

# LOSS-ON-IGNITION AS AN ESTIMATE OF ORGANIC MATTER AND ORGANIC CARBON IN NON-CALCAREOUS SOILS

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## *Summary*

Examination of data on North Wales soils shows that a good correlation exists between loss-on-ignition and organic C values, determined by Tinsley's method. Ignition for half an hour at 850° C, and for 16 hours at 375 ± 5° C have both been employed. The latter has advantages over the former procedure. Regression lines and prediction limits for organic C from loss-on-ignition are given from the data obtained. Although these regressions are not necessarily expected to be generally applicable, examination of some published data suggests that closely similar expressions may be. The method, because of its simplicity, can be usefully applied in a wide range of survey, analytical, and ecological studies, in spite of the known sources of error.

## *Introduction*

IN the usual procedure for determining loss-on-ignition a weighed quantity of soil is dried overnight in an oven at 105° C, reweighed and then ignited in a muffle-furnace at 850° C for half an hour. The value of loss-on-ignition is then expressed as the percentage of weight lost after the 105° C moisture loss.

Organic matter content can be estimated from a determination of the organic carbon content of soil, or vice versa if an assumption is made that the proportion of elemental C to organic matter is constant. This assumption is not precise but may be employed with reasonable accuracy. Methods employing oxidation of C by dichromate are widely used for organic carbon determination. Tinsley's (1950) modification of the Walkley and Black (1934) digestion procedure was used in this work. Soil containing 10–25 mg C is boiled under a cold-finger condenser with 25 ml of a 0.4N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> mixture (400 ml conc. H<sub>2</sub>SO<sub>4</sub> and 200 ml H<sub>3</sub>PO<sub>4</sub> per l) for 1 hour. The excess dichromate is then titrated with 0.4N Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O. This method gives a high recovery of organic carbon and avoids the use of the arbitrary recovery factor necessary with the original Walkley and Black procedure, although the gravimetric procedure of Shaw (1959) gives results which correlate better with the more laborious dry combustion techniques.

Bremner and Jenkinson (1960a) have pointed out that, in dichromate oxidation estimates of soil organic C, it is assumed that two atoms of oxygen supplied by dichromate are required to oxidize one atom of C. This is true for cellulose but is not so for many other carbon compounds present in soil organic matter. Comparison of Tinsley's method with the wet combustion method of Shaw, in which liberated CO<sub>2</sub> is determined gravimetrically, showed that for lignin the Tinsley method gave high results, and, for humic acid, pectin, and tannic acid, low results.

These authors found recoveries by the Tinsley method from 87 to 106 per cent organic carbon for a range of fifteen soils of varied texture and origin, giving a mean recovery of 96.3 per cent, almost identical to that originally quoted by Tinsley. These results were substantially less variable than those obtained with the Walkley and Black technique with or without the use of the recovery factor in calculation. The failure to obtain a general 100 per cent recovery was found to be due partly to the error in the initial assumption about oxidation conditions and partly to incomplete recovery of organic carbon.

### *Errors in organic matter estimates from loss-on-ignition*

Loss-on-ignition has been widely dismissed as crude and inadequate as an estimate of organic matter or organic C content of soils (see, for example, Duchaufour, 1956, p. 276; Jackson, 1958, p. 207; Metson, 1956, p. 170; Piper, 1942, p. 213; Robinson, 1949, pp. 28-29 and 253) on the grounds that, in addition to the main weight losses from the destruction of organic matter, further weight losses occur due to loss of CO<sub>2</sub> from carbonates in calcareous soils, to loss of elemental C, and to loss of structural water from clay minerals. The main error in calcareous soils can be corrected, if the amount of carbonates present is known, but attention is confined here to non-calcareous soils. The main losses of CO<sub>2</sub> from carbonates, especially calcite, occur at high temperatures, and thus this error is likely to be eliminated or substantially reduced in the low-temperature ignition procedure, although this point has not been checked for natural calcareous soils. Carbonized materials, inert or elemental C, may be present in soils as added coal, clinker, and soot or as wood charcoal after burning. Such materials are believed to contribute little to the cycle of humus C (Bremner and Jenkinson, 1960*b*). These authors obtained high recovery rates of elemental C by all the wet combustion methods tested, including those of Shaw and Tinsley, and therefore these methods do not distinguish between humus and elemental C. Elemental C is also destroyed in ignition procedures, both at 850° and 375° C, and an estimate of active soil C or organic matter is only obtained, both by ignition and by wet oxidation, for soils free of added elemental carbon in any substantial amount.

The contribution to loss-on-ignition values resulting from loss of structural water from clay minerals varies according to the percentage of clay in the soil and according to the mineralogical nature of the clay. Weight losses at 850° C are for mica-clays 5-9 per cent, for kaolinite 13-20 per cent, and for montmorillonite 14 per cent (Grim, 1953). Detailed data are given by Leenheer *et al.* (1957) for percentage loss-on-ignition at 850° C in fifty non-calcareous soils of varying texture, having little or no organic matter present. These authors obtained mean loss-on-ignition values on these soils which varied from 1.3 per cent in soils of 5-10 per cent clay, through 3.6 per cent in soils of 25-30 per cent clay to 5.2 per cent in soils of 55-60 per cent clay. The potential inaccuracy arising from variation in clay content, between a light soil containing 5 per cent clay and a heavy one containing 50 per cent clay, is thus

generally of the order of 4–6 per cent loss-on-ignition. Many projects deal with soils with a much smaller range of clay content than this. Using ignition at 850° C it is possible to reduce the potential error, where a wide textural range is involved, either by applying a correction factor to high clay soils or by deriving separate correlations for organic carbon against loss-on-ignition in soils grouped texturally according to the material being studied. Differences due to variation in clay mineralogy are unlikely to be highly significant for most purposes, especially in relation to normal sampling errors.

An alternative procedure to the usual 850° C ignition is to carry out ignition at a carefully controlled lower temperature. The greatest part of the weight loss due to water loss from clay minerals which have been dried at 105° C occurs in the range from 450° to 600° C. Ignition at a lower temperature than this, which should therefore avoid such losses, has been investigated by Keeling (1962) in determining the ignition loss of the clay mineral in clay samples. He demonstrated by experiments on a number of clays that heating at 375° C for 16 hours removed more than 90 per cent of the carbonaceous material without loss of structural water. Ignitions at temperatures below 375° removed appreciably less of the carbonaceous matter and were therefore not suitable. Because of the variation in amount of organic matter destroyed in relation to temperature and time of ignition over this low temperature range it is necessary to have a closely controlled furnace temperature. In tests of the use of low-temperature ignition for soils, a furnace temperature of 375° C was maintained to  $\pm 5^\circ$  C by a pyrometer-controller.

#### *Materials Studied*

The soils used in this work were from Snowdonia, North Wales. Soil textures were mainly loams, but included a range from sandy loam to silty clay loam. The main groups of soils employed were:

Sixty-seven upland soils (organic carbon range 1–40 per cent), covering three different parent materials (rhyolite, dolerite, and pumice-tuff), established the correlation of loss-on-ignition at 850° C against organic carbon. A further fifty upland soils were used as a check on this correlation.

Fifteen soils of those used to establish the 850° C correlation, plus the fifty soils used as a check, established the correlation of loss-on-ignition at 375° C against organic carbon.

Since these samples were hill soils of humus forms varying from moder to mor, a group of twenty-two lowland soils from the Conway Valley formed on different parent material (dominantly Ordovician shale) and with humus forms ranging from mull to moder was analysed and used as a check on the correlations at both ignition temperatures.

An additional eleven soils of high organic matter were analysed since there was a limited number of samples in this range.

The results presented were obtained using routine procedures of single sample analysis. Because of overlap, not all the points used in determining the regressions are able to be shown separately in Figs. 1 and 2.

### Results

*Ignition at 850° C.* Initial investigation dealt with the relationship between 850° C ignition loss and organic C (Tinsley's method). Regression equations calculated independently for soils of the three parent material groups did not differ significantly and, therefore, the results for all samples were combined to give an unweighted regression of  $y = 0.462x - 1.36$  ( $y$  = organic C,  $x$  = loss-on-ignition, number of samples = 67, correlation coefficient = 0.99, organic C range 1.1–38.9 per cent, loss-on-ignition range 5.8–88.5 per cent).

Confidence limits calculated from this unweighted model imply that the error in using loss-on-ignition to calculate organic C is of the same order for all values of organic C, that is to say that the error is proportionally very much greater at low values, which is contrary to the observed results. Prediction limits and a new regression equation were therefore obtained from these data using a weighted regression procedure (Williams, 1959). In this it is assumed that the deviation of any observation from the regression line does not have a constant variance for all values of  $y$ , but is proportional to  $y(1-y)$ ,  $y$  being expressed as a proportion, rather than a percentage. This model corresponds better to the variability shown by the scatter diagram than does an unweighted model. Although the percentage error is greater for lower values of  $x$  and  $y$ , this is not exaggerated as it is in the unweighted model. The regression obtained from the 850° C ignition data by this approach on these soils is:

$$y = 0.476x - 1.87. \quad (1)$$

The 95 per cent prediction limits in terms of percentage are obtained from the relationship,  $y \pm 200s$ , where

$$s^2 = (133 \times 10^{-6}) \frac{Y}{100} \left(1 - \frac{Y}{100}\right) + 9 \times 10^{-7} + (474 \times 10^{-7}) \left(\frac{x}{100} - 0.15\right)^2.$$

These limits and the regression are shown in Fig. 1, on which are also plotted results for the soils used as checks on the correlation.

*Ignition at 375° C.* Loss-on-ignition was determined at  $375 \pm 5^\circ$  C over 16 hour periods (65 soils) to determine the correlation with organic C. The weighted regression line and prediction limits from data on this second group of soils are as follows:

$$\text{Regression:} \quad y = 0.458x - 0.4 \quad (2)$$

$$\text{Prediction limits:} \quad y \pm 200s,$$

where

$$s^2 = (804 \times 10^{-6}) \frac{Y}{100} \left(1 - \frac{Y}{100}\right) + 5 \times 10^{-7} + (573 \times 10^{-7}) \left(\frac{x}{100} - 0.112\right)^2.$$

Fig. 2 shows this regression and the calculated prediction limits and also includes results for the samples used as a check on the correlation.

Comparison of Figs. 1 and 2 shows that the regression line derived from the 375° C ignition passes more nearly through the origin. This confirms that losses in weight due to non-organic material have been

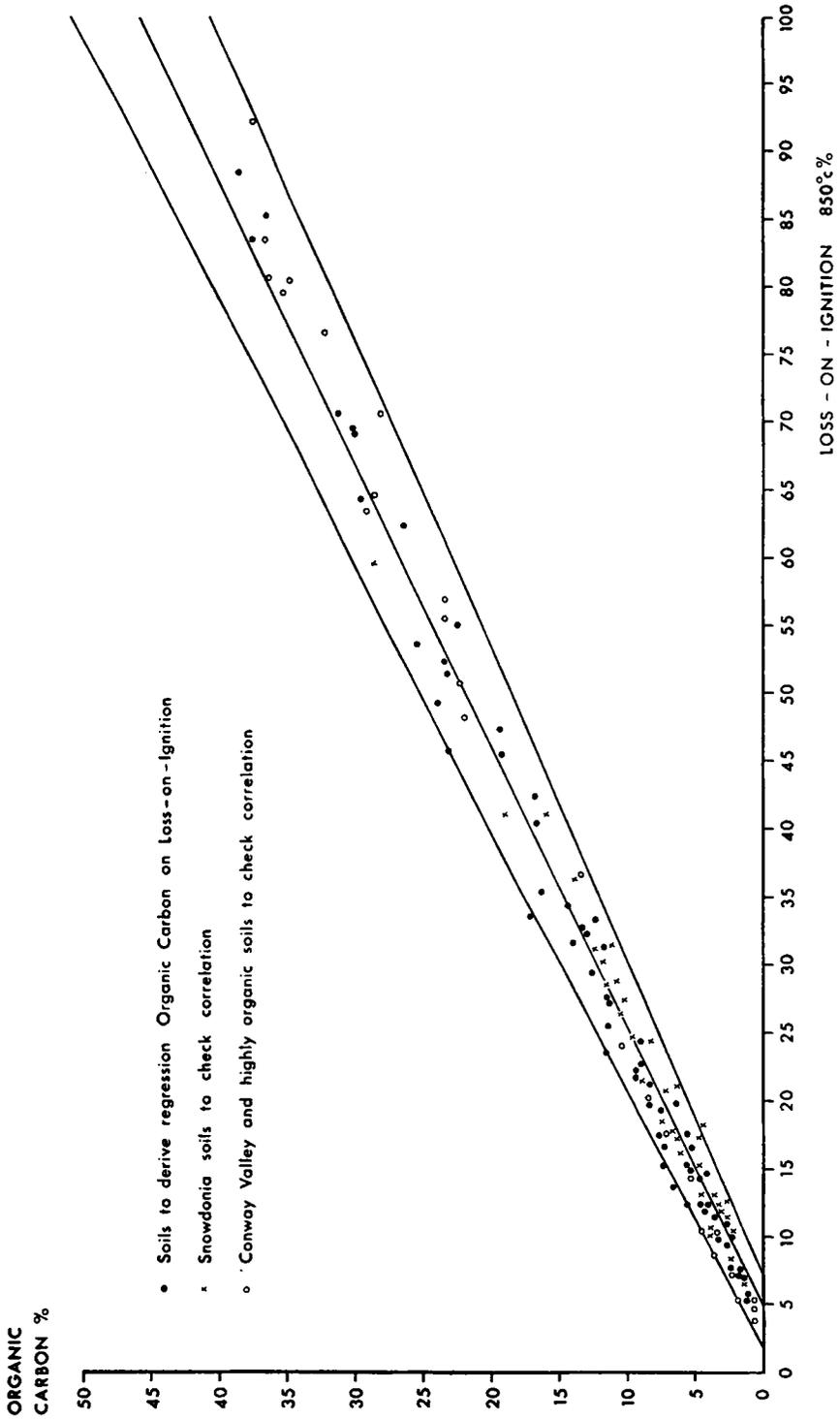


FIG. 1. Loss-on-ignition (850°C) against organic carbon (Tinsley).

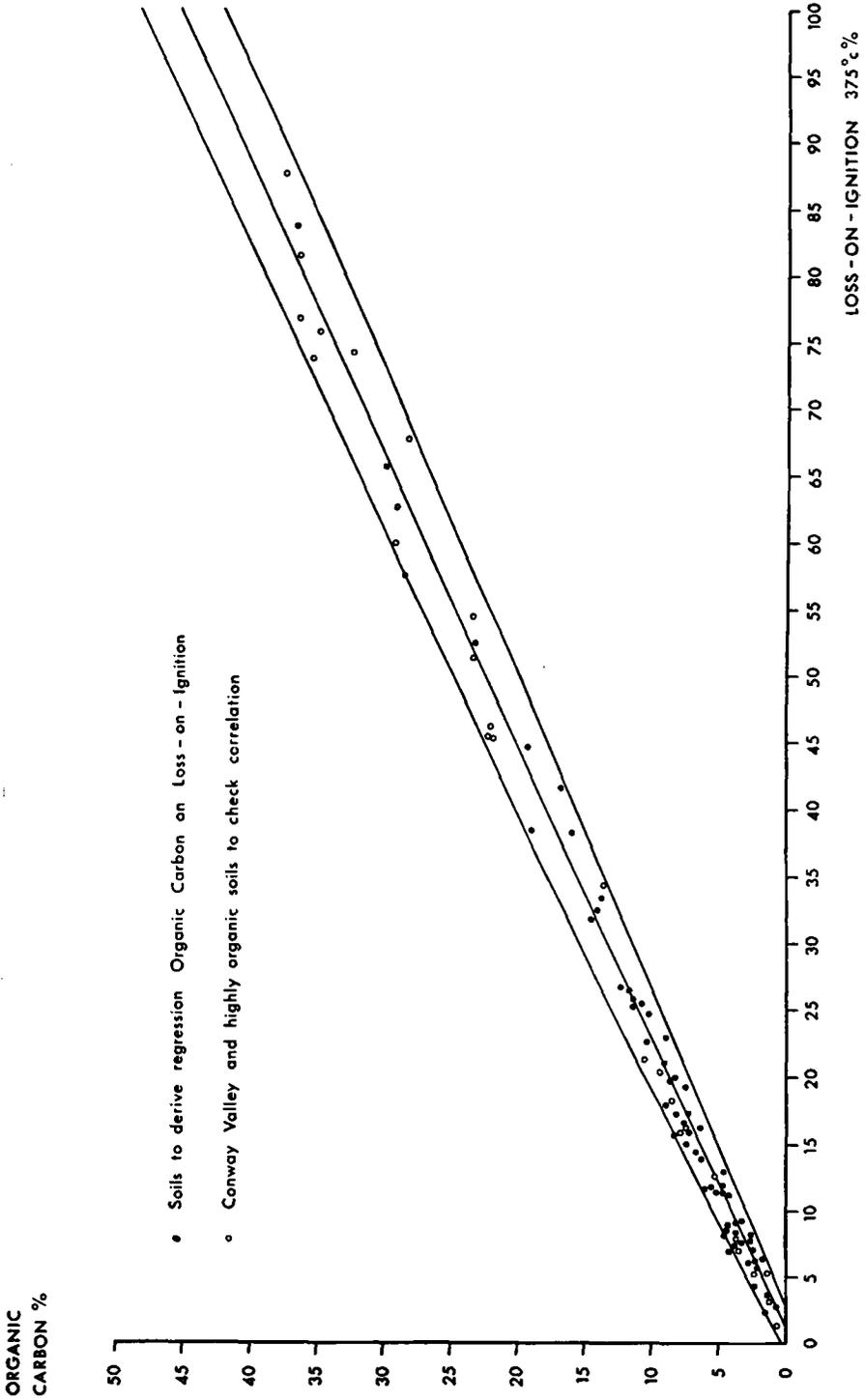


FIG. 2. Loss-on-ignition (375°C) against organic carbon (Tinsley).

reduced. Prediction limits are also narrower for the 375° C ignition at all values of organic matter. The soils used as checks on the correlation fit the regression well in each case. The relationships obtained may be of wider applicability and some data from the literature have been used to test this for the 850° C ignition. An independent test of the 375° C ignition loss/organic C correlation is not possible. Fig. 2 indicates that ignition at this temperature is preferable.

(A) Leenheer *et al.* (1957) studied three groups of soils: (1) alluvial or loessic > 25 per cent clay (7 soils), (2) alluvial or loessic < 25 per cent clay (22 soils), (3) formed on Palaeozoic rocks (two soils only). When the mean values on these three groups for organic C and loss-on-ignition are compared with organic C values obtained by applying equation (1) to their loss-on-ignition data, the results given in Table 1 are:

TABLE I  
(Data from Table 3 of Leenheer *et al.* 1957)

	<i>Loss-on-ignition</i> (mean for group, %)	<i>Organic C</i> (mean for group, %)	<i>Organic C</i> (calculated from weighted equation (1))
(1)	17.0	7.04	6.1 ± 1.6
(2)	13.6	7.18	4.6 ± 1.4
(3)	15.6	5.98	5.6 ± 1.5

Correlation of the results from application of equation (1) is good with the two sedentary soils of group 3, moderate with group 1, and poor with group 2. This is to be expected, because sandy alluvium will have little structural water loss from clay, although allowance for such loss is included in the equation obtained on the Welsh soils. If equation (2), corresponding to an analysis in which the effect of clay is insignificant, is applied to samples 2, the calculated carbon value is  $5.8 \pm 1.3$ .

(B) Park *et al.* (1962) give mean values for loss-on-ignition and carbon in a range of soils, of 20.4 and 7.8 per cent respectively. Using equation (1) to estimate organic C from their loss-on-ignition, a value of  $7.7 \pm 1.8$  per cent organic C is obtained.

(C) For soils discussed in the North Caernarvonshire soil survey memoir (Ball, 1963) the data in Table 2 are available, the loss-on-ignition values being determined at Rothamsted.

A closer assessment of the true organic C is obtained by application of equation (1) to values of loss-on-ignition at 850° C than is obtained by the use of the conventional recovery factor with Walkley and Black data.

(D) A further demonstration of this is given in data for some soils on ultra-basic rocks from the island of Rhum, using loss-on-ignition data determined at the Macaulay Institute (Table 3).

(E) The estimates of organic C, on the twenty-two Conway Valley soils plotted on Figs. 1 and 2, derived by Tinsley oxidation; by Walkley and Black oxidation using the arbitrary recovery factor; and by calculation from loss-on-ignition using the regressions (1) and (2) have been

compared. In the 850° C ignition, one-third of the calculated values were closer to the Tinsley estimate than were the Walkley and Black figures, two-thirds were less close. For the 375° C ignition, one-half the calculated values were closer to the Tinsley estimate than were the Walkley and Black figures. Employing ignition at 375° C, eighteen of the calculated values were closer to the Tinsley figures than were the values obtained from 850° C ignition loss; only four were less close.

TABLE 2  
*Data from North Caernarvonshire Soil Memoir*

Soil	Loss-on-ignition %	Organic C (Walkley and Black) %	Organic C (Tinsley) %	Organic C (calculated from equation (1)) %
Cn63/2	8.6	2.8	2.1	2.2 ± 1.2
/3	6.2	1.5	1.1	1.1 ± 1.0
CN64/1	30.7	16.8	11.2	12.7 ± 2.5
/2	9.1	6.3	3.6	2.5 ± 1.25
/3	7.7	2.8	1.7	1.8 ± 1.2
Cn68/1	27.2	28.6	15.6	11.1 ± 2.3
CN70/1	49.2	33.6	21.4	21.6 ± 3.0
/2	16.1	9.6	5.9	5.8 ± 1.7

TABLE 3  
*Data from Rhum, Inverness-shire*

Loss-on-ignition %	Organic C (Walkley and Black) %	Organic C (Tinsley) %	Organic C (calculated from equation (1)) %
21.0	10.6	7.7	8.1 ± 2.1
12.9	5.9	5.1	4.3 ± 1.3
6.0	2.7	1.8	1.0 ± 1.0
21.9	11.6	7.2	8.6 ± 2.2
15.9	10.4	4.9	5.7 ± 1.7
12.7	8.0	4.8	4.2 ± 1.3

### Conclusion

Loss-on-ignition is a simple and rapid method of assessing organic matter in soils and justifies more consideration than most standard reference works recommend. Investigation of the relation between loss-on-ignition (determined at the conventional 850° C and also at 375 ± 5° C) and organic C determined by Tinsley's modified Walkley and Black procedure shows that correlation is good.

From the results presented, and from application of the regressions obtained to some published and test data, ignition at both temperatures gives estimates of organic matter and organic carbon of an accuracy acceptable for many purposes. This accuracy is greater for the 375° C ignition, which eliminates variations due to weight loss of structural water from clay minerals. Although this temperature of ignition must

be maintained for a much longer time than the 850° C ignition, the improved accuracy makes its use preferable where a furnace with accurate temperature control is available.

It is not asserted that the specific equations given above are applicable to all types of soil and all forms of soil organic matter. Published data have shown substantial variations in organic C/organic matter ratio. It is probable that these or similar equations can be of wide applicability where a high degree of accuracy is not required. Many pedological, archaeological, and ecological studies fall into this category.

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