

An experiment at Rothamsted on newly sown Italian ryegrass tested ammonium sulphate, ammonium nitrate and sodium nitrate applied at 0.5 and 1.0 cwt. N/acre as solids and as solutions containing 5% N. Solid ammonium sulphate gave higher yields of dry grass and higher N uptake than the same fertiliser applied in solution. Differences between yields from solid and liquid forms of the other two fertilisers were not significant. (Nowakowski and Penny.)

The effects of nitrogen fertilisers on the forms of soluble nitrogen in grass

Work on the accumulation of various forms of soluble nitrogen in grass after manuring with different N fertilisers was continued, and a field experiment on Italian ryegrass at Rothamsted confirmed previous results. Grass receiving ammonium nitrate and sodium nitrate contained much more $\text{NO}_3\text{-N}$ than grass supplied with ammonium sulphate; 10 days after applying ammonium nitrate grass contained 0.92% $\text{NO}_3\text{-N}$ in the dry matter and after ammonium sulphate 0.56%. Grass given ammonium sulphate contained more $\text{NH}_4\text{-N}$, amide-N and α -amino-N than grass given ammonium nitrate or sodium nitrate. One month after applying the dressings, all the $\text{NO}_3\text{-N}$ contents of the grass had decreased, but the difference in $\text{NO}_3\text{-N}$ level between grass receiving nitrate fertiliser and grass receiving ammonium sulphate became greater. Grass grown with solid fertilisers and with solutions had similar $\text{NO}_3\text{-N}$ contents 10

days after the fertilisers were applied, but 3 weeks later grass given solid forms had twice as much nitrate as grass given solutions. (Nowakowski.)

Comparisons of potassium fertilisers

Comparisons of muriate of potash with potassium sulphate and potassium bicarbonate begun in 1959 (*Rep. Rothamst. exp. Sta.* for 1959, p. 51) were continued. At Woburn all forms of K lessened potato yields, which were less with the double dose of K than with the single. The losses were least with bicarbonate and greatest with muriate, which depressed yields by as much as 3 tons/acre. At Rothamsted K manuring increased yields considerably. Two levels of N were tested; with 0.75 cwt. N/acre, muriate was the best form of K, but with 1.5 cwt. N/acre, the double dose of potash lowered yields—muriate by 3½ tons/acre and sulphate by nearly 1 ton/acre. Bicarbonate did no damage; but it was inferior to the other two forms of K at the low rate of N; at the high rate of N it gave lowest yields with K at the lower rate, but it gave the highest yield at the higher K rate. (Widdowson and Cooke.)

Rothamsted reference plots

A micro-plot rotation experiment was established in spring 1956 on Great Field IV after ploughing very old grass. Responses to N, P, K and to FYM are measured on a five-course rotation of wheat, kale, barley, clover-grass ley and potatoes. The standard factorial lay-out testing all combinations of N, P and K (eight plots) was supplemented with four additional treatments testing double dressings of N (with PK), and also FYM with and without fertilisers.

Although the soil had long been under grass and was high in total N, all crops except clover responded well to fertiliser N. Kale and permanent grass especially depended on N for high yields, and both needed the full dressing of 2.0 cwt. N/acre. Kale and potatoes were the only crops to benefit appreciably from P fertiliser. Wheat, clover and potatoes all gave large responses to K. Only barley gave regular and similar responses to N, P and K. FYM improved yields of all crops, even when NPK was also applied, and potatoes, kale and clover responded particularly well.

A similar experiment was begun in 1960 on old arable soil on Stackyard Field, Woburn; sugar beet and oats replace kale and winter wheat, and there is a strip of plots with soft fruit bushes. All crops except clover gave very large responses to N in 1960, P and K increased yields only little. (Widdowson, Penny, Cooke and Williams.)

Phosphate fertilisers

Previous work, mostly in collaboration with N.A.A.S. Soil Chemists, valued fertilisers that could serve as alternatives to superphosphate for annual arable crops and for grass. On very poor soils, and with crops that are specially sensitive to P-deficiency, the "starter" action of phosphate for young plants may be very important. For such quick action, water-soluble phosphates have obvious advantages over insoluble materials, but we know little

about *rates of action* of fertilisers that are insoluble, or only partly soluble in water. Nor do we know enough about residual effects of alternative phosphate fertilisers, and these are at least as important as immediate effects where the main value of new dressings is to maintain reserves of soil P. Experiments were therefore started both on rates of action and on residual values of a range of phosphate fertilisers.

Rates of action of phosphate fertilisers

Micro-plot experiments on ryegrass grown on acid soils were done in 1959 and 1960 to test superphosphate, dicalcium phosphate, potassium metaphosphate, basic slag, Gafsa rock phosphate and three granular nitrophosphates having none, one-quarter and one-half of their total P water-soluble. Grass sown in 1959 grew slowly because of drought and was not cut until April 1960. There were good responses to superphosphate and dicalcium phosphate, both of which gave equal yields; none of the materials tested was significantly different from these two standard fertilisers. Grass sown in 1960 grew more rapidly, and the first harvest was taken after 14 weeks. Powdered dicalcium phosphate dihydrate gave higher yields than granular superphosphate; all the phosphates tested (except potassium metaphosphate) gave significantly less grass than did dicalcium phosphate. Gafsa rock phosphate and basic slag were significantly inferior to superphosphate. Nitrophosphate with no water-soluble P, gave less grass than the other nitrophosphates.

The second harvest, 22 weeks after sowing, showed dicalcium phosphate and superphosphate to be nearly equal; Gafsa rock phosphate and the nitrophosphate with low water-solubility had both improved and gave significantly more grass than superphosphate. The total yield for the two cuts showed powdered dicalcium phosphate to be significantly better than granular superphosphate, which, in turn, was not significantly different from any of the other phosphates tested. This experiment showed the expected differences in rates of action. Rapid action, illustrated at the first cutting, was obtained from water-soluble materials and from finely powdered dicalcium phosphate (perhaps because it had a high surface area), but the slower materials were more effective later and, over the whole season, differences between materials were less than at first. The consistent superiority of powdered dicalcium phosphate dihydrate over granular superphosphate was significant in these 1960 experiments. In earlier work the two materials were more nearly equivalent. (Mattingly and Penny.)

Residual values of phosphate fertilisers

In two experiments designed to last 6 years and started in 1959, heavy dressings (3.0 cwt. P_2O_5 /acre) of the phosphate fertilisers listed in the previous section were mixed with surface soil by ploughing and rotary cultivating in autumn/winter 1959. The first crops were sown in 1960, when new small dressings of superphosphate were applied to provide a scale for measuring residual effects of the larger dressings applied in 1959. Residues from 3 cwt. P_2O_5 /acre given as superphosphate in 1959 gave larger yields of swedes,

potatoes and barley in 1960 than a new dressing of 0.5 cwt. P_2O_5 broadcast before sowing. Gafsa rock phosphate residues gave significantly less potatoes than superphosphate residues, but the other phosphates were not significantly different from superphosphate. For swedes all the fertilisers tested had similar residual effects. (Yields given by nitrophosphates in 1960 were not a good indication of the value of the phosphate, because these are NP fertilisers and about 3 cwt. of extra N/acre had to be applied with the P dressing and this had not leached completely from the soil by summer 1960.) (Mattingly.)

Urea

Urea is potentially valuable as a fertiliser because it is highly concentrated and cheap to make, but experiments in the last few years have shown that it may have disadvantages. Some samples, particularly of granulated products that have been heated in processing, may contain biuret, which is toxic. The other disadvantage, common to all samples, is that when urea decomposes rapidly ammonium carbonate or bicarbonate is formed, and the pH near to the fertiliser particles rises. High pH and high ammonia concentrations near to young seedlings may damage them—this happened in barley experiments reported last year. The same decomposition may also lead to loss of N (as NH_3 gas) (and therefore reduced efficiency) when urea is applied as a top-dressing for grass or arable crops, or when it is not buried deeply enough in a seedbed.

Losses of nitrogen from urea applied to soils

In laboratory experiments, urea equivalent to 0.9 cwt. N/acre was applied in different ways to several soils at each of several water contents and at 5° and 25°. Air was passed over the soils and the NH_3 lost was measured. Losses were more serious from light than from heavy soils. The least loss of N (<1%) was from heavy soil at 5°, the greatest (20%) from light soil at 5°. Except with a light soil containing little organic matter, losses were less when urea was mixed with the surface inch than when it was broadcast on the soil. Changes in moisture content of soil within the range 40–60% of the water-holding capacity generally made little difference to losses of NH_3 , but again the poor light soil was an exception, at 25° much more NH_3 was lost from the driest sample. Temperature had little effect on total losses of NH_3 but they occurred much more quickly at 25° than at 5°. The exception was again the light soil with low organic matter, from which twice as much NH_3 was lost at 5° as at 25°. (Gasser.)

Effects of biuret and urea on germination

The effects on germination of kale of samples of urea having different biuret contents were measured in a field experiment. The fertilisers were tested at 0.9 and 1.8 cwt. N/acre, both broadcast on the surface and placed immediately under the seed, which was sown in rows 9 inches apart. Germination percentages, averaged for the rates and methods of application and taking the plants established on unfertilised plots as 100%, were: granular urea (6% biuret) 56%, prilled urea (1.5% biuret) 85%, crystalline urea (0.6% biuret)

71%, pelleted urea (0.5% biuret) 82%, ammonium sulphate 73% and calcium nitrate 91%. Only the granular urea was more dangerous than ammonium sulphate or calcium nitrate. Damage was greater at the higher rate of dressing and also when the fertilisers were placed directly under the seed. (Gasser and Penny.)

In a pot experiment with kale high rates of urea usually depressed germination, with no difference between the four samples; germination was higher in moister soil. Yields and total-N uptakes at harvest were proportional to biuret contents, and were least with the sample containing most biuret. (Gasser and Close.)

EXPERIMENTS IN FOREST NURSERIES

Long-term rotation experiments

In 1951 two long-term rotation experiments of about 350 plots each, cropped with Sitka spruce seedlings or transplants, were laid down in nurseries at Wareham (Dorset) and Kennington (Oxford). (Their design and lay-out is described in *Rep. For. Res. For. Comm.* for 1952-53, pp. 84-100.) The experiments compare continuous conifer cropping with a rotation in which one conifer crop in three is replaced by either bare fallow or a "green" crop (rye, ryegrass or yellow lupins); they also compare annual applications of compost made from bracken and hop-waste with a mixture of "Nitro-Chalk", superphosphate, KCl and MgSO₄. Over eight seasons there has been no advantage, at either centre, from interrupting continuous cropping with conifers by any of the three green crops, but at Kennington bare fallow improved growth. At both centres fertiliser-grown seedlings have been 20-30% larger than those having compost.

In a supplementary experiment on continuously cropped seed-beds, compost and fertiliser each increased seedling height; at Wareham the unmanured crop failed completely. At Kennington compost and fertiliser applied together were no better than fertiliser applied alone, but at Wareham the two manures together produced larger plants than either material applied alone. This result might indicate that compost had other functions besides supplying nutrients, but there is no present evidence that this is so. The fertiliser-grown plants suffered from Cu deficiency (*Nature, Lond.* (1956), 178, 864), and in several seasons also showed K-deficiency symptoms. During wet seasons compost plots, as well as fertiliser-treated plots, pass through periods of nitrogen deficiency, and these periods are rarely coincident on the two kinds of plots. Therefore plants grown with compost plus fertiliser have received a steadier supply of N, and have been less subject to either K or Cu shortages. The experiments are being modified to eliminate, as far as possible, differences that may come from lack of nutrients in the fertiliser. (Benzian.)

The problem of "worn-out" nurseries

In some of the Forestry Commission's nurseries (mostly those started on farming land between 1920 and 1940) some conifers, such as Sitka spruce, *Tsuga heterophylla* and *Pinus contorta* remain small and stunted, even with ample plant nutrients applied either as

compost or fertiliser. This stunted growth normally occurs on soils with high pH, but there are exceptions, such as failures on acid soil at Ringwood. In several nurseries stunting is associated with root damage caused by fungi and, at Ringwood, with root-parasitic nematodes as well. Root damage of either kind is greatly decreased by "partial sterilants" such as formalin and chloropicrin; but these materials are also of benefit where no effects from pests and pathogens have yet been recognised. Improvements have even been observed in highly productive nurseries like Wareham, where growth seems normal.

It is difficult to isolate the factor or factors responsible for stunting and for the dramatic improvement in growth with "partial sterilisation"; members of several departments at Rothamsted are working together on the problem. Materials like steam, formalin and chloropicrin not only profoundly change the soil flora and fauna, but chemical changes in the soil may alter nutritional conditions, such as producing and maintaining a higher concentration of ammonia. "Partial sterilants" and acidifying agents both tend to increase the amount of Mn in seedlings, and the untreated plants may well suffer from Mn and/or Fe deficiency. Mn as well as Fe chelates have been tested as foliar sprays, but so far without success in the nurseries, and with only very slight benefit from Mn chelate in pots.

As stunting is confined to older nurseries, it is important to discover whether, with modern manuring, cultivation and weed control, nurseries can remain fertile indefinitely. The two long-term rotation experiments mentioned above will indicate problems that may come from intensive conifer cropping. Although during the first eight seasons, there were no signs of deterioration in growth resembling the troubles encountered in "worn-out" nurseries, size (1959) and plant number (1960) were much decreased on many compost-treated plots at Kennington. In 1959 the Wareham plots suffered from an attack of *Fusarium* wilt, which was much more severe with compost than with fertiliser. The future will show if these failures are isolated occurrences or if they are the first indications of a gradual deterioration. (Benzian.)

Tests on soil diluents

Most recent work on partial sterilisation has been done at Ringwood and Old Kennington Nurseries; at both, but particularly Ringwood, poor soil structure is likely to have contributed to stunted growth. Close packing may interfere with supplies of water and air, as well as with drainage, and may obstruct growing roots and so favour attacks by parasites. To open up the soil, two materials were incorporated into the top 6 inches a few weeks before sowing: 12 lb./sq. yd. of coarse-grade sedge peat and 100 lb./sq. yd. of quartz grit from St. Austell. At Old Kennington the diluents were tested together with formalin; at Ringwood bad weather made this impossible. In the Old Kennington experiment, peat alone increased height by 40% and formalin alone by 70%. With formalin, peat improved growth only slightly; and with peat, the response to formalin was lessened. Bad physical conditions may have aggravated attacks by pathogens. However, in the very

wet 1960 season part of the benefit from peat may have been from a slow release of N. At Kennington there was no response to St. Austell grit. At Ringwood peat improved growth but, in contrast to Kennington, the nutritionally inert quartz grit gave a slight benefit. (Benzian.)

ANION-CATION RELATIONSHIPS IN CROP NUTRITION

Uptake of nutrient cations by crops is usually decreased by other cations present in the soil; similar effects exist with anions. Uptake of positive (or negative) ions is also affected by the presence of ions of opposite charge. These relationships are particularly important when heavy dressings of fertilisers are used, and they are more pronounced on light than on heavy soils. Such effects alter the efficiencies of nutrients supplied as fertilisers, affect the use that plants make of nutrients in soil and may influence the quality of crops. As there is little systematic information, work began in summer 1960 to obtain information about ion relationships. Micro-plot field experiments done with ryegrass and kale on heavy soil at Rothamsted will extend to light soil at Woburn in 1961. The effects of combinations of fertiliser salts on crop composition are being examined. (Cunningham.)

NITROGEN IN SOILS

Using the mass spectrometer for determining ^{15}N

Almost all of Newman's time was spent in making the mass spectrometer (*Rep. Rothamst. exp. Sta. for 1959*, p. 41) work satisfactorily and in establishing a service for determining ^{15}N in soils and plant materials. The instrument has been used effectively since May 1960.

The steps in the analyses of the samples are: (i) Kjeldahl digestion; (ii) distilling the digest from alkali and then titrating with acid; (iii) converting ammonium salt solution to nitrogen gas; and (iv) analysing the gas sample in the mass spectrometer. Each step can lead to errors in the final ratio of $^{15}\text{N} : ^{14}\text{N}$ and each has been examined to make the total error small.

Some workers state that Kjeldahl digestion can lead to an impurity (possibly methylamine), which gives ions of mass/charge ratios of 45, 31 and 29; this is particularly likely with Cu-Se catalysts (which we generally use), but it never happens with Hg catalyst. During the mass spectrometric analysis of N, the ratio of ions 28 ($^{14}\text{N}_2^+$) to 29 ($^{14}\text{N}^{15}\text{N}^+$) is measured, and the impurity leads to an over-estimate of ions $^{14}\text{N}^{15}\text{N}^+$, giving an erroneously high ratio of $^{15}\text{N} : ^{14}\text{N}$. Samples from incubation experiments with soil and straw (described below) gave this impurity (which was diagnosed by ions produced at mass 31), and it prevented reliable estimates of ^{15}N in these samples. In further work soils alone were digested with Cu-Se, and with Hg catalysts and soil-straw incubations were digested with Cu-Se catalysts. No trace of this impurity was found in any sample, and $^{15}\text{N} : ^{14}\text{N}$ ratios were normal. It is considered the "impurity" was caused by too short digestion time.

During distillation of the digests a sample high in ^{15}N may leave

a micro-amount of ^{15}N , which may contaminate a following sample having normal ^{15}N content. In distillation tests where appreciable contamination occurred only with 14% of ^{15}N , distilling several blanks eliminated the effect.

Converting NH_4 solutions to N_2 gas is tedious, as all air must be removed from the solutions to prevent diluting the ^{15}N content in the sample. Only small quantities of N_2 are needed for analysis, and good high-vacuum technique is essential; we have been able to convert only five or six samples per day. A simpler technique is now being tested.

Consistent results are obtained only by standardising operations rigidly. Calibrating the resistance chain on which the final ratio depends showed maximum error of about -11% and ratios are now corrected to allow for this. The instrument shows "mass discrimination" errors and tends to over-estimate the lighter mass ions at the expense of the heavier; this over-estimate increases as the sample is consumed during analysis. Extensive modification would be necessary to lower this mass discrimination; but for the ratio 28 : 29 the effect is small enough to be neglected, the ratios obtained from the Kjeldahl experiment described above were examined statistically and found to be accurate enough for determining small enrichments with ^{15}N . For greater enrichments of ^{15}N , the few results show errors of $\pm 5\%$; more work is being done to improve this.

For other mass ratios, e.g., N_2 to O_2 , or N_2 to A (both necessary for correcting nitrogen samples for the presence of air), the results have not been very consistent; apart from the mass discrimination effect mentioned above, N_2 : O_2 has varied from 5.5 to 6.1 (the volume composition of air gives 3.7), and although it is not unusual for mass spectrometers to give a value for this ratio of 6, its variability makes correcting for air in the samples rather uncertain. Using the instrument for gas analysis is also hindered by this inconsistency.

With an assistant preparing gas samples from NH_4 solutions, we have been able to analyse 20-25 samples each week. 200 samples have now been analysed, over half of which were for members of the Soil Microbiology Department. (Newman.)

Non-symbiotic fixation of nitrogen

Detectable amounts of N can be fixed when soil is incubated with much (13%) added straw carbon (for example, J. M. Bremner, *J. agric. Sci.* 1958, **51**, 23). Using ^{15}N in the mass spectrometer measures N fixation more sensitively than the Kjeldahl method used by Bremner, and work was done to see whether there is any fixation with small amounts of straw under aerobic and anaerobic conditions. Straw (1% addition of straw carbon) was mixed with calcareous soil low in organic matter, and the soil alone, soil + straw, and soil + straw + KNO_3 , were incubated for 42 days in atmospheres containing N_2 gas enriched with ^{15}N . The 250 p.p.m. of $\text{NO}_3\text{-N}$ added more than sufficed for the microbial population attacking the straw and (under aerobic conditions) $\text{NO}_3\text{-N}$ did not fall below 67 p.p.m., in contrast to treatments with straw alone, with which the initial $\text{NO}_3\text{-N}$ of the soil (15 p.p.m.) fell to zero in

20 days and did not increase again. Four atmospheres were used: aerobic with removal of CO₂, aerobic allowing CO₂ to accumulate, anaerobic with removal of CO₂ and anaerobic allowing CO₂ to accumulate. The nitrogen gas contained 6 atom % excess ¹⁵N, although in a few experiments a 20 atom % excess was used. Total N in the incubated soils, as determined by Olsen's modification of the Kjeldahl procedure, showed no fixation in any of the treatments; in the anaerobic treatments NO₃ had been quantitatively lost in contrast to the aerobic treatments, where there was no loss of added NO₃. Determining ¹⁵N in the Kjeldahl distillates was difficult because of an unidentified contaminant discussed above (possibly methylamine), which increased the height of the ¹⁵N peak in the mass spectrometer. Some samples therefore showed an *apparent* fixation of nitrogen which was not caused by an excess of ¹⁵N. But "fixation" (real + apparent) was never greater than 10 p.p.m. of N. (Jenkinson, Birecki, Newman.)

Some other factors governing non-symbiotic fixation of N are being investigated. The amount of N fixed when straw was added to water-logged soil under anaerobic conditions increased linearly with straw additions of 2.5–20%, were maximal when 30% was added and fell with larger additions. The maximum fixation (50 mg. N/100 g. of soil or 1.7 mg. N/g. of straw) was the same in old arable soil and old grassland soil. (Jenkinson and Barrow.)

The effects of 1-year leys and fertilisers on soil nitrogen status

A field experiment (done by Widdowson and Penny and described above, p. 52) measured the effects of growing clover or ryegrass on the N needed by following wheat. Soil samples from this experiment showed differences in N status. When the leys were ploughed in autumn plots where clover had grown contained more mineral-N, and more N that could be mineralised in laboratory incubations, than plots where ryegrass had grown; giving N-fertiliser to the ryegrass raised the N residues left in the soil. These differences in N-status disappeared by the following spring.

In another field experiment (*Rep. Rothamst. exp. Sta.* for 1958, p. 46) urea-formaldehyde (UF) fertiliser was compared with "Nitro-Chalk" (NC); UF was much less efficient than NC for 2 years of cropping with grass, presumably because it did not decompose quickly or completely enough. In the following spring, when cultivations were complete, levels of mineral N in the soil were higher on plots fertilised with UF than on unfertilised plots. Increases (in p.p.m.) of NH₄-N + NO₃-N on incubating the air-dry soils were:

| Treatment in the field experiment | | | Mineralisable N | |
|-----------------------------------|------|----|-----------------|-------------|
| 1958 | 1959 | | Autumn 1959 | Spring 1960 |
| — | None | NC | 36 | 39 |
| — | None | UF | 41 | 41 |
| NC | — | — | 52 | 50 |
| UF | — | — | 43 | 38 |
| NC | NC | — | 47 | 41 |
| UF | UF | — | 41 | 39 |
| | | | 62 | 54 |
| Significant difference (P < 0.05) | | | 8.7 | 9.1 |

Inorganic N fertiliser had little effect on the N reserve of the soil, but applying UF in spring 1959 significantly increased the mineralisable-N in both autumn and spring 1959/60; UF given to plots in 1958, with none in 1959, had much less effect on the soil than the 1959 dressings. (Gasser.)

SOIL PHOSPHATE

Losses of phosphate from sandy soil

In a field experiment on very light and acid (pH 4.5) sandy soil in the Forestry Commission's Research Nursery at Wareham (Dorset) several phosphate fertilisers equivalent to 16 mg. of P/100 g. of soil were applied between 1955 and 1959. Samples taken at the beginning and end of the period showed the following changes in total P contents and in isotopically exchangeable P ("A" values) measured in pot experiments on ryegrass:

| Annual treatment | Changes from 1955 to 1959 | |
|-----------------------------|---------------------------------|-----------|
| | Total P mg. P/100 g. of soil | "A" value |
| No phosphate | -1.0 | -0.1 |
| Superphosphate | -0.4 | +1.1 |
| Basic slag | +4.3 | +4.2 |
| Gafsa rock phosphate | +9.8 | +3.8 |

Yields of grass in pot experiments on the soils were highest with basic slag and rock phosphate and much less with superphosphate; differences in favour of the less-soluble materials were much greater in 1959 than in 1955. The total P content of the soil, 5.6 mg./100 g. in 1955, declined during the period on the "no phosphate" and the superphosphate plots, but rose on the basic-slag plot by one-half, and on the plot receiving rock phosphate it doubled. All the superphosphate not used by the crops grown had leached out in the 4 years, and three-quarters of the P applied as basic slag and half of that added as rock phosphate had also probably been lost. Rock phosphate and basic slag clearly have much greater residual values than superphosphate.

TABLE 7

Changes in P content of sand and silt fractions between 1955 and 1959 (in mg. P/100 g. of soil)

| Particle size range (microns) | Phosphate applied as | | |
|----------------------------------|----------------------|-------------------|---------------|
| | Super phosphate | Rock phosphate | Basic slag |
| 2,000-200 | +0.3 | +2.0 | +0.5 |
| 200-75 | -0.5 | +5.0 | +0.7 |
| 75-20 | -0.3 | +1.3 | +0.9 |
| 20-5 | +0.9 | +1.3 | +1.3 |
| Total | +0.4 | +9.6 | +3.4 |

Measuring the total P contained in four size fractions of the soil separated by sedimentation (Table 7) showed little change from adding superphosphate, but considerable changes from adding the other phosphates, and particularly rock phosphate. Three-quarters

of the total P accumulated in the rock phosphate plots was in the coarse and fine sand fractions. All the fractions were enriched by basic slag, but the fine material more than the coarse.

Distribution of phosphate was also measured in an acid clay soil at Rothamsted used for a field experiment testing super-phosphate and rock phosphate (described in *Rep. Rothamst. exp. Sta.* for 1959, p. 49). Most of the extra phosphate supplied by super-phosphate was in the clay and silt. Phosphate had accumulated in the fine sand (200–75 μ) fraction only on plots treated with rock phosphate. This confirms the result at Wareham and suggests that rock phosphate accumulates in acid soil as undissolved particles. (Mattingly.)

Fractionating inorganic soil phosphorus by ion-exchange resins

The rates were measured at which Ca, Al, Fe and P are removed from soil (<40 mesh) in 10% aqueous suspension by Cl⁻-saturated Amberlite IRA-401 (20–30 mesh) and Na-saturated Zeo-Karb 225 (10–20 mesh) exchange resins. (The resins were rapidly and completely separated from soil by stainless-steel sieves.) Varying the "wet resin" : soil ratio suggested an optimum weight ratio of 1.5 : 1 for both anion- and cation-exchange resins to remove most phosphate ions and cations from the soil at equilibrium. In IRA-401 (AER) and Zeo-Karb 225 (CER) respectively this weight ratio is equivalent to average values of 55 : 1 and 35 : 1 for the ratio of the total exchangeable ions in the resin to those in the soil. Phosphate was not recovered completely from AER by successive extraction with 0.01M–2.0M solutions of Na₂SO₄, NaCl and NaOH (even on boiling), and the resin had to be incinerated at 550° before analysis. Before extracting with resins, the soils were brought to equilibrium with carrier-free ³²P for 320 hours to label the isotopically exchangeable phosphate uniformly. The rate of change of the specific activity of P removed from soil by resin was measured.

Soils provided by Mr. J. W. Blood (N.A.A.S., East Midlands Region) in 1958 from unmanured and phosphate-treated sections of a field experiment at Shardlow (Nr. Derby) were examined. The phosphate extracted from soils that had been maintained in the field at pH 4 or pH 6, with CER or AER, had a constant specific activity at all times, indicating that each extracting mechanism removed only part of the isotopically exchangeable phosphate (P_e). The amount removed in 1 day was directly proportional to P_e , and was larger with the soil of higher pH. Equilibrium was reached rapidly with CER, but with AER 12 days contact was required. The fraction of P_e extracted by AER increased only slightly with increase in soil pH (which remained constant during extraction). By contrast, CER extracted approximately twice as much of the total P_e from soil with pH 6 than from soil with pH 4, and during extraction with this resin the pH rose by 1–1.5 units. Table 8 shows that the total phosphate ions removed by AER plus CER are equal to the "rapid" isotopically exchangeable phosphate " P_r ". Therefore phosphate ions in " P_r " are held: (a) as anions compensating positive charges on the soil, and (b) as counter ions associated with cations in the ion atmosphere surrounding the soil colloid. The proportion of P_r held on positively charged sites diminishes

with increasing soil pH, and a larger proportion appears as counter ions in the micellar liquid.

The specific activity of phosphate extracted by a mixture of AER plus CER falls slowly after several days' reaction with soil, indicating that non-isotopically exchangeable forms of phosphate are extracted together with P_e . When completed this work will clarify the nature of slowly exchanging and non-exchangeable forms of soil phosphate and their relationships with soil pH.

TABLE 8

Phosphate removed by AER and CER from two Shardlow soils in 1 day and the isotopically exchangeable phosphate in mg. P/100 g. of soil

| Field treatment | Soil of pH 4.2 | | Soil of pH 5.9 | |
|----------------------------------|----------------|------|----------------|------|
| | No P | P | No P | P |
| <i>Phosphate removed</i> | | | | |
| By Cl ⁻ -AER ... | 3.7 | 6.3 | 5.1 | 6.9 |
| By Na ⁺ -CER ... | 1.9 | 3.7 | 6.5 | 9.0 |
| Total with resin | 5.6 | 11.0 | 11.6 | 15.9 |
| <i>Isotopically Exchangeable</i> | | | | |
| P_r | 8.3 | 12.0 | 11.3 | 16.7 |
| P_e | 17.6 | 26.0 | 28.8 | 35.3 |

With partially unsaturated CER ($\frac{H}{Na + H} = 0.11$), either alone or mixed with AER, non-exchangeable phosphate was removed from soil within $\frac{1}{2}$ -1 hour of contact, and soil pH was lowered by 1-2.5 units. (Vaidyanathan and Talibudeen.)

" Available phosphate "

About 180 individual soils, taken from sites of field experiments done between 1951 and 1956 to test P fertilisers, were analysed by seven methods for " readily soluble " P. None of the methods was outstandingly successful in predicting crop responses to P fertilisers. The best methods were those using a slightly acid acetate buffer solution, a solution of 0.5M-NaHCO₃ or extraction with 0.01M-CaCl₂ solution. Methods using acetic, citric, sulphuric or hydrochloric acids were less useful. Partial regression coefficients (calculated by J. H. A. Dunwoody) showed that analyses by an individual method rarely accounted for more than half of the variation between the effects of P fertilisers on crop yields; there was little improvement when the values for soluble P determined by two or more methods were used together. The soils used for the field experiments had all been selected as being P-deficient, so they did not extend over the normal practical conditions of extreme shortage to a large surplus of soluble soil P; nevertheless, the soils used ranged from those where arable crops failed without added P fertiliser to those where P fertilisers did not increase yields. This work shows that no close relationship between soluble P determined by a chemical method and uptake of soil P by crops can be expected within the steeply rising part of the curve relating chemical measurements of soluble P to uptake of soil P by crops. Weather, and soil

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structure and depth, must affect the accessibility of soil P to roots; pests and diseases may also influence yield and response to P fertiliser. Many similar investigations have produced disappointing correlations between soluble P in soils and effects of P fertilisers on the crops grown, and have tended to discredit soil analysis as a tool for advising on P manuring. Too much attention has been given to erratic relationships between low values for soluble P and the uptake of soil P by crops, and too little to the fact that the better methods of analysis generally predict very well the richer soils where crops obtain enough P without added fertilisers. Future work should aim to establish limiting values for soluble P in soils above which responses to extra P will not be expected, and where P fertilisers will be needed only to maintain reserves. Crops grown on soils with less soluble P will need normal P manuring. (Williams and Cooke.)

All methods used in this country to measure soluble P for advisory purposes are empirical. The better ones often succeed because of compensating errors; they measure a quantity of P that combines the "intensity" of immediately available P that is ionised and the "pool" of useful P that could ionise and which acts as a reserve (the terms were defined by R. K. Schofield (*Soils & Fert.* 1955, **18**, 373-375). In the early stages of growth a crop must depend on the ionised P expressed by the intensity factor, and there is a good argument for using this quantity as a measure of "available" P (and neglecting the reserve in the "pool") for immediate advisory purposes. Schofield gave a theoretical basis for using the amount of soil P in equilibrium with 0.01M-CaCl₂ solution as a measure of P intensity. This method is therefore being examined for practical limitations or difficulties, and to see if the values are stable throughout the year, and whether they are affected by variations in laboratory techniques. (Cooke, Blakemore and Mattingly.)

Soils from old arable fields where FYM has not been applied within the last 50 years were sampled at different periods during the year. When under fallow or cropped with a cereal, changes in NaHCO₃-soluble P within the range 0.3-1.5 mg. P/100 g. of soil show changes of less than 10% during the year. (Warren and Johnston.)

Relationship of soluble P and total P

Soils from the sites of four Classical Experiments (Agdell, Hoosfield Permanent Barley, Barnfield and Exhaustion Land) show that "soluble" P increases with "total" soil P. The increase is slowest for Agdell and fastest for the Exhaustion Land. ("Soluble" P was determined with 0.5M-NaHCO₃, and "total" P by the perchloric acid method.) With 30-40 mg. "total" P/100 g. of soil, "soluble" P would probably be negligible; the insoluble P includes the stable organic P in soil. (The temperature of the NaHCO₃ solution affects the amount of P dissolved, and must therefore be kept within $\pm 1^\circ$ of a selected temperature.) (Johnston.)

SOIL POTASSIUM

Categories of soil potassium

In continued glasshouse cropping the amounts of non-exchangeable K released from many soils tend to become directly proportional to time. Extrapolating this rate of release of "native" K to zero time estimates the total uptake of K from other sources. In most soils examined this estimate was much larger than the fall in exchangeable K, showing that a second category of non-exchangeable K contributes to uptake by crops. This category of "loosely held", or "fixed", K can be depleted rapidly; it is not known whether the K is released from vermiculite-type minerals which have fixed K or whether it is from some of the more highly weathered planes in hydrous micas which rapidly lose their remaining inter-layer K and expand when exchangeable K is lost. Because the estimated releases of "loosely held" K increased in many soils with the total K contents, and with the amounts of fine-clay, highly weathered hydrous micas may be important sources of this fraction.

Rothamsted soil releases "native" K very slowly, and the "loosely held" K may be largely derived from accumulated residues of K-fertilisers held in vermiculite-type minerals. During exhaustive glasshouse cropping, soils from the Agdell Rotation Experiment released non-exchangeable K at rates that were linearly related to the initial exchangeable K values; this indicates that an equilibrium is established between exchangeable K and "loosely held" K. Ratios for (release of non-exchangeable K) : (fall in exchangeable K) with Agdell soils are about 2.6 for top-soils, and 3.8 for subsoils, whether or not potash fertiliser has been applied. (Arnold and Close.)

Effects of time of sampling and previous cropping on soluble potassium

Soils from old arable fields where FYM had not been given within the last 50 years have been examined at different times of the year and in different years. Under fallow values for soluble K do not vary significantly during the year, or from year to year. On soils with little soluble K, crops with moderate K requirements (cereals) change soluble K little; crops needing much K (kale, swedes, sugar beet) decrease the soluble K values so much that succeeding cereals showed K deficiency symptoms when young. This decrease is only temporary, and soluble K returns nearly to the initial level during the year. On soils having "medium" soluble K the values do not return to their initial levels after growing crops that need much K, but the decrease is less than the amount of K removed by the crop. (Warren and Johnston.)

Measuring "readily-available" potassium

The K status of soils was assessed by measuring the K concentration in dilute solutions of CaCl_2 shaken with soil. For calcareous Rothamsted soil cropped with perennial ryegrass in the glasshouse the uptakes of K in the first harvest increased steadily as the free energy of exchange (calculated for the replacement of K by Ca) in the soils decreased over the range 4,300–3,100 calories/g. equivalent.

Yields of grass were greatest when the energy of the K-Ca exchange was less than about 3,700 calories. K uptakes by the grass were better correlated with the energies of exchange than with the exchangeable K contents of the soils.

Cotswold soils, examined in collaboration with Mr. R. D. Russell (N.A.A.S., South-western Region), usually have values for exchangeable or "easily-soluble" K that are poorly correlated with the field performance of crops. Crops on many of the soils yield badly and give little response to K fertilisers; equilibration tests using 0.01M-CaCl₂ as the "blanketing" electrolyte gave energies of replacement of K by Ca in the range 4,500-3,900 calories. By comparison with Rothamsted soils these Cotswold soils are very deficient in K. In laboratory tests dressings equivalent to 3 cwt./acre of KCl mixed with the Cotswold soils did not change the energies of exchange of K by Ca to levels that favour uptake of K by plants. This may explain the poor response of such soils to K-fertilisers; larger dressings of K fertilisers will be required, or the dressings that are used should be localised near the roots of crops. With some soils having high Ca status and high exchangeable K values, equilibration with dilute electrolyte underestimates the abilities of the soils to supply K to ryegrass in the glasshouse; until such anomalies can be explained the technique cannot be applied generally. (Arnold.)

THE VALUE OF PHOSPHORUS AND POTASSIUM RESERVES IN SOILS OF THE LONG-TERM EXPERIMENTS

Agdell Rotation Experiment

The effects of the residues of P fertilisers, accumulated in the soil during the main period of this Experiment (1848-1951), on yields of barley, potatoes and sugar beet were again measured. Responses to fresh dressings of superphosphate in Table 9 are means from experiments in 1959 and 1960. The three crops responded to new

TABLE 9

Mean crop responses to new dressings of superphosphate supplying 1.0 cwt. P₂O₅/acre on the site of the Agdell Rotation Experiment

| Manuring and rotation treatment in old experiment | Barley grain (cwt./acre) | Potatoes (tons/acre) | Sugar beet roots (tons/acre) | P soluble in 0.5M-NaHCO ₃ mg./100 g. soil |
|---|--------------------------|----------------------|------------------------------|--|
| None { Clover | 12.7 | 4.8 | 2.7 | 0.4 |
| | Fallow | 6.5 | 5.9 | 3.8 |
| PK { Clover | 4.4 | 4.2 | 1.2 | 0.8 |
| | Fallow | 2.5 | 2.2 | 0.5 |
| NPK { Clover | 1.1 | 2.8 | 0.7 | 1.2 |
| | Fallow | 0.9 | 4.0 | 1.3 |

dressings of superphosphate in the same way as in experiments on the Exhaustion Land in 1957 and 1958. Barley and sugar beet used residues of P fertilisers that had accumulated in the soil during the main Rotation Experiment (1848-1951), and where these residues had raised the level of soil P to 1.0 mg. P/100 g. of soil (soluble in 0.5M-NaHCO₃) fresh dressings of P fertiliser gave only small

increases in yields of both crops. In contrast, potatoes needed fresh P fertiliser, even though this crop removed the same amount of P as barley and less than sugar beet.

The residual effects of a single dressing of superphosphate applied in 1959 at 1.0 cwt. P_2O_5 /acre were measured on the three crops in 1960. Table 10 gives the amounts of "new" (i.e., 1960) superphosphate that were equivalent to the residues of the 1959 dressings. These figures are derived from broadcast superphosphate response curves for the range 0.5–1.5 cwt. P_2O_5 /acre on plots where the crops responded to these new dressings. On the two soils where crops gave the largest responses to fresh P (clover and fallow sections of the old unmanured plot) the 1959 dressing had high residual values for both barley and sugar beet in 1960. (Two of the four values indicated that residues were nearly equal to a new dressing of the same amount of superphosphate, one value suggested that the residue was more effective than a new dressing.) For potatoes grown in 1960 the residues of the 1959 dressing were only about

TABLE 10

The residual values of single dressings of superphosphate applied in 1959 at 1.0 cwt. P_2O_5 /acre for crops grown in 1960 (data are cwt./acre of new P_2O_5 required to give the same yields as the residues in the soil)

| Manuring and rotation treatment in old experiment | | | | Barley | Potatoes | Sugar beet |
|--|--------|-----|-----|--------|----------|------------|
| None | Clover | ... | ... | 0.8 | 0.5 | 0.9 |
| | Fallow | ... | ... | 1.5 | 0.4 | 0.6 |
| PK | Clover | ... | ... | 0.4 | 0.4 | 0.4 |

half as effective (relative to a new dressing) as for the other two crops. Potatoes differ from other crops in ability to use old residues of P fertilisers that have accumulated in soil. This effect has also been demonstrated in work on the very old residues on the Exhaustion Land. The 1960 experiments on Agdell show that it also occurs with 1-year-old residues in the highly responsive soils of the "unmanured" plots. The soil of the clover section of the PK plot of the old Rotation Experiment has a higher P status (barley and sugar beet gave smaller responses to new P), and estimates for all crops were that the residues were equal to broadcasting a new dressing of 0.4 cwt. P_2O_5 /acre. Therefore, for barley and sugar beet grown on the richer soil, 1-year-old residues were only half as effective as residues in the poorer soils, but the residues had the same value for potatoes on all three soils.

The results of the new micro-plot experiments on Agdell in 1959 and 1960 showed several differences from the results of comparable work on the Exhaustion Land in 1957 and 1958. On each experimental field the increases in yields given by fresh P fertilisers differ in the two years; this effect was greater on Agdell, where there was the extreme behaviour of sugar beet not responding to new superphosphate in 1959 on a very P-deficient soil, but a large response (nearly 6 tons of roots/acre) in 1960. On the Exhaustion Land, but not on Agdell, a single broadcast dressing of fresh superphosphate (1.0 cwt. P_2O_5 /acre) increased the yields of crops on the P-deficient

soils to the same levels as those obtained on soils enriched with fertiliser residues and which had received the same amount of new superphosphate. Another difference between the two sets of results was in the response curves to new phosphate. On the Exhaustion Land superphosphate supplying 1.0 cwt. P_2O_5 /acre was adequate for all crops on P-deficient soils. On Agdell, however, some of the response curves in 1959 were linear up to the highest amount of P fertiliser tested (1.0 cwt. P_2O_5 /acre). In 1960 the highest rate used on Agdell was 1.5 cwt. P_2O_5 /acre, and some of the response curves still did not show a maximum.

The Agdell soil is much heavier than the Exhaustion Land soil, and this, by making broadcast fertiliser less accessible to plant roots, may be partly responsible for the differences in the results on the two fields. The very high values of the 1-year-old P fertiliser residues (in terms of new dressings) for barley and sugar beet on Agdell in 1960 are partly explained by the new dressings of P fertiliser being relatively ineffective. The conventional method of broadcasting new fertilisers is probably not satisfactory for providing P response curves that will evaluate fertiliser residues in the soil, and different methods of incorporating P fertilisers are therefore now being compared. Broadcast dressings of K fertilisers may also be unreliable, even when used as a basal manure, with crops needing much K. In the new experiments with P fertilisers planned for 1961 part of the basal dressing of K is being ploughed in, and the remainder will be applied during seedbed preparation.

Continuous Wheat and Barley sites on Stackyard Field, Woburn

A 2-year programme of micro-plot experiments was begun to measure the value of the residues of P and K fertilisers left in the soil from dressings given annually between 1877 and 1927 to the Classical Wheat and Barley Experiments. The test treatments and basal manuring were similar to those used on Agdell, but only barley and potatoes were grown. The 1960 results gave two estimates for each crop of the separate effects of the P and K residues. The first estimate was made by comparing yields on the plots of the old experiments after applying a basal fertiliser (NK in testing P residues, and NP for testing K residues). In the old experiments each treatment had only a single plot, the old manuring schemes were identical for the barley and the wheat sites except for differences in occasional light dressings of lime on a few subplots; as the two sites have been cropped and manured (with N only) in the same way since 1954, there are pairs of plots with identical manuring history for each treatment. In 1960 the plots that have residues of P fertilisers gave 6 cwt./acre more barley than the old unmanured plots, but less than 1 ton/acre more potatoes. The residues of K fertilisers had much smaller effects on barley than the P residues; on the site of the old Wheat Experiment there was no increase in yield. K residues increased yields of potatoes by 3 tons/acre.

The second estimate of the value of P and of K residues in the soils was derived from the yield-response curves obtained with new dressings of P and K fertilisers. For each crop, and each nutrient, this second estimate is higher than the first. This difference is because new fertiliser dressings did not increase yields on the old

unmanured plots to the level of yields obtained where the same new dressings of fertilisers had been given to old fertiliser plots; as in the Agdell Experiment, this may be because "adequate" amounts of nutrients applied broadcast when making the seedbed are less effective than nutrients distributed through the whole soil. (Warren, Johnston and Penny.)

ANALYSES OF SOILS BURIED UNDER A ROMAN EARTHWORK

Samples were taken in 1959 from soil buried under a Roman earthwork on the Chalk at Winterslow, near Salisbury. The site was excavated by the Ministry of Works. The mound was porous and very calcareous (75% CaCO_3); the buried soil underneath was well drained and aerated. Contents of CaCO_3 , total K and exchangeable K were the same in the buried soil and in the surrounding field. Total N and C and organic P were only one-third as much in the buried as in the surface soil. If the C and N contents of the buried soil were about the same when the earthwork was built as they are now in the surface soil, approximately 60–70% of the C, N and organic P has been mineralised in 1,500 years. Much of the mineralised organic P has been retained by the buried soil as inorganic P soluble in 0.5M- NaHCO_3 . Similar changes may occur when well-aerated soils on the Chalk are cultivated. These soils contain much P, perhaps partly because inorganic P, derived from the decomposition of organic matter, is adsorbed on the surface of CaCO_3 . (Mattingly and Williams.)

MAGNESIUM

Magnesium in basic slags

As the nutritional disorder in ruminants called hypomagnesaemia may be related to the Mg contents of soils and crops, and because basic slag is widely used on grassland, the value of the Mg in a few slags was investigated in greenhouse experiments with perennial ryegrass. Four sandy loams having the following characters were used:

| pH | Exchangeable Mg * (m. eq./100 g.) |
|-----|--------------------------------------|
| 4.0 | 0.07 |
| 5.1 | 0.26 |
| 6.8 | 0.37 |
| 7.4 | 0.14 |

* In N-ammonium acetate.

Two Open-Hearth slags and one Bessemer-process slag were used, they contained:

| Process | No. | Total Ca (%) | Total Mg (%) | Mg soluble in 1% citric acid (%) |
|-------------|---------|--------------|--------------|----------------------------------|
| Open-Hearth | ... | 31 | 5.4 | 44 |
| | ... { 2 | 27 | 5.9 | 31 |
| Bessemer | ... 3 | 33 | 1.2 | 55 |

The experiments tested three levels of MgSO_4 as standard; there was also a test of three levels of CaCO_3 to value the liming effect of the basic slags.

Magnesium-deficiency symptoms on the grass were most severe with the very acid soil; they were unaffected by CaCO_3 dressings or by Bessemer slag, but the grass was normal where MgSO_4 or Open-Hearth slags were applied. Slight symptoms of Mg-deficiency on the alkaline soil appeared after the first cutting where no Mg had been given. All the sources of Mg had only small effects on yield of grass with all soils except the very acid one, with which all Mg treatments increased yield and slag 1 was superior to slag 2 in one experiment; on this soil Bessemer slag also gave much lower yields than the two Open-Hearth slags. The amounts of Mg in the crops were measured by A. Weir (Pedology Department). Magnesium uptakes were compared in pots with slags and in those where liming had given the same pH values. The following results are averages of three experiments with the very acid soil and one with each of the other soils. The second Open-Hearth slag was superior

Percentage "magnesium sulphate equivalents" for two basic slags (amounts of Mg (given as MgSO_4) that would have given the same Mg uptake as the basic slags, expressed as % of total Mg supplied by the slag)

| | | | | | Cut 1 | Cut 2 |
|--------|-----|-----|-----|-----|-------|-------|
| Slag 1 | ... | ... | ... | ... | 23 | 23 |
| Slag 2 | ... | ... | ... | ... | 40 | 54 |

in all experiments, and continued to release Mg as the experiment progressed; slag 1 did not release more Mg after the first cut with the acid soils, but uptake by grass on the alkaline soil continued. The uptake of Mg decreased with increasing Ca levels in the soils of pH 5.1 and 6.8 and for the lower level of Ca on the alkaline soil. Increasing the CaCO_3 level (up to 8 cwt./acre) of the very acid soil increased Mg uptake during several cuts; with more CaCO_3 , which raised soil pH to 4.9, Mg uptakes did not alter further. Part at least of the Mg in basic slags is clearly available to grass. Where dressings of 5–10 cwt./acre of Open-Hearth slags of the kinds tested are applied, 25–50 lb./acre of Mg will also be supplied; even if only a proportion of this Mg is available, as in these experiments, it may have a worthwhile effect on crops grown on Mg-deficient soils. No general advice can be given without much more work, because, even for slags of similar Mg contents, the value of the Mg to crops varies for unexplained reasons; Ca–Mg interactions, such as were demonstrated here, will also affect Mg uptake. (Heintze.)

Magnesium in soils

Little of general use is known of the Mg status of British soils, but reports of Mg-deficiency in crops on certain soil types are common. Information is needed on factors that govern the supply of Mg to crops and on the need for Mg fertilisers. Work has begun, in parallel with existing work on soil K, to determine the nature, importance and properties of fractions of soil Mg. (Salmon and Arnold.)

The effect of nitrogen fertilisers on magnesium contents of grass

In a field experiment at Woburn in 1959 ammonium sulphate and sodium nitrate were supplied at 1.0 cwt. N/acre and tested without Mg and with magnesium sulphate supplying 93 lb. Mg/acre. The grass had the following Mg contents:

| | Without Mg to grass | With Mg manuring |
|-------------------------------|-----------------------|------------------|
| | % of Mg in dry matter | |
| Without nitrogen | 0.101 | — |
| With ammonium sulphate | 0.146 | 0.209 |
| With sodium nitrate | 0.164 | 0.209 |

The amount and form of N manuring can obviously greatly affect the Mg content of grass, but much more work is required to establish general principles. (Nowakowski and Otter.)

MOLYBDENUM

The 1959 lucerne of the Ley-Arable Experiment on Highfield contained only 0.1 p.p.m. of Mo (*Rep. Rothamst. exp. Sta. for 1959, p. 56*). In 1960 strips of the 2-year-old lucerne in both the Highfield and the Fosters Field Experiments were sprayed three times with sodium molybdate at 4 oz./acre; the first spray was when the crop was half-grown before the first cut, and the second and third were immediately after the first and second cuts. Table 11 gives analyses of the cuts. (Mo was not determined in the first cut as there

TABLE 11
Amounts of Mo, N, P and K in dry matter of lucerne from the Rothamsted Ley-Arable Experiments

| | | Highfield Experiment | | | |
|------------|-------------|--------------------------|------|------|------|
| | | Mo | N | P | K |
| | | (p.p.m.) | % | % | % |
| First cut | { -Mo } ... | — | 3.14 | — | — |
| | { +Mo } ... | — | 3.38 | — | — |
| Second cut | { -Mo } ... | 0.01 | 3.07 | 0.35 | 2.28 |
| | { +Mo } ... | 0.40 | 3.07 | 0.36 | 2.26 |
| Third cut | { -Mo } ... | 0.03 | 2.80 | 0.31 | 1.72 |
| | { +Mo } ... | 0.29 | 2.87 | 0.30 | 1.88 |
| | | Fosters Field Experiment | | | |
| First cut | { -Mo } ... | — | 3.19 | — | — |
| | { +Mo } ... | — | 3.36 | — | — |
| Second cut | { -Mo } ... | 0.24 | 2.94 | 0.36 | 2.20 |
| | { +Mo } ... | 1.05 | 2.95 | 0.37 | 2.20 |
| Third cut | { -Mo } ... | 0.32 | 2.71 | 0.32 | 1.86 |
| | { +Mo } ... | 1.88 | 2.85 | 0.31 | 1.93 |

was risk of contamination by the spray, N determinations for this cut were on the uppermost growth; the lowest growth was excluded in samples from the second and third cuts.) The sprayed and unsprayed lucerne looked alike, and yields were not increased by the sprays. Mo contents of lucerne on unsprayed plots on Highfield were even lower (0.01 and 0.03 p.p.m. Mo) than in 1959. Applying

sodium molybdate increased Mo to 0.3–0.4 p.p.m.; this did not affect N, P and K contents of the lucerne, which were normal and similar to values for lucerne in the Fosters Field experiment, where the levels of Mo were much higher. The very low Mo content of untreated lucerne on Highfield had not affected N fixation. (Warren, Johnston and Foale.)

ANALYTICAL WORK

Natural radioactivity in soils

A scintillation counter for the assay of the natural β - and γ -radioactivity in soils was designed and made in collaboration with B. Edwards (Workshop). The counter consists of two parts: (a) The sample holder, which can receive 100–300 g. of dry soil, has an interchangeable face so that it can be used with different solid phosphors; (b) the photo-multiplier tube assembly incorporates a 5-inch diameter EMI 9530B photomultiplier tube with associated voltage distribution circuitry and cathode follower unit. The whole assembly is housed in a deep-freeze unit at 0°. (Talibudeen.)

Determining carbon in aqueous extracts of soil organic matter

A method for determining carbon in acid or alkaline extracts of soil organic matter without preliminary concentration was developed. The solution is adjusted to pH 4, boiled to drive off absorbed CO₂ and cooled; excess sodium persulphate is added and the solution boiled. The evolved gases are bubbled through saturated KI solution to remove halogens, over hot CuO to oxidise CO to CO₂, dried over MgClO₄, and CO₂ is determined gravimetrically by absorbing in soda-lime. The organic carbon in N-HCl : N-HF extracts of soil can be determined by this method, even when they contain only 50 mg. organic carbon/litre. (Jenkinson.)

Measuring low phosphate concentrations in the presence of much electrolyte

Colorimetric methods for measuring phosphate in the range 0.04–4.0 p.p.m. P are very insensitive when M-KCl solutions are used. A method of using Cl⁻-saturated anion exchange resin and standard ³²P-labelled phosphate solutions containing M-KCl was developed to overcome this difficulty. The solutions were shaken with resin for varying times and at different resin : solution ratios. The fractional activity in solution under the conditions tested was a linear function of: (a) log of phosphate concentration; (b) log of time of shaking; and (c) resin : solution ratio. The relationship between the fractional activity left in solution and the *initial* phosphate concentration was steeper with smaller reaction times. Resin : solution separation is instantaneous, so the method is simple and rapid, and its accuracy depends only on the accuracy of reproducing the weight of wet resin. The resin : solution ratio used at present is 800 mg. wet resin : 100 ml. M-KCl containing between 0.04 and 4.0 p.p.m. of P. By decreasing the resin : solution ratio and the time of shaking, phosphate concentrations <0.01 p.p.m. can be measured

in the presence of other electrolytes. The method could be used to determine isotopically exchangeable forms of phosphate in solution when they are mixed with soluble non-exchangeable forms that are hydrolysed by the acid treatment necessary in colorimetric measurements. The method could also be used to assay ions of other elements for which there are suitable radio-isotopes. (Vaidyanathan and Talibudeen.)