Methods for the determination of metals in organic compounds are generally described very briefly in the standard text-books of microchemical analysis. Since a considerable number of organic compounds containing a variety of metals has been synthesised, methods of analysis would seem to be of some importance and, indeed, a survey of the literature indicates that far more methods have been described than the text-book treatment would suggest.

At first sight it would seem that a simple mineralisation, followed by a standard finish, would be all that is necessary; but an examination of the methods which have been applied indicates that the procedure may not necessarily be as simple as this, because certain types of compounds behave anomalously. For example losses by volatilisation can occur during mineralisation, or products of decomposition may be formed which can interfere with the final determination.

Since the present authors were interested in the determination of a number of metals in organic compounds, a research of the literature was undertaken and the number of methods described was found to be far more extensive than had been anticipated. Accordingly, the work is now presented here as a comprehensive review of the subject.

Sodium.

Tabern and Shelberg\(^1\) found that the commonly accepted sodium sulphate (sulphated ash) method was unsatisfactory for many organic sodium salts especially barbiturates; these findings were supported by Collins\(^2\). In the case of barbiturates, they found that if the organic matter was first removed by acidification and extraction, the sulphated
ash determination was then satisfactory, but the method became tedious. Accordingly the salt was titrated electrometrically with 0.1 N hydrochloric acid, 0.1 ml of which produced a pH change of several units at the end point.

It was claimed later by Martin that complete conversion to the sulphate can be obtained in the sulphated ash method by heating the compound, contained in a platinum boat, in a stream of oxygen and sulphur dioxide.

The general method now appears to consist of ashing the compound by careful ignition, extracting with diluted acid, evaporating to dryness, and dissolving the residue in water. The sodium can then be precipitated as the magnesium, or zinc, uranyl acetate using the standard procedure of Kahane or Barber and Kolthoff respectively, the precipitate being weighed or titrated in the usual way.

Holmes and Kirk, in a drop scale method, have succeeded in reducing the uranyl salt with cadmium, followed by titration with ceric sulphate. This method gave results accurate to ±1% for 0.13-4.13 mg of sodium, and Hallett has suggested that the method may be applied to the analysis of organic compounds.

Potassium, especially in high concentrations, interfered in these methods and was generally removed as the sparingly soluble acid tartrate.

**Potassium.**

The determination of potassium by precipitation as potassium sodium cobaltinitrite has been widely studied, but the results are inaccurate and not reproducible, because the precipitate is of variable composition, unless very carefully controlled conditions are used.

The most reliable method, after preliminary mineralisation, appears to be precipitation and weighing as the chlorplatinate. One modification of this method was based on the following reactions, and was devised by Shohl and Bennett.

\[
\begin{align*}
K_2PtCl_6 + 6 KI & = K_2PtI_6 + 6 KCl \\
K_2PtI_6 + 2 Na_2S_2O_3 & = K_2PtI_4 + 2 NaI + Na_2S_4O_6
\end{align*}
\]

Bullock and Kirk preferred to reduce the precipitate with metallic magnesium in neutral solution; the chloride ion produced was then titrated with silver nitrate.

**Lithium.**

Very little work has been done on the determination of lithium in organic compounds. Gilman and Haubein, however, have used a modification of the method of Ziegler, Crössman, Kleiner and Schäfer, in