

Agricultural

Determination of Iron in Humates. N. A. Clark and D. H. Sieling (*Ind. Eng. Chem., Anal. Ed.*, 1936, **28**, 256–257.)—Humic extracts from soils and synthetic humates prepared from carbohydrates act as iron carriers and the iron adsorbed or combined, unlike inorganically combined iron, is not precipitated as hydroxide at *p*H values of 3 to 5, but remains in solution even at *p*H 8 or 9, and is thus available to plant life under alkaline conditions (Burk, Lineweaver and

Horner, *Soil Science*, 1932, **33**, 413). Determination of the iron-content of such extracts involves the preliminary oxidation of the organic matter and determination of iron colorimetrically by means of potassium thiocyanate. The thiocyanate colour is not stable and fresh standards are required at frequent intervals. The reagent suggested by Yoe (*J. Amer. Chem. Soc.*, 1932, **54**, 4139; Abst., ANALYST, 1933, **58**, 54) for the colorimetric determination of iron, viz. 7-iodo-8-hydroxy-quinoline-5-sulphonic acid $C_9H_4N(OH)I(SO_3H)$ is obtainable commercially, and Hahn and Whiffle (*Amer. J. Med. Sci.*, 1936, **191**, 24) have suggested for it the name "ferron." With ferric ions it gives a green colour; with ferrous ions it does not react. Yoe found that some seventy other ions gave no reaction. Cupric ions give a precipitate, and a few salts which undergo hydrolysis, such as tin and titanium compounds, must be removed if present in more than small amounts. Strong acids and bases destroy the colour. Yoe gives as the optimum condition a slight acidity to methyl orange paper, but the authors find that a more precise adjustment to a pH value of from 2.7 to 3.2 is necessary. Concentrations of iron of the order of 1 in ten million, give a greenish-yellow colour; higher concentrations give a green colour. The method can be combined with the preliminary oxidation of organic matter by means of hydrogen peroxide and sulphuric acid, and it is suggested that oxidation by a mixture of perchloric and nitric acids may also be used. The organic matter, containing about 0.1 mg. of iron, is placed in a flask marked at 70 ml. (a small Pyrex distillation flask from which the side tube has been sealed off is recommended) with 5 ml. of 10 per cent. sulphuric acid and a small bead to prevent bumping, and the mixture is heated until fumes are evolved. The flask is then cooled slightly, a few drops of 30 per cent. hydrogen peroxide are dropped directly into the liquid, and heating is continued until sulphur trioxide condenses within 5 cm. of the top of the neck. Oxidation is repeated, if necessary, until the liquid is clear. When cold, the solution is diluted to 70 ml. Ten ml. of the solution are taken and $N/10$ potassium hydroxide solution is added until a faint blue colour is produced with bromophenol blue indicator when the liquid is diluted to 40 ml. $N/10$ sulphuric acid is then added to bring the pH within the limits 2.7 and 3.2. The amount of standard acid added is deducted from the amount of potassium hydroxide, and the corresponding amount of alkali is added to a fresh 25-ml. portion of the original solution, which is then diluted to 100 ml. One ml. of 0.2 per cent. solution of the reagent is added, and the colour produced is compared with that of standard solutions of iron. The standard solution used was prepared by oxidising pure ferrous ammonium sulphate with bromine and boiling off the excess bromine, the solution being then diluted to an iron concentration of 2 mg. per litre. This standard solution deteriorates after 3 days if the pH is above 2. The permanence of the green colour produced with the standard solution is almost indefinite, no change being found in 25 days. By adding 0.1 to 1 mg. of silica in the form of sodium metasilicate to the standard solutions it was shown that silica has no interfering action. The advantages of the method are the stability of the colour produced, and the lack of interference by other ions, the latter being a very desirable characteristic of methods for the examination of soil extracts.

A. O. J.

Proportions of Certain Poisonous Substances in Feeding Stuffs, and their Effect on Livestock. J. F. Tocher. (*Vet. Record*, 1935, 15, 477-481.)—

Effect of Salt on Pigs.—Salt has no toxic effect when fed to pigs in quantities from 0.25 to 3 oz. daily, provided the quantity is increased gradually, but pigs will not consume the same high proportion of salt if the change is direct from a non-salt diet. The rate of growth was improved by about 12 per cent. on adding 0.25 and 0.5 oz. of salt with ground limestone and cod-liver oil to the control feed.

Effect of Salt on Poultry.—Poultry were able to tolerate salt to the extent of 2.5 per cent. of the food supplied, but higher proportions affected their health, and 7.0 per cent. and over caused death.

Linseed and Linseed Cake.—No sample of cake examined was ever found to contain more than 0.05 per cent. of hydrocyanic acid developed from the glucoside present. Experiments on pigs showed that 2.5 grains of pure hydrocyanic acid had no ill effect, but 6.16 grains caused death in half-an-hour. If, therefore, a pig were able to eat 4.4 lbs. of powdered linseed cake moistened with water, and if, as is unlikely, the acid were immediately developed in the stomach, the pig would die as the result of consuming this quantity of linseed cake. It is therefore concluded that it is unlikely a pig would die as the result of being liberally fed with linseed cake if the food contained 0.18 per cent. of glucoside found in any sample. Linseed treated with boiling water can contain no poison, for any hydrocyanic acid liberated from the glucoside is lost by volatilisation; hence potassium cyanide mixed in the ration and allowed to stand had no ill effect on the pig, as the liberated prussic acid was partly lost by volatilisation and partly converted into non-toxic substances. Administered in a capsule, however, potassium cyanide in quantity equivalent to 6 grains of prussic acid, proved rapidly fatal. In experiments on stirks a dose of potassium cyanide equivalent to 12 grains of prussic acid caused no ill effects, so that even 3½ lbs. of linseed cake containing the highest proportion of prussic acid yet found can have no bad effect.

Castor Seed.—Pigs are found to be much more tolerant to castor seed than has hitherto been thought, and 40 castor seeds in a ration did not cause death or even illness. Castor seed was safely fed to stirks up to 16 g. (247 grains) in 2 lbs. of maize (approx. 1.76 per cent.), but 24 g. caused scouring. From the work with stirks it is concluded that a certain immunity was established by frequently feeding with rations containing small quantities of castor seed. Castor shells were found to be free from ricin and caused no disturbance when fed. The proportion of seed corresponding to 0.005 per cent. of husks in a feeding stuff varies from 0.016 to 0.024 per cent. of seed, which is well within the limit of safety found in the experiments; in fact, 1 to 1.5 per cent. of seed gave rise to no symptoms in the case of the stirks 2 years old. Cattle may vary considerably in their resistive power to ricin. The conclusions reached by the author are reviewed in light of prosecutions under the Fertilisers and Feeding Stuffs Act. In his opinion an action against a seller, *on the ground of injury to cattle*, for supplying a feeding stuff containing 0.024 per cent. of castor seed would be unwise. Whether (b) of the Fifth Schedule would apply to such a proportion would depend upon the legal meaning of a poisonous substance. The Fifth Schedule of the Act provides for legal action in the case of actual occurrence, not of remote contingencies. D. G. H.

Determination of Phosphorus in Soils. W. McLean. (*J. Agric. Sci.*, 1936, **26**, 331–336.)—The sample is dried in air and passed through a 2-mm. sieve, and a weighed quantity (*e.g.* 1 to 10 g., or an amount equivalent to 0.15 to 0.25 g. of the final blue-black precipitate) is heated over a small flame in a 500-ml. Kjeldahl flask with 10 to 15 ml. of conc. sulphuric acid and 15 ml. of nitric acid. The flame is raised until white fumes appear, but if after 45 to 75 minutes the dark colour of the liquid and the presence of a residue indicate incomplete oxidation, the liquid must be cooled and more nitric acid added. This, however, is necessary only when the amount of organic matter present is high. The liquid is then cooled, a little water is added, and the mixture is filtered, the residue being washed until the volume of the filtrate amounts to 150 ml. The filtrate is neutralised with ammonia (*d.* 0.88), 20 ml. of conc. nitric acid and 30 ml. of 50 per cent. ammonium nitrate solution are added, and the mixture is heated to 70° to 75° C. and treated with 60 ml. of 3 per cent. ammonium molybdate solution. On the following day the precipitate is collected in a Gooch crucible which has been packed with asbestos and washed with 1 per cent. nitric acid and ignited and weighed, and the precipitate is ignited gently until it is blue-black in colour. It is then weighed, and the weight is multiplied by the factor 0.038 to obtain the amount of P_2O_5 . The method may be used for extracts of soils in hydrochloric acid, and it has been found that on boiling soil with this acid (b.p. 110° C.) for 48 hours under a reflux condenser, complete extraction is always attained; carbonate soils require a shorter period of extraction than acid soils. The results obtained in this way are in agreement with those found by the use of sulphuric and nitric acids as described above, but the latter method is preferable, being more rapid. In view of the insignificant proportion of phosphorus obtained on re-extracting the residues obtained after digestion with hydrochloric acid, it is suggested that the phosphorus extracted by digestion of the soil with sulphuric and nitric acids represents a definite category of soil phosphorus, and that this may be taken as the total phosphorus present in the soil; any phosphorus which resists either method of extraction is probably included within soil minerals, or is so insoluble as to play no part in the phosphorus cycle of the soil. Data are given for 22 soils of different kinds.

J. G.