

The Chemical Composition of Indiana Soils and Methods for Soil Analysis.

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The data presented herein concerning the composition of Indiana soils was desired by the department for the study of soil origin. It includes the results of the chemical analysis of twenty different samples of surface and subsoils collected by the departmental assistants, Messrs. Shannon, Ward and Ellis.

Description of Soil Samples.

Laboratory Number.		Collector.
1. No. 94,	first foot, Oolitic, light soil.....	C. W. Shannon
2. No. 94,	second foot, Oolitic, light soil.....	C. W. Shannon
3. No. 94,	third foot, Oolitic, light soil.....	C. W. Shannon
4. No. 95,	first foot, Harrodsburg, light soil....	C. W. Shannon
5. No. 95,	second foot, Harrodsburg, light soil..	C. W. Shannon
6. No. 95,	third foot, Harrodsburg, light soil....	C. W. Shannon
7. No. 208,	surface, Huron, light soil.....	C. W. Shannon
8. No. 208,	subsoil, Huron, light soil.....	C. W. Shannon
9. No. 1,	volusia silt loam.....	L. C. Ward
10. No. 2,	Miami silt loam.....	L. C. Ward
11. No. 3,	Waverly silt loam.....	L. C. Ward
12. No. 4,	Waverly silt loam.....	L. C. Ward
13. Nos. 2-5,	Upland limestone soil.....	L. C. Ward
14. No. 6,	Upland limestone soil.....	L. C. Ward
15. No. 7,	limestone and shale soil.....	L. C. Ward
16. No. 26,	second foot, silt loam.....	R. W. Ellis
17. No. 26,	first foot, silt loam.....	R. W. Ellis
18. No. 10,	first foot, limestone	R. W. Ellis
19. No. 16,	Ohio valley	R. W. Ellis
20. No. 10,	second and third foot, limestone.....	R. W. Ellis

The chemical examination to which the samples were subjected includes the determination of the total soil nitrogen, soil acidity, moisture, volatile and organic matter, matter insoluble in hydrochloric acid of 1.115 specific gravity, and the silica, iron, alumina, lime, magnesia, soda, potash, sulphate and phosphate in the portion of the soil soluble in hydrochloric acid.

METHODS OF ANALYSIS.

The determination of the soil constituents was made by the Official Method for Soil Analysis* modified as follows:

Preparation of the Sample.—Reduce the material to a fairly fine state of division by gently crushing on a clean hard wood board with a wooden roller and allow to dry for several days in the air at room temperature. The air dry soil is further reduced by gently rubbing in a mortar with a wooden pestle and the fine earth removed through a 20 mesh sieve.

(1) *Reaction of the Soil.*—Mix 10-20 grm. of soil with water to make a thin paste, allow to settle and introduce a piece of sensitive litmus paper into the supernatant fluid. (This test may be satisfactorily made by dividing a lump of moist soil with a clean knife, placing the test paper between the freshly exposed surfaces and pressing firmly together. Avoid touching the paper with moist fingers. Perspiration shows acid reaction with litmus.

(2) *Moisture.*—Weigh out accurately 5 grm. of air-dried soil, which has passed through a 20 mesh sieve, in a weighed porcelain crucible and dry to constant weight in an air bath at 105° Cent. From the percentage of moisture calculate the moisture factor. [See (6) p. 53.]

(3) *Volatile and Organic Matter.*—Heat the crucible and soil from determination (2) to full redness until all organic matter is burned off. If appreciable quantities of carbonates are present in the soil, moisten the contents of the crucible, after cooling, with a few drops of ammonium carbonate solution. Then dry, heat carefully only to dull redness and expel ammonium salts, cool and weigh. The loss represents water of combination, salts of ammonium, organic matter, etc.

(4) *Hydrochloric Acid Digestion.*—Place 20 grm. of air-dried soil and 200 cc. of hydrochloric acid of sp. gr. 1.115 in a flask fitted with a ground-glass stopper, carrying a condenser. Place in a water bath so that the level of the water comes above the level of the soil, and boil for ten hours continuously, shaking the flask once an hour. Pour contents of flask into a 400 cc. beaker and thoroughly rinse out the flask. Then filter the solution, using two 12.5 cm. filters if necessary, and employing suction with a platinum cone in the tip of the funnel if advisable. Wash thoroughly with hot water until free from chlorides. Dry the residue, ignite thoroughly and weigh. (*Insoluble matter.*)—Evaporate the clear filtrate in an

*Bull. 46, U. S. Department of Agriculture, Division of Chemistry.

evaporating dish, not more than 100 cc. at a time, until the total bulk is about 500 cc. then add 10 cc. of concentrated nitric acid, cover until spattering ceases and evaporate to dryness. Take up with aqua regia (1 part conc. nitric acid and 3 parts conc. hydrochloric acid), cover until spattering ceases and evaporate again to dryness. Repeat until organic matter is oxidized. Then take up with conc. hydrochloric acid and evaporate to dryness. Repeat once. When dry place in air bath and heat at 120° until there is no odor of hydrochloric acid. Take up with conc. hydrochloric acid, mix thoroughly and add water; warm until everything but the silica is dissolved. Filter and wash with hot water. Dry the residue, ignite and weigh (*Soluble Silica*).

Make the filtrate to 1000 cc. and label it Solution (A).

(a) Fe_2O_3 , Al_2O_3 and P_2O_5 collectively.—To 100 cc. of solution A add 4-5 cc. of conc. hydrochloric acid and bring almost to a boil. Then carefully pour in a slight excess of ammonium hydroxide and boil for about one minute. Allow the precipitate to settle for a few minutes, filter and wash with hot water a few times. Dissolve the precipitate in dilute nitric acid. Heat until the solution becomes perfectly clear and reprecipitate with a slight excess of ammonium hydroxide. Filter, wash thoroughly with hot water, dry the precipitate and ignite in a weighed platinum crucible, cool, moisten with a few drops of nitric acid, ignite again and finally heat with the blast. Cool in a desiccator and weigh. The increase in weight represents Fe_2O_3 , Al_2O_3 and P_2O_5 .

(b) *Calcium Oxide*.—Have the combined filtrates from (a) slightly alkaline with ammon. hydroxide. Bring to a boil and add 20-25 cc. saturated solution of ammon. oxalate. Boil for a few minutes, then let stand until the precipitate settles. Filter and wash a few times with water. Dissolve the calcium oxalate through the paper with warm dilute nitric acid (1-5), add a few drops of ammon. oxalate to the solution, render alkaline with ammon. hydroxide and boil. Let stand as before and filter through the same paper. Wash the precipitate thoroughly with 1% ammon. oxalate solution. Dry and ignite the precipitate gently at first, then finish with the blast to constant weight so that the calcium oxalate may be entirely converted into calcium oxide.

(c) *Magnesium Oxide*.—Have the combined filtrates from (b) concentrated to about 200 cc., cool and slightly alkaline, and add slowly with constant stirring about 30 cc. of 10% sodium phosphate solution. Let stand one hour and add 30 cc. conc. ammon.

hydroxide; then let stand 12 hours. Filter and wash with water containing $\frac{1}{4}$ its volume of ammon. hydroxide. Dry the precipitate and ignite*. Moisten with a few drops of nitric acid and ignite again. Finish with the blast and weigh as magnesium pyrophosphate. Calculate to magnesium oxide. The factor for MgO in $Mg_2P_2O_7$ is 0,36036.

(d) *Ferric Oxide*.—(1) Evaporate 100 cc. of solution A in an evaporation dish with 10 cc. of dilute sulphuric acid to fumes of SO_3 . Dilute carefully with water and transfer to a 600 cc. Erlenmeyer flask. Have an excess of sulphuric acid present. Add zinc and keep on water bath till iron is all reduced. Test this by removing a drop and adding hydrochloric acid and ammon. sulphocyanide. When no red color is produced by this treatment, showing that no ferric iron is present, cool rapidly with ice, decant from any undissolved zinc that may be present and titrate with $N/10$ K MnO_4 . If more convenient the iron may be reduced in a sulphuric acid solution by passing it through a column of shot zinc, known as a "reductor." Calculate to F_2O_3 .

(2) The iron may be determined in 100 cc. of solution A by the Zimmerman-Richards method, as follows: Drive off the excess of hydrochloric acid, reduce the hot solution with a solution of stannous chloride, using one drop in excess after the color disappears, and add ice to cool rapidly. Then add 20 cc. of saturated mercuric chloride solution (the separation should have a silky appearance), 10 cc. of titrating solution (manganese sulphate and phosphoric acid) and titrate quickly with standard potassium permanganate.

(e) *Phosphoric Acid Anhydride*.—Evaporate 100 cc. of solution A to about 25-30 cc. Neutralize with ammon. hydroxide and add enough nitric acid to dissolve the precipitate. Add 10 grm. solid ammon. nitrate and warm to about 60° . Then pour in 50 cc. of molybdic solution, shake well and let stand in a warm place

*Caution is necessary if the operation is conducted in a platinum crucible. Carbon reduces magnesium pyrophosphate at 950° . The phosphorous, resulting from this reduction, attacks and seriously damages platinum through the formation of crystalline platinum phosphide. This filter paper with the precipitate should be completely ashed over a Bunsen burner. Do not ignite strongly nor heat to the fusing point of the residue until the material in the crucible is white (carbon free). If the ashing of the paper has been imperfect (mass not white), allow the crucible to cool, moisten the residue with a few drops of nitric acid, carefully evaporate the acid and heat again in the Bunsen flame. This treatment must be repeated until the residue becomes white, then ignite strongly with the blast until the residue ceases to decrease in weight.

over night. Filter off the yellow precipitate* and wash thoroughly with a solution containing 1% nitric acid and 10% ammon. nitrate. Dissolve in dilute ammon. hydroxide, wash the filter thoroughly with hot water, neutralize the solution with hydrochloric acid, then make very slightly alkaline with ammon. hydroxide. (The volume of the solution should, if necessary, be reduced to about 200 cc.) Add 15-20 cc. of magnesia mixture slowly with constant stirring. Let stand an hour and add 30 cc. conc. ammon. hydroxide. Allow to stand 12 hours, filter, wash with water containing one-fourth its volume of ammon. hydroxide, dry, ignite** and weigh as magnesium pyrophosphate as under (c). Calculate to P_2O_5 . The factor for P_2O_5 in $Mg_2P_2O_7$ is 0.63758.

(f) *Aluminum Oxide*.—Add together the weights of Fe_2O_3 and P_2O_5 . Subtract the sum from the combined weight of the oxides determined in (a). The result is the weight of Al_2O_3 .

(g) *Sulphuric Acid Anhydride*.—Evaporate 100 cc. of Solution A to dryness. Take up with 2 cc. of concentrated hydrochloric acid and 150 cc. of water. Heat to boiling and add drop by drop 5 cc. of a 10-per cent solution of barium chloride. Continue boiling for 5 minutes and let settle. If the precipitate does not settle quickly, boil again, or keep at a temperature just below boiling until it does settle. Filter and wash with hot water until the washings show no trace of chlorides. Ignite, let cool, moisten with a few drops of concentrated sulphuric acid and ignite again, gently at first. Weigh $BaSO_4$ and calculate SO_3 . The factor for SO_3 in $BaSO_4$ is 0.34296.

(h) *Potassium and Sodium Oxides*.—Treat 100 cc. of solution A., or the filtrate from (g), except that the precipitate of iron should be dissolved in hydrochloric instead of nitric acid, as in (a). Evaporate filtrate and washings to dryness, heat below redness until ammonium salts are expelled, dissolve in about 25 cc. of hot water, add 5 cc. of barium hydroxide solution, and heat to boiling. Let settle a few minutes and test a little of the clear liquid with more barium hydroxide solution to be sure than enough has been added. When no further precipitation is produced, fil-

*This residue may be washed with 1 per cent nitric acid, then with 1.5 per cent potassium nitrate solution, the filter and contents placed in the precipitation flask, or beaker, a known amount of standard sodium hydroxide solution added to dissolve the precipitate and the excess of sodium hydroxide determined by titrating with standard nitric acid, using phenolphthalein as indicator, 46 parts of sodium correspond to 1 part phosphorus pentoxide.

**See foot note, page 50.

ter and wash with hot water. Add ammonium hydroxide and carbonate to complete precipitation of barium, let stand a short time on the water bath, filter and wash the precipitate thoroughly with water. Evaporate filtrate and washings to dryness in a porcelain dish, expel ammonium salts by heat below redness, take up with a little hot water, add a few drops of ammonium hydroxide and a drop or two of ammonium carbonate, let stand a few minutes on the water bath and filter when cool into a weighed platinum dish. Evaporate to dryness on the water bath and heat to dull redness, until all ammonium salts are expelled and the residue is nearly or quite white. The heat must not be sufficient to fuse the residue. Cool in a desiccator and weigh KCl and NaCl. Dissolve the mixed chlorides in a little water. If there is an insoluble residue, filter, evaporate filtrate to dryness in a weighed platinum dish, heat to dull redness, cool and weigh the chlorides again. If all the residue dissolves place in a small porcelain evaporating dish, add a drop of HCl and 3-5 cc. of platinic chloride solution, and evaporate on a water bath almost to dryness. *Remove the dish before the mass is entirely dry.* Let cool and add 80% alcohol. Allow to soak for about 15 minutes, then filter** through a weighed Gooch filter, wash with alcohol, dry in air bath and finally heat up to 120° to constant weight. From weight of K_2PtCl_6 calculate KCl and subtract from weight of combined chlorides. The difference is NaCl. Calculate to Na_2O . From K_2PtCl_6 calculate K_2O .

(5) *Nitrogen*.—Place 10 grm. of soil in a long-necked Kjeldahl flask, together with 10 grm. of potassium bi-sulphate and 30 cc. pure conc. sulphuric acid. Support the flask at an angle of 45° on a wire gauze and heat cautiously with a small flame. After the first violent action ceases add about 1 grm. of C. P. copper sulphate and continue boiling until oxidation is complete.

When cool transfer the digested mixture to a 700 cc. Erlenmeyer flask, equipped with a three-hole rubber stopper bearing a separatory funnel, a glass tube of sufficient length to almost touch the bottom of the flask and a potash safety bulb, connected with a long Liebig's condenser. Tie down the rubber stopper and place 25 cc. of normal hydrochloric acid in a receiving flask

**This double salt may be decomposed by gently heating in a weighed platinum dish with a few crystals of oxalic acid, the platinum residue washed with water, dried, ignited and weighed. The weight of metallic platinum multiplied by 0.48125 gives the weight of potassium oxide; the weight of platinum multiplied by 0.76142 gives the weight of potassium chloride. The weight of sodium chloride multiplied by 0.53078 gives the weight of sodium oxide.

(cap. 500 cc.) at the end of the cooler. Run in a strong solution of caustic soda through the separating funnel to alkaline reaction and distill with steam until the distillate attains a volume of almost 400 cc. Titrate the distillate in the presence of methyl-orange with normal sodium hydroxide. One cc. of normal hydrochloric acid corresponds to .014 grams of nitrogen.

(6) Calculate the results of the analysis of the air-dried soil to a moisture free basis by multiplying the percentage of each constituent by the moisture factor. The factor is found as follows: If moisture in the air dry sample is 3.19%, then $100 - 3.19 = 96.81$; $100 \div 96.81 = 1.032$ moisture factor.

TABLE SHOWING THE RESULTS OF THE ANALYSES.

COLLECTOR, SOIL SAMPLE, DESCRIPTION.	Shannon, No. 94. First Ft.	Shannon, No. 94. Second Ft.	Shannon, No. 94. Third Ft.	Shannon, No. 95. First Ft.	Shannon, No. 95. Second Ft.	Shannon, No. 95. Third Ft.	Shannon, No. 208. Surface.	Shannon, No. 208. Subsoil.	Ward, No. 1. Volusia Silt Loam.	Ward, No. 2. Miami Silt Loam.
LABORATORY NUMBER.....	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
Reaction to litmus.....	Acid.	Acid.	Acid.	Acid.	Acid.	Acid.	Acid.	Acid.	Acid.	Acid.
Moisture from air dry at 105°C.....	3.19	5.24	6.77	2.87	5.81	9.20	3.95	5.56	3.87	3.66
Total soil nitrogen.....	.222	.074	.121	.201	.103	.120	.138	.140	.115	.088

ANALYSIS OF FINE EARTH DRIED AT 105°C.

COLLECTOR, SOIL SAMPLE, DESCRIPTION.	Shannon, No. 94. First Ft.	Shannon, No. 94. Second Ft.	Shannon, No. 94. Third Ft.	Shannon, No. 95. First Ft.	Shannon, No. 95. Second Ft.	Shannon, No. 95. Third Ft.	Shannon, No. 208. Surface.	Shannon, No. 208. Subsoil.	Ward, No. 1. Volusia Silt Loam.	Ward, No. 2. Miami Silt Loam.
Volatile and organic.....	4.860	3.976	5.252	4.188	5.620	9.380	3.737	2.147	3.910	3.359
Insoluble in 1.115 HCl.....	85.230	80.939	74.847	86.078	75.110	61.083	84.208	79.200	83.272	84.567
Soluble Silica.....	.044	.066	.014	.045	.190	.065	.065	.050	.034	.086
Ferric oxide (Fe ₂ O ₃).....	3.560	5.201	6.646	3.517	6.216	8.716	4.061	5.230	3.993	4.345
Alumina (Al ₂ O ₃).....	4.668	8.191	10.363	4.432	10.376	15.775	6.034	9.870	7.860	6.167
Phosphoric acid anhydride (P ₂ O ₅).....	.159	.182	.137	.131	.213	.248	.184	.150	.137	.153
Calcium oxide (CaO).....	.509	.541	1.278	.692	.732	2.695	.523	1.277	.169	.428
Magnesium Oxide (MgO).....	.543	.520	.568	.519	.769	1.139	.709	.889	.378	.639
Sulphuric acid anhydride (SO ₃).....	.072	.066	.045	.067	.066	.068	.033	.044	.046	.021
Potassium Oxide (K ₂ O).....	.367	.482	.535	.336	.505	.704	.415	.677	.359	.383
Sodium oxide (Na ₂ O).....	.200	.197	.222	.214	.206	.385	.234	.373	.098	.164
Total.....	100.212	100.361	100.007	100.219	100.003	100.278	100.263	99.907	100.256	100.372

TABLE SHOWING THE RESULTS OF THE ANALYSES—Continued.

COLLECTOR, SOIL SAMPLE, DESCRIPTION.	Ward, No. 3. Waverly Silt Loam.	Ward, No. 4. Waverly Silt Loam.	Ward, No. 5. Upland Limestone S.	Ward, No. 6. Upland Limestone S.	Ward, No. 7. Limestone and Shale Soil.	Ellis, No. 26. Second Ft.	Ellis, No. 26. First Ft.	Ellis, No. 10. First Ft.	Ellis, No. 16. Ohio Valley.	Ellis, No. 10. Second and Third Ft.
LABORATORY NUMBER.....	11.	12.	13.	14.	15.	16.	17.	18.	19.	20.
Reaction to litmus.....	Neutral.	Neutral.	Acid.	V. F. Acid.	V. F. Acid.	Acid.	Neutral.	Neutral.	Neutral.	Acid.
Moisture from air dry at 105°C.....	2.63	2.20	1.23	4.73	3.74	1.95	1.50	2.87	2.29	4.08
Total soil nitrogen.....	.165	.280	.101	.116	.183	.133	.099	.108	.092	.085

ANALYSIS OF FINE EARTH DRIED AT 105°C.

COLLECTOR, SOIL SAMPLE, DESCRIPTION.	Ward, No. 3. Waverly Silt Loam.	Ward, No. 4. Waverly Silt Loam.	Ward, No. 5. Upland Limestone S.	Ward, No. 6. Upland Limestone S.	Ward, No. 7. Limestone and Shale Soil.	Ellis, No. 26. Second Ft.	Ellis, No. 26. First Ft.	Ellis, No. 10. First Ft.	Ellis, No. 16. Ohio Valley.	Ellis, No. 10. Second and Third Ft.
LABORATORY NUMBER.....	11.	12.	13.	14.	15.	16.	17.	18.	19.	20.
Volatile and organic.....	5.940	6.428	3.268	4.353	6.342	2.170	2.623	4.002	5.207	3.501
Insoluble in 1.115 HCl.....	85.270	80.029	93.033	78.695	74.985	90.835	92.902	85.808	83.691	83.445
Soluble silica.....	.071	.044	.124	.076	.075	.043	.074	.085	.074	.096
Ferric oxide (Fe ₂ O ₃).....	3.047	5.290	1.094	5.370	6.508	2.313	1.715	3.488	4.053	4.359
Alumina (Al ₂ O ₃).....	3.253	5.536	1.673	8.588	7.195	3.205	2.037	5.350	4.357	7.214
Phosphoric acid anhydride (P ₂ O ₅).....	.275	.220	.111	.210	.571	.177	.119	.176	.278	.244
Calcium oxide (CaO).....	1.162	1.444	.306	.764	1.300	.597	.266	.229	.852	.190
Magnesium oxide (MgO).....	.437	.932	.201	.859	.380	.621	.267	.572	1.003	.627
Sulphuric acid anhydride (SO ₃).....	.050	.056	.042	.036	.039	.024	.018	.032	.057	.033
Potassium oxide (K ₂ O).....	.321	.148	.347	.726	.855	.226	.131	.371	.491	.346
Sodium oxide (Na ₂ O).....	.171	.132	.233	.252	.044	.138	.111	.120	.206	.148
Total.....	99.997	100.260	100.432	99.929	99.914	100.349	100.268	100.233	100.269	100.203