Test Method

Total Surface Area by retention of Ethylene Glycol Monoethyl Ether (EGME) RC 355.01

1. Scope

This test is used to determine the total surface area of powdered samples of rock or soil. The total surface area is here defined as the sum of the external and internal surface areas of the clay minerals present in the sample. As the clay mineral species are the major contributors to surface area in powdered mineral sample, the test gives some indication of the amount of clay present in a sample.

2. Apparatus and Materials

(a) Vacuum pump - capable of reducing pressure to 0.1 mm Hg or better.

(b) Calibrated pressure gauge - range 0.05 to 1.0 mm mercury. A McLeod gauge (Fig. 1) has been found suitable.

(c) Vacuum desiccator - with porcelain support and safety shield (Fig. 1).

(d) Culture chamber - consisting of glass dish with cover and brass mesh support for aluminium weighing cans (Fig. 1).

(e) Porcelain evaporating dish - (Fig. 1).

(f) Aluminium weighing cans - with fitted lids, measuring about 50 mm diameter, 25 mm deep.

(g) Analytical balance with a limit of performance not exceeding 0.0005 g.

(h) Oven - thermostatically controlled to operate at a temperature of 220 ± 2°C.

(i) Sieves - 200mm diameter, AS sizes: 13.2, 9.5 and 2.36mm, 75 µm.

(j) 1000 mL pyrex glass beaker.

(k) Stainless steel spatula - about 150mm long.

(l) 2 mL dropping pipette.

(m) Anhydrous calcium chloride - Ca Cl_2 laboratory grade, 8 - 24 mesh.

(n) Ethylene glycol monoethyl ether (EGME) - CH_3 - CH_2 - 0 - CH_2 - CH_2 - OH, laboratory grade.

(o) Phosphorus pentoxide - P_2O_5 laboratory grade.

Additional Apparatus and Materials required for removal of Organic Matter where necessary

(p) Water bath.

(q) Vacuum filtration apparatus - consisting of water pump, Buchner funnel and filter flask.

(r) Class fibre filtering paper - Whatman grade GF/C.

(s) 500 mL pyrex beaker.

(t) Dilute hydrochloric acid - about 1%.

(u) Hydrogen peroxide - 27% w/w laboratory grade.

(v) 2 molar calcium chloride - prepared by dissolving 60g of laboratory grade calcium chloride dihydrate, Ca $C1_2$ - $2H_2O$, in a 200mL volumetric flask with distilled water and diluting to volume.

3. Safety

Exercise great care in using phosphorus pentoxide in particular, since it avidly absorbs moisture to form phosphoric acid.

Rubber gloves and eye protection should be worn when handling phosphorus pentoxide as this chemical has a strongly irritating and corrosive effect on the skin. In case of accidental skin contact quickly wash the affected area with copious amounts of running water.

When not in use phosphorus pentoxide should be kept in a covered porcelain dish and stored in a drying cabinet. For the purposes of this test, the chemical is exhausted when an abundance of discoloured lumpy pieces are apparent.

4. Test Sample Preparation

4.1. Rock Aggregate

(a) Select about 80g of typical aggregate pieces passing a 13.2mm sieve and retained on a 9.5mm sieve.

(b) Grind the pieces to pass a 75 µm sieve.

4.2. Non-Organic Soil

(a) Split out about 60g of soil passing a 2.36mm sieve.

(b) Grind the soil to pass a 75 µm sieve.

4.3. Soil Containing Organic Matter

(a) Powder the soil to pass a 2.36 mm sieve.

(b) Weigh out 8g of powdered soil, place in a 500mL beaker and add 100mL of distilled water.

(c) Add dilute hydrochloric acid dropwise to obtain a pH of 6.

(d) Add 5 mL portions of 27% w/w hydrogen peroxide to the soil-liquid mix while swirling the mix in the beaker. Allow frothing to subside prior to adding each further portion.

(e) When further additions of hydrogen peroxide cease to produce frothing, stop the addition and place the beaker over a water bath for an hour at a temperature of 60- 70°C.

(f) Vacuum filter the mix using a glass fibre paper, grade GF/C. Wash the retained powder with three 20mL portions of distilled water.

(g) Saturate the sample with calcium by washing with three 20mL portions of 2 molar calcium chloride.

(h) Remove the excess calcium chloride by washing as in (f).

(i) Air dry the washed sample.

(j) Grind the sample to pass a 75 µm AS sieve.

5. Solution Preparation

Prepare a fresh $CaCl_2$ - EGME solvate prior to testing.

(a) Weigh 108 ± 1 g of $CaCl_2$ in a porcelain dish and place in an oven at 220 ± 2°C for at least 2 hrs to remove all traces of water.

(b) Remove the $CaCl_2$ from the oven and let it cool for 5 minutes.

(c) Transfer the dry $CaCl_2$ to a 1000 mL pyrex beaker and add 30 ± 1 g of EGME.

(d) Quickly mix the contents thoroughly with a spatula to allow the exothermic reaction to proceed.

(e) While the solvate is still hot, spread it uniformly over the bottom of the culture dish and replace the cover. Let the solvate cool to near ambient conditions before placing samples within the culture chamber.

6. Procedure

Operation of a McLeod gauge

To make a pressure reading, tilt the gauge so that the end connected to the rubber tubing is pointing upwards. Prior to making a pressure reading, make sure that the system has been evacuated to a degree that the pressure is within the range of the gauge. (Evacuating continuously for 5 minutes has been found sufficient).

If the pressure is much above the range of the gauge, an air lock will form upon gauge inversion, the removal of which is a lengthy and tedious task.

(a) Prepare $CaCl_2$ - EGME solvate as given in 5 above.

(b) Weigh an aluminium can to the nearest 0.0001 g and record (M_c).

(c) Place approximately 1 g of prepared sample into the can, weigh to the nearest 0.0001 g and record (M_t) (Note 1).

(d) Spread the sample evenly in the can and place the lid at a slightly skewed position (see Fig. 1) to eliminate powder loss through sudden surges that may occur in the evacuated system.

(e) Place about 250g of anhydrous phosphorus pentoxide in a porcelain dish and transfer to the bottom of the dessicator.

(f) Place the aluminium can on the porcelain support and transfer to the dessicator.

(g) Evacuate the dessicator for 1 hour (stopcock A closed, stopcock V open - See Fig. 1).

(h) Check the pressure gauge to ensure that evacuation has been achieved. This is indicated by a reading equivalent to a pressure of less than 0.50mm Hg.

(i) Close stopcock V then open stopcock A to atmospheric pressure prior to turning the vacuum pump off. This prevents back pressure from developing on the non return valve of the vacuum pump.

Make sure that all the stop cock movements are gradual so as to avoid excessive pressure surges within the system.

Switch off the vacuum pump and maintain the dessicator sealed for 45 minutes.

(j) Determine the pressure of the evacuated system by reading the pressure gauge and using a correlation table to convert the reading to pressure in mm of mercury. Record the pressure.

(k) Repeat (g) to (j) for one further cycle.

If the equilibrium pressure as measured in step (j) is above 0.50mm Hg after the 2 hours of evacuations, repeat steps (g) to (j) until the pressure as measured in step (j) is maintained at less than 0.50mm Hg for 45 minutes.

Check for leaks if further evacuations do not reduce the pressure to the required level.

(l) Open the stopcock to the air entry to allow the dessicator to regain atmospheric pressure then remove the can.

(m) Seat the can lid correctly and, to minimise adsorption of atmospheric moisture by the sample, immediately weigh the can with vacuum dried sample to the nearest 0.0001 g and record (M_d)

(n) Wet the sample in the can with about 2mL of EGME and swirl it gently to form a mineral-adsorbate slurry. Replace the can lid at a skew, allowing a small edge opening for vapour loss.

(o) Transfer the can to the culture chamber, replace the cover and allow the sample to stand for 1 hour.

(p) Remove the dish containing the posphorus pentoxide from the dessicator and store as described in 4 above.
Place the culture chamber in the dessicator.

(q) The pressure during the evacuation stage should reduce to 0.25 mm Hg and rise to 0.50 mm Hg during the sealed stage. If these pressures are not attained, check for and eliminate leaks and repeat the cycles.

(r) Remove the can from the dessicator, seat the can lid correctly and immediately weigh the can with sample to the nearest 0.0001 g and record (M_e).

7. Calculations

Calculate the total surface area (S_t) to the nearest 0.1 from:

$$S_t = \frac{M_e - M_d}{2.86\times10^{-4}} \times \frac{1}{M_d - M_C}$$

where:

M_c = mass of empty can (g)

M_d = mass of can containing vacuum dry sample (g)

M_e = mass of can containing sample with equilibrium amount of EGME (g)

8. Reporting

Report the total surface area in m^2/g to the nearest whole number.

Notes

Note 1

M_t is not used in determining the total surface area however it can be used to determine the amount of moisture loss taking place during the evacuation of the sample. This has been shown to correlate well with the amount of interlayer water present in the sample.

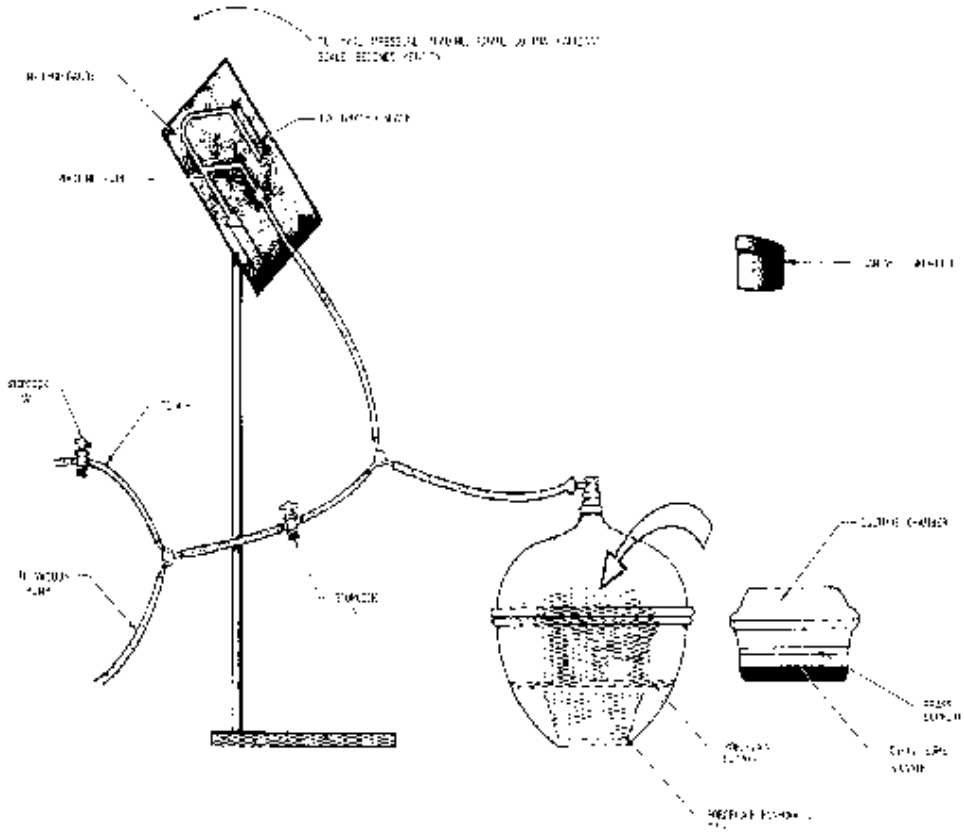

Figure. 1. Arrangement of apparatus

Test Method - Revision Summary

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Date	Clause Number	Description of Revision	Authorised by
June 2012	Full document	Re-styled with minor corrections made	Principal Advisor – Pavements & Materials