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Determination of Organic Matter for the Classification of Soil Samples

Détermination de la teneur en substance organique pour la classification des échantillons de sols

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SUMMARY

The organic matter in 53 samples, which according to U.S.C.S. classification should contain organic material, was determined by the wet combustion methods of Schollenberger, and Walkley and Black. It was desired to show whether an estimate of the amount of organic substance in soil could be obtained from the sodium hydroxide test and whether a relationship existed between the sodium hydroxide test and the displacement of the Atterberg limits after oven drying. Neither could be shown. The rapid method of Walkley and Black for the determination of organic matter is recommended in place of the sodium hydroxide test for the classification.

SOMMAIRE

La teneur en substance organique a été déterminée sur 53 échantillons de sols classifiés selon l'U.S.C.S. Les échantillons contenant des substances organiques etaient analysés d'une part par la méthode de combustion humide selon Schollenberger, d'autre part selon Walkley et Black. Il s'agissait de contrôler si l'essai avec la soude caustique fournissait des données quantitatives sur la teneur en matières organiques et de vérifier s'il existait une corrélation fondée entre les résultats de l'essai à la soude caustique et la modification des limites de consistance selon Atterberg. Les essais ont montré qu'il n'existe pas de corrélation quantitative. Les auteurs proposent de déterminer la teneur en substance organique pour classifier les sols à l'aide de la méthode rapide de Walkley et Black au lieu de l'essai à la soude caustique.

THE PARTICLE SIZE DISTRIBUTION, plastic properties, and organic impurities (in so far as they affect the plasticity) are decisive for the classification of soil samples according to U.S.C.S. (SNV, 1959). The organic matter is determined by the displacement of the Atterberg limits, i.e. by determinations on fresh and oven-dried (105 C) samples, and by the sodium hydroxide test (British Standard 812, 1951). We have had doubts about the usefulness of these methods. The organic matter content of 53 samples from various districts of Switzerland, which had been shown by routine classification to contain organic material, was determined, for comparison, by wet combustion followed by titrimetric determination of the unused oxidizing agent (Schollenberger, 1927; Jackson, 1958; Walkley and Black, 1934). We tried to establish whether an estimate of the amount of organic matter in the soil could be obtained from the sodium hydroxide test and whether a relationship exists between the values obtained from the sodium hydroxide test and the displacement of the Atterberg limits. We tested various methods for the determination of organic matter with regard to their suitability as rapid methods for the classification of soil samples.

METHODS

1. Classification by U.S.C.S. (SNV standard sheets 70.005 and 70.008, 1959).
2. Particle size distribution with hydrometer by the conventional method of Casagrande (Lambe, 1951). Dispersion with 0.75 per cent sodium hexametaphosphate.
3. Atterberg limits (Lambe, 1951) by SNV standard sheet 70.345 (1960) determined on fresh and oven-dried (overnight, 105 C) material.
4. Sodium hydroxide test by British Standard 812 (1951).
5. The organic matter was determined by wet combustion on air-dried material, with particle size smaller than 0.2 mm. A series of samples were crushed in a hammer mill. Depending on the colour, up to 2 grams of the sample were weighed out for the determinations.

In the Schollenberger (1927) and Jackson (1958) determination, the sample was treated for 20 minutes at 160 C with 10 ml 8 per cent $K_2Cr_2O_7$ and 15 ml conc. H_2SO_4. The unused dichromate was titrimetrically determined with 0.2 N Mohr'sches salt solution. According to Jackson (1958), 90 per cent of the organic matter is detected by this method. Daily capacity amounts to 12–15 determinations.

The determination of organic matter according to Walkley and Black (1934) and Piper (1944) is a rapid method, similar in principle to the Schollenberger determination, heating to 160 C being omitted. We back-titrated with 0.2 N Mohr'sches salt solution with a normal burette, i.e., no longer under hydrogen. The factor of the Mohr'sches salt solution showed little alteration for weeks if the solution was protected from light and kept closed. After standing for ½ hr (time for oxidation) we brought the volume to 250 ml with distilled water after the addition of conc. H_3PO_4. After a further ½ hour, i.e., when the suspended matter had settled, we pipetted 50 ml of the supernatant solution, added diphenylamine-sulphuric acid and titrated with Mohr'sches salt solution. Thus we obtained a clear titration end-point and the titration was reproducible. About 76 per cent of the organic matter is determined by the Walkley and Black rapid method. Daily capacity is 20–25 determinations.

RESULTS

The results are given in Table I.

DISCUSSION

If the colour of the supernatant sodium hydroxide solution in the sodium hydroxide test was a measure of the

TABLE I. CLASSIFICATION OF 53 SOIL SAMPLES BY U.S.C.S. AND COMPARATIVE DETERMINATIONS OF THE ORGANIC MATTER BY THE SODIUM HYDROXIDE TEST AND BY WET COMBUSTION METHODS

			Atterberg limits water content in per cent				Organic matter		
			liquid limit		plastic limit			Wet combustion tests	
Sample no.	U.S.C.S. classification	Particle size distribution (per cent passing 0.002 mm)	air-dried	oven-dried	air-dried	oven-dried	NaOH test 0–5	Schollenberger* (per cent organic content)	Walkley-Black* (per cent organic content)
1	CL	15.1	30.0	24.0	15.2	13.5	0	0.3	0.1
2	CL	5.4	27.2	25.2	16.5	16.2	0	0.3	0.1
3	OH	6.0	55.5	51.5	35.2	34.0	5	3.9	3.3
4	OH	5.0	72.2	51.8	46.4	32.5	5	3.5	2.7
5	OL	12.2	46.1	38.3	30.4	27.0	4	2.5	2.0
6	OL	16.0	44.0	46.3	26.3	28.9	5	2.4	1.8
7	OH	4.8	60.0	48.8	32.1	28.4	5	2.8	2.6
8	OH	20.0	55.3	53.0	31.0	30.2	5	2.7	2.2
9	CL	9.3	38.0	34.8	18.7	16.2	3	0.8	0.6
10	CL	7.0	27.0	20.2	14.3	14.6	2	0.1	0.1
11	SC–CL	4.6	22.7	22.1	14.6	13.7	0	0.3	0.2
12	peat						5	40.2	32.0
13	OL	4.3	39.6	39.2	27.1	30.6	5	2.6	2.6
14	OL	6.3	35.2	31.4	23.7	21.9	5	0.8	0.9
15	OL	27.6	46.2	42.3	15.4	18.3	3	0.3	0.2
16	OL	5.8	46.1	42.8	28.5	27.1	4	3.1	2.6
17	CL	9.3	25.3	25.3	17.1	16.8	0	0.8	0.5
18	OL	7.4	38.3	35.9	24.8	23.4	4	2.8	2.3
19	CL	14.0	28.5	28.5	17.8	17.0	1	0.8	0.6
20	CL	8.5	26.3	25.7	17.4	17.5	1	0.9	0.7
21	CL	23.8	33.1	33.5	16.7	14.8	1	0.2	0.1
22	CH		44.7	30.0	16.1	13.5	1	0.0	0.1
23	OL	11.7	38.0	23.4	14.7	14.8	3	0.3	0.2
24	OL	19.8	43.7	36.0	17.4	16.3	4	0.7	0.3
25	OH	7.6	178.0	92.6	58.8	65.5	4	6.3	5.3
26	OL	4.1	35.3	32.3	18.2	18.0	2	0.3	0.1
27	OL	5.0	30.1	21.1	17.7	15.0	5	0.9	0.6
28	OH	4.8	69.4	47.0	39.1	34.6	5	2.0	1.4
29	OL	8.4	41.5	36.0	30.0	24.3	3	1.3	0.9
30	OL	11.5	45.6	37.3	23.9	22.4	5	1.4	0.8
31	OH	17.0	55.6	52.7	14.5	17.3	1	0.5	0.3
32	OL	20.0	36.0	31.8	15.0	15.1	4	0.8	0.5
33	OL	7.6	36.4	27.6	21.4	16.2	5	0.8	0.6
34	OL	10.0	38.6	30.5	22.5	17.0	5	1.2	0.8
35	OL	5.2	29.0	23.4	16.1	14.2	5	0.7	0.5
36	OH	24.1	51.5	49.0	17.7	18.4	4	1.1	0.9
37	OH	21.6	66.5	51.5	25.4	23.1	5	2.0	1.5
38	OH	33.2	58.5	48.1	20.9	20.7	4	0.9	0.6
39	OH	24.2	60.0	54.1	15.3	16.7	3	0.4	0.3
40	OL	9.5	29.4	25.5	17.6	18.0	5	0.5	0.3
41	OH	36.2	54.6	50.8	19.8	20.0	3	0.6	0.3
42	OL	14.9	44.0	37.6	16.4	15.7	2	0.4	0.2
43	OH	5.0	53.0	35.6	25.6	21.2	5	1.4	0.9
44	OH	12.3	54.8	42.8	19.3	17.3	5	1.2	0.7
45	OH	8.6	50.8	36.3	22.3	21.1	5	1.2	0.9
46	OL	15.5	38.4	32.0	15.8	16.2	3	0.7	0.5
47	OL	17.6	46.1	37.8	18.6	17.6	4	1.5	0.6
48	OH	2.2	52.5	33.0	25.0	21.1	4	1.7	0.9
49	OL	17.1	45.5	37.5	22.4	19.6	3	0.6	0.4
50	OL	9.0	34.7	26.6	20.4	17.1	3	0.2	0.2
51	OL	8.1	43.0	32.0	25.7	22.1	1	0.2	0.1
52	CL	24.5	44.6	43.3	21.0	22.0	0	0.5	0.2
53	OL	6.0	43.0	34.1	22.0	19.9	5	1.1	1.0

*Average from at least two determinations.

amount of organic matter in the samples, the scale values 0–5 should correspond to increasing content of organic carbon. This is not the case, however, for more than half of the samples. The values obtained by the sodium hydroxide test are generally too high (e.g., samples 14, 24, 27, 32–35, 38, 40, 43–45, 53). For samples 1, 2, 11, 17, and 52, the sodium hydroxide test indicates no organic material although this is clearly shown to be present by the chemical determinations. The colour of the supernatant sodium hydroxide solution therefore gives no useful indication of the organic matter present in soil. For this to be the case, the different types of organic material in soil would have to be equally extracted by the sodium hydroxide solution. This goes against experience in soil science with the sodium hydroxide extraction of organic mater.

No definite relationship between the values obtained from the sodium hydroxide test and the displacement of the Atterberg limits could be established. For samples 1, 10, 22, and 51 a decrease in the liquid limit of more than 20 per cent is observed after oven drying, although the scale values obtained from the sodium hydroxide tests are 2 and smaller. For samples 3, 6, 8, 13, 16, 18, and 36 the displacement is less than 10 per cent while the sodium hydroxide tests show high organic contents (scale values 4 and 5). It is stated in the classification (SNV standard sheet 70.008, 1959) that noticeable reductions of the liquid and plastic limits on oven drying are obtained only with samples which contain finely distributed organic impurities. For other soils the Atterberg limits are not influenced by oven drying. No definite indication of the presence of such finely distributed organic impurities can be drawn from the sodium hydroxide test. The statements made in the classification mentioned above are speculative. Other soil components and properties besides organic impurities are known to be responsible for the displacement of the Atterberg limits after oven drying. To demonstrate the dependence of the displacement of the Atterberg limits on the presence of finely distributed organic impurities a method is required with which, within the limits of the classification, the inert (e.g., charcoal-like material, slightly decomposed plant remains) and the active (in respect to the plasticity) carbon can be determined. To our knowledge, no such method as yet exists.

A method, in which the carbon from carbonates is not determined seems most suitable as a rapid method for the determination of organic matter for soil classification since complete destruction of the carbonates, particularly for samples containing dolomites, is time consuming if the organic matter is not to be damaged. We therefore tested wet combustion methods with, respectively, colorimetric determination (Springer and Klee, 1955) of the Cr^{3+} formed by reduction of the dichromate, and titrimetric determination (Walkley and Black, 1934) of the unused dichromate. The rapid method of Springer and Klee (1955) gave no reliable results and the expenditure of time and apparatus (centrifuge, colorimeter) is at least as great as, if not greater than, that when using the rapid method of Walkley and Black (1934). Substances which can be oxidized or reduced such as pyrites, Fe^{2+} oxides, chlorides, and manganese oxides interfere in the Walkley and Black (1934) method (Walkley, 1947; Jackson, 1958). Only about 10 per cent of the elemental carbon such as charcoal and graphite is detected (Walkley, 1947). The considerable differences in the values obtained by the Schollenberger and the Walkley and Black methods are probably due to the presence of such inert carbon. For classification the limited detection of such inert carbon is not a disadvantage. The Walkley and Black rapid method can be carried out by chemically untrained personnel in almost the same length of time as the sodium hydroxide test, gives reliable results, and requires no special apparatus except a chemical balance.

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