The determination of total iron in soils by atomic absorption spectrophotometry, after digestion with strong oxidizing acids and treatment with hydrofluoric acid to eliminate silica, has been described \(^1\). Little attention appears to have been paid to the use of the method for determining iron in reagents designed either to extract particular iron species from soils or to measure the potential availability if soil iron to plants. A study was therefore made of the atomic absorption method for determining iron in three types of extracts, particularly with a view to seeing if satisfactory results could be obtained by aspiration of the extracts without preliminary treatment. In this way considerable time could be saved and the full potentialities of the flame in destroying organic matter (which is invariably present in soil extracts and which interferes with chemical methods of determining iron) would be realized. The results obtained by direct atomic absorption of the extracts were compared with those obtained using the colorimetric 2 : 2'-dipyridyl method after destruction of organic matter in the extracts.

**Soil extracting reagents**

* \( N \) ammonium acetate, pH 7.0. This is commonly used for determining exchangeable bases in soils \(^2\). With regard to iron this reagent extracts exchangeable, including water-soluble, \( \text{Fe}^{2+} \). 77 g of ammonium acetate is dissolved in about 960 ml of water, adjusted to pH 7.0 with acetic acid or ammonium hydroxide and diluted to 1 litre.

* \( N \) ammonium acetate, pH 3.0. This reagent \(^3\) extracts 'exchangeable \( \text{Fe}^{3+} \)' (species of the form \((\text{Fe(OH)}_n)_{(3-n)+}\)) in addition to watersoluble and exchangeable \( \text{Fe}^{2+} \). 77 g of ammonium acetate is dissolved in about 800 ml of water, concentrated hydrochloric acid (about 150 ml) is added to bring the pH to 3.0 and the solution is then diluted to 1 litre.

* Morgan's reagent (0.5N acetic acid – 0.75N sodium acetate, pH 4.8). This reagent is widely used \(^4\) for assessing the status of major and trace elements in soils. 100 g of sodium acetate trihydrate is dissolved in water, 30 ml of glacial acetic acid is added and the solution then diluted to 1 litre.
Soils and extraction method

Samples of two alluvial loams, pH 7.1 and 5.2, and two sandy soils, pH 7.0 and 4.9 were used after air-drying and grinding to pass a 2 mm sieve. Samples of air-dried soils were extracted as such and other samples were extracted after incubation (at 30°C for 3 weeks) in sealed bottles (to give anaerobic conditions) with sufficient water added to just cover the soils. In each case 25 g of soil was shaken (to-and-for shaker) for 15 minutes with 75 ml of extracting reagent and then filtered through No. 1-Whatman paper using dry apparatus. Duplicate extractions were made of each soil.

Atomic absorption determination of iron in the extracts

The instrument used was the Unicam SP90 Atomic Absorption Spectrophotometer with the iron hollow cathode lamp. The operating conditions for maximum sensitivity were found to be the same irrespective of the extractant used and were as follows: wavelength 279.5 nm, lamp current 12 mamp, slit width 0.1 mm, burner height 1 cm, air flow 5 l per minute, acetylene flow 1.2 l per minute. Standard curves for iron were prepared for each reagent both without and with scale expansion so as to give broad and narrow ranges of iron concentrations.

Colorimetric determination of iron in the extracts using 2 : 2'-dipyridyl

The method used was that described by Kumada and Asami with slight modification. A suitable aliquot of the extract was evaporated to dryness and organic matter destroyed by heating to dryness after addition of 30% hydrogen peroxide. The residue was dissolved in 2 ml of 6N hydrochloric acid and transferred to a graduated flask with the aid of 10 ml of 3N sodium acetate (to give a final pH of 5.1). 2 ml of 10% (w/v) hydroxylamine hydrochloride and 2 ml of 0.1% (w/v) 2 : 2'-dipyridyl were then added, the solution diluted to the mark and extinction measurements made after 15 minutes with the Hilger Spekker Absorptiometer.

Results and discussion

The standard curves for iron in the atomic absorption measurements were similar for all extracting reagents, but differed sufficiently to necessitate determining the unknowns from the appropriate standard curve. Where an extract had a relatively high iron concentration it was suitably diluted, using the appropriate reagent, before aspiration. This was done so that the determination would be made at maximum sensitivity (maximum scale expansion) in order to reduce the effects of possible interfering factors. The range covered at maximum sensitivity was 0.5 to 10 ppm of iron, with 5 ppm giving an absorbance near 0.25.

Table 1 presents the results obtained by atomic absorption by aspirating the filtrates without pretreatment (except dilution where necessary) compared with those obtained using the colorimetric method preceded by destruction of organic matter. There was good agreement between the two methods of determining iron in each type of extract from all the soils.