U. S. DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

Determination of the True Density of Pulverized Coal Samples

by

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ABSTRACT

A method using the gas-comparison pycnometer with helium gas as the penetrating medium measures precisely the true volume of a pulverized coal sample. The true density of a solid is calculated as the true unit volume of the solid exclusive of its pore space which is divided into the weight of the sample.

The method is similar to that used to determine the density of refractory materials but the procedure is modified to yield precise density determinations of coal samples. These modifications diminish effects of trapped moisture and gases on the volume measurement.

The helium gas-comparison pycnometer method is rapid, reliable, precise, and requires minimal analytical equipment and sample preparation, and also is non-destructive to the coal sample. Using this method, densities can be determined on coal samples of subbituminous to low-volatile bituminous rank and perhaps also on samples of lignite. The density of anthracite samples has not been determined by this method.

INTRODUCTION

This report describes a method of using a helium gas-comparison pycnometer to obtain the true volume of a coal sample. This method is a modification of the American Society of Testing Materials procedure (ASTM, 1979a) used to determine the density of refractory materials.

The modified procedure suggested here has been used specifically on coal samples; results of experiments used to determine the limits and conditions for this procedure are given in the appendices.

METHODS USED TO DETERMINE DENSITY OF COAL SAMPLES

The density of a solid is defined as the mass of a unit volume at a specified temperature; the volume shall be that of the impermeable part of the solid exclusive of pore volume (ASTM , 1979b).

Precise measurements of the mass of a solid can be easily obtained using an analytical balance; however, precise measurements of the solid's volume exclusive of its pore volume are difficult to obtain (Tschamler and de Ruiter, 1963). The principle types of density are defined as follows:

True density - the mass of a unit volume of a solid in air exclusive of any pore volume whether permeable or impermeable. The determination of this value requires that all pores are penetrated by a medium that does not interact with the substance (Tschamler and de Ruiter, 1963).

Apparent density - the weight in air of a unit volume of a solid impermeable material at a stated temperature (ASTM, 1979a). The determination of this value permits that impermeable pores be included in the measurement of the volume of the solid.

Bulk density - the weight in air of a unit volume of a solid including both the permeable and impermeable voids which are normal to the material at a stated temperature (ASTM, 1979a).

The most common methods of measuring true density of coal use helium as the penetrating medium (Palvelev, 1939; Smith and Howard, 1942; Franklin, 1949; Pruss, 1958; Ettinger and Zhupakhina, 1960; Tshamler and de Ruiter, 1963; Gan and others, 1972; Mahajan and Walker, 1978, 1981;and Institute of Gas Technology (IGT), 1978).

Apparent and bulk density determinatons are commonly made by displacement of liquids that do not penetrate all pores (IGT,1978). The Hogarth specific gravity bottle (pycnometer) (Blair, 1906) has been used to determine the "true specific gravity" of coal (Fieldner and Selvig, 1951; U.S.Bureau of Mines (USBM), 1967) and coke (ASTM, 1981a). This pycnometer method uses distilled water that is mixed with the coal and boiled to wet the hydrophobic coal particles (Ettinger and Zhupakhina, 1960). However,because of the inability of water to totally displace air contained in pores, the Hogarth pycnometer method actually only measures apparent densities rather than true specific gravities (Tschamler and de Ruiter, 1963; IGT, 1978) and yields only approximate results (Ettinger and Zhupakhina, 1960). A modification of this method uses 'OP-7' as a wetting agent which is a mixture of polyethyleneglycol-monoalkylphenyl ethers (Ettinger and Zhupakhina, 1960). Other liquids such as alcohol, kerosene, toluene, xylene (Ettinger and Zhupakhina, 1960) methanol, benzene, and carbon tetrachloride (Tschamler and de Ruiter, 1963) have been used as media to determine densities of coal but these liquids are not satisfactory because they may not penetrate completely and may react with the coal chemically and physically (Ettinger and Zhupakhina, 1960). Density determinations have also been made on micro-scale (10 mg) samples using a graded density aqueous solution of potassium mercuric iodide contained within a hollow prism (Jones, 1961).

Four important factors in the determination of true density are:1) the pore size of a solid substance; 2) the permeability of the solid substance; 3) the molecular size of the penetrating medium; and 4) the possibility of chemical or physical interaction between the solid substance and the penetrating medium.

The smallest pores in coal average 5-8 Å in diameter (van Krevelen, 1961). A large number of the pores of high volatile C bituminous coals range from 90 to 220 Å; high volatile A bituminous coals have pores smaller than 35 Å (Thomas and Damberger, 1976, p. 35). Impermeability due to closed pore space in coal can cause erroneous density determinations. A review of the literature on pore space in coals of different ranks reveals that much is not known concerning the signifcance of pore volume and how much error can be attributed to closed pore volume. Kotlensky and Walker (1960) showed data for one anthracite sample in which as much as 34 percent of the total pore space is closed to helium penetration; this data was determined by comparison of pore spacing (revealed by x-ray diffraction) to helium density. Franklin (1949) stated on the contrary that there is no appreciable volume of pores in coals that are closed to helium that would significantly affect the density determinations.

Helium has a molecular diameter of about 2 Å (Franklin, 1949) and is the smallest available atom other than hydrogen (Mahajan and Walker, 1981) which can penetrate pores in coal. Helium however, may be adsorbed weakly by coal (Kini and Stacey, 1963) or not significantly enough to cause additional errors in measurement (Maggs and others, 1960). Mahajan and Walker (1981) concluded that in the literature, densities determined with helium may be high because of helium adsorption; in practice, however, they assume no helium adsorption in their methods of volume measurement (Mahajan and Walker, 1978). Significant adsorption does occur in activated carbons and chars so that high temperatures are required to prevent adsorption during volume measurements (Kini and Stacey, 1963; Maggs and others, 1960).

The methods to determine density that use helium as a penetrating medium can be separated into three: 1) one method involves measuring pressures at a known temperature of a known volume of helium of a sample holder with and without the coal sample and then calculating the volume (hence density) of the sample using the ideal gas law (Smith and Howard, 1942; Franklin, 1949; Fugii and Tsoboi, 1967; Gan and others, 1972; and Mahajan and Walker, 1978). This method requires an elaborate vacuum system and the procedure is cumbersome and time-consuming (Mahajan and Walker, 1981; Davis, 1978) although a high degree of precision is obtainable (1.320 + 0.002 g/cm^3 for 7 grams of coal; Mahajan and Walker, 1978).

2) A second method involves measuring the actual volume of a helium saturated holder with the sample compared to the volume of the holder without the sample (Pruss, 1958). This method uses a glass burette in which the volume of the displaced gas is precisely measured to the nearest 0.004 cm^3. Although apparently simpler than the first method, it requires maintaining very stable pressure and temperature conditions.

3) A third method measures volumetric differences in the volume displaced in two cylinders of an air comparison pycnometer, one with and the other without the sample (Smith and Kerr, undated; IGT, 1978). This method (Beckman Instruments, Inc., 1977) has been used primarily as a method for determining "specific gravity" in refractory materials (ASTM, 1979a). ASTM (1979a, p. 566) presented a formula for calculating the "true

specific gravity of the sample at room temperature as compared to water at 4°C as:

$$S = W/V$$

where
S= true specific gravity,
W= grams of sample and
V= average sample volume, cm^3 (average of two measurements)."

This calculation however, yields the sample's density and not its "true" specific gravity.

Precision estimates using this method were determined to be 0.0107 g/cm^3 within a laboratory and 0.0106 g/cm^3 between laboratories (ASTM, 1979b, p. 566).

This third method as described below, has been used by the U. S. Geological Survey,in Reston, Va. with modifications that permit reproducible density determinations. These modifications include using helium gas as a penetrating medium, adequate drying of the coal sample to remove moisture, and increased periods of time required for equipment equilibration. Smith and Kerr (undated) described unsuccessful attempts to determine the volume of ground coal using the a gas-comparison pycnometer; however, no modifications to the method were used.

GAS-COMPARISON PYCNOMETRY OF COAL SAMPLES

I.PRINCIPLE OF THE GAS-COMPARISON PYCNOMETER

The principle of the gas-comparison pycnometer (fig. 1) is as follows: each of two chambers (A and B) contains a piston (reference piston and measuring piston, respectively). The chambers are connected by a coupling valve. With the coupling valve closed, any change in the position of either piston must be compensated by a change in the other to maintain the same pressure reading on the differential pressure indicator.

If a sample (Vx) is inserted into the sample cup of chamber B, the coupling valve is closed, and both pistons are advanced from position 1 to 2, the pressures will not remain the same. However, if the pressures in both chambers are to be maintained equal (which means that the differential pressure indicator remains on zero), piston B must be moved to position 3. The volumetric difference between positions 2 and 3 (dx) is equal to the volume occupied by the sample (Vx). If the piston in chamber A is always moved the same distance each time a measurement is made (restricted by mechanical stops), the differential volume (dx) will be proportional to the volume of the sample (Vx). The volume dx is calibrated and read directly in cubic centimeters on a scale or digital counter.

II. PROCEDURE FOR DENSITY DETERMINATIONS

Apparatus

The basic apparatus required for this method are:

1. Analytical balance, 200 gram capacity with sensitivity of 0.01 gram.
2. Desiccator, with drying agent
3. Oven, capable of heating to and maintaining 110°C.
4. Grinding unit or mortar and pestal, to reduce sample size to pass a no. 20 sieve.
5. Air (gas) comparison pycnometer, readability to 0.01 cm^3 and equipped with external purge and vacuum manifold.
6. Helium which is passed through a drying agent such as magnesium perchlorate or calcium sulfate.

Sample preparation

1. A representative subsample (approximately 500 grams) from a pulverized (-8 mesh) bulk sample is pulverized to pass a no. 20 sieve in order to permit further representative subsampling . Approximately 25 cm^3 of coal (approximately 20 grams) is used for the density determination (the volume of the sample cup is 50 cm^3).
2. The sample is placed in a flat glass dish and dried in an oven for 4 or more hours at 105-110°C.
3. After drying, the sample is placed in the desiccator and permitted to cool to room temperature in the presence of a drying agent such as calcium sulfate.

Weighing of the sample

1. The dried sample is placed into the clean pycnometer sample container
2. The tared sample weight is measured using an analytical balance and the weight is recorded to the nearest 0.01 gram .

Volume measurement of the sample

1. The reference piston of the gas comparison pycnometer is moved to stop A. The measuring piston is moved to an estimated volume (approximately 20 cm^3).
2. The sample and cup are locked firmly into the unit (empty if determining zero correction).A zero correction additive is determined for each sample by measuring the volume of the empty sample container and then calculating the difference from 0.00. This value is added to the average of the three subsequent sample volume measurements.
3. The purge valve and coupling valve are opened.
4. The vacuum pump is turned on and the vacuum valve is opened for 10 seconds (longer depending on pump); the valve is then closed.
5. The gas valve is opened for 5 - 10 seconds to purge the system with dry helium gas at 3 - 4 psi.
6. The vent valve is opened for 5 seconds -- escaping gas should be audible.
7. The purge valve is closed.
8. The reference piston is moved to position 1.(fig. 1)
9. The measuring piston is moved to position 1.(fig. 1)
10. The measuring piston is then moved to the calibrated reading on the volume indicator (starting number). This moves the piston in the direction towards position 3. (fig. 1)

11. After 60 seconds (temperature equilibration), the coupling valve is closed.
12. Both pistons are advanced simultaneously so that the pointer on the differential pressure indicator is kept to the left of zero on the scale until the reference piston comes to stop A.
13. After 60 seconds (equilibration), the measuring wheel is advanced to bring the pointer on the pressure indicator to zero.
14. After 10 seconds, the coupling valve is opened.
15. If the pointer on the pressure indicator does not move, the reading is accepted; if pointer moves, the procedure is repeated again beginnning at step 2.
16. Each sample is measured 3 times within a single run -- readings should not differ more than 0.20 cm^3, this difference causes a change of .01 in the density calculation (using 20 or more grams of sample). Depending on outgassing problems of the sample, readings are often within 0.10 cm^3. Zero is checked between sample runs and a different zero correction may be required for each sample.

    NOTE 1. If the zero check yields a positive reading, this value is given a negative value for adding to the sample reading (in effect subtracting the absolute value). If the zero check yields a negative reading, then the absolute value of this reading is added to the sample reading.

Calculation of density

The sample's true density is calculated by dividing the average of the three volume measurements into the sample weight in grams:

$$D = G/x$$

where D=sample density, G=sample weight in grams, and x = average sample volume in cm^3.

III. LIMITS AND CONDITIONS

As determined from four experiments (see appendices A-D for details) the following empirical limits and conditions are suggested for this method:

Sample particle size (Appendix A)	pass through no.20 sieve
Sample amount (Appendix B)	
minimum	5 grams coal
maximum	about 25 grams coal
Drying temperature (Appendix C)	105 - 110° C
Drying time (Appendix C)	
minimum	1 hour

maximum	not determined
suggested	4 hours
Rank of coal(see Note 2) (Appendix C)	subbituminous to low-volatile bituminous
Precision (Appendix D) (for 26 grams of coal)	
volume measurements	.07 cm^3
density	.007 g/cm^3

NOTE 2: This method has been used, in this lab, for the determination of the density of lignite samples, and the applicability and limitations of this method for lignite is being evaluated. Data reported by Dulhunty (1947, 1950) and Dulhunty and Penrose (1951) suggest that irreversible changes accompanying air-drying of Australian lignites caused deformation of micelles and reduction of intervening spaces so as to preclude determination of density in helium. This method has not been evaluated for determining the density of anthracite.

The instrument accuracy of the pycnometer is calibrated periodically (once every week) by measuring the volume of solid steel balls of known volumes. The standardization is accurate to within + 0.015 cm^3(Beckman Instruments, Inc., 1977).

Humidity affects the zero correction in the volume measurement (fig. D-1, Appendix D) because of the moisture absorption tendency of coal which makes it necessary to check the zero between each sample volume determination (set of three) . Optimum laboratory conditions for minimum zero correction are estimated to be about 50 percent relative humidity and 70° F.

SUMMARY

The helium gas-comparison pycnometer can be used in the determination of the density of coal. The method is a modification of the ASTM (1979a) standard procedure used for density determinations of refractory materials.

The coal samples are pulverized to a particle size of minus 20 mesh, air-dried in an oven at 110°C to remove moisture and then weighed to the nearest 0.01 gram. The volume of the weighed sample is determined using a helium gas-comparison pycnometer. Sample density is then calculated from the sample weight in grams divided by its average volume in cubic centimeters.

The main disadvantage of this method is that it is not as precise as some others. Its precision is +.007 g/cm^3 (Appendix D) as compared to +.002 g/cm^{3}for the method of Mahajan and Walker (1978) in which the volume is calculated from the ideal gas law.

The advantages of the helium gas-comparison method for the determinations of densities of subbituminous and higher rank coals and associated rocks are: 1) density determinations are accomplished easily and quickly, 2) densities can be determined for the same sized particles as those used for petrographic work, and 3) samples are not altered chemically and can be analyzed further using destructive techniques such as determination of ash content or forms of sulfur.

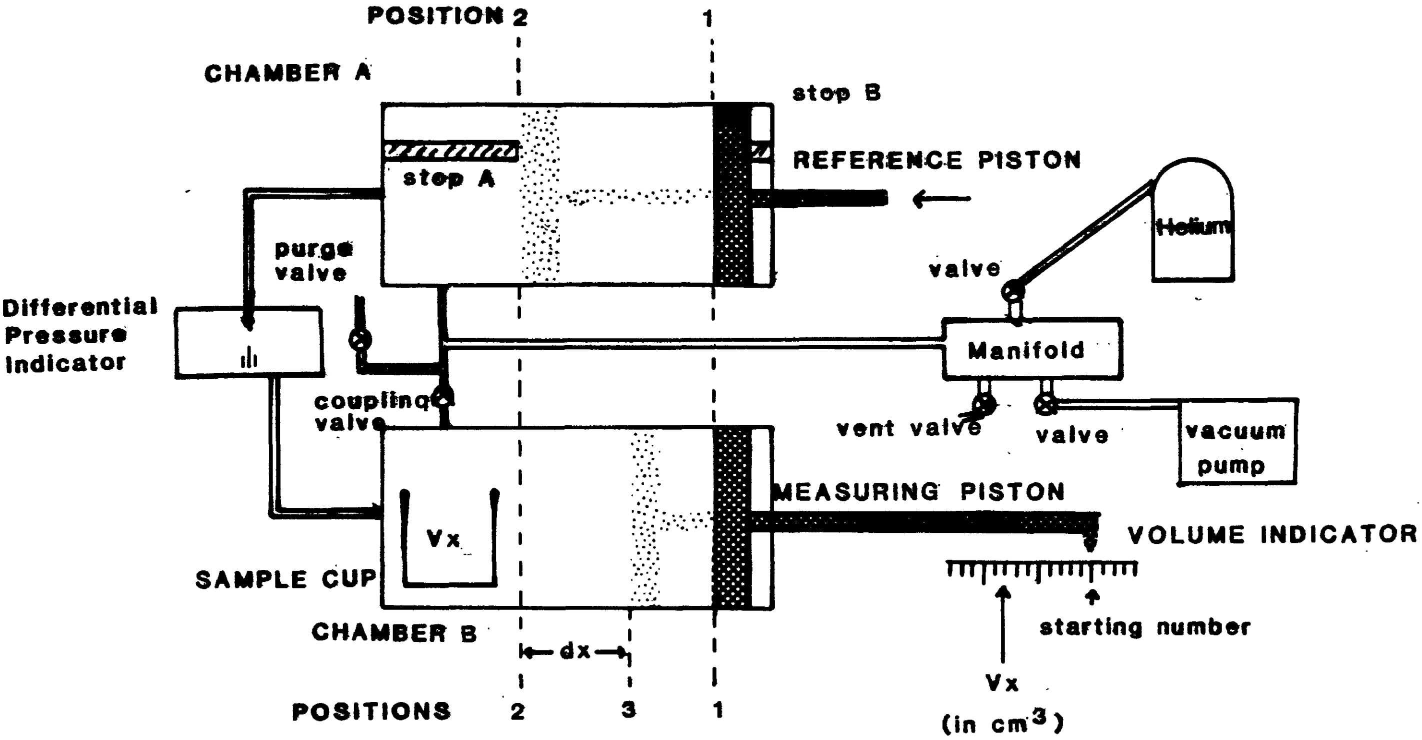

Figure 1 – Schematic diagram showing gas–comparison pycnometer (modified from Beckman Instruments, Inc., 1977)

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APPENDIX A
EFFECT OF SAMPLE PARTICLE SIZE

PARTICLE SIZE (mesh)	WEIGHT (grams)	DENSITY (grams/cm^3)
- 20	22.99	1.365
- 60	22.59	1.379
- 140	20.36	1.373
- 200	18.62	1.381

average of all four determinations =1.3745 g/cm^3
standard deviation of all four determinations =0.007 g/cm^3

A coal sample of approximately 20 grams of the Upper Freeport coal bed was pulverized to pass through a no. 20 sieve and then oven dried. The volume of this sample was determined as an average of three measurements. The density was then calculated as the weight divided by the average volume and then rounded off to the nearest 0.001 g/cm^3. Additional density determinations on the same subsplit were made after each subsequent stage of pulverizing and redrying to pass through no. 60, no. 140, and no. 200 mesh sieves.

On the basis of these results, pulverizing to a grain size less than 20 mesh does not yield different densities +.01 g/cm^3.

APPENDIX B
EFFECT OF SAMPLE AMOUNT

SAMPLE WEIGHT (grams)	VOLUME (cm^3)	DENSITY (grams/cm^3)
26.30	18.58	1.416
10.00	7.11	1.41
5.00	3.55	1.41
2.00	1.40	1.43
2.00	1.46	1.37
1.00	0.72	1.4
1.00	0.70	1.4
0.50	0.33	1.5
0.48	0.35	1.4

In this experiment, the effect of sample amount (weight) on volume measurements was investigated. Average volumes from triplicate measurements were obtained on smaller and smaller splits of a medium volatile coal pulverized to pass 20 mesh. The results indicate that reliable results may be obtained on samples as small as 5 grams. Densities of samples in amounts less than 5 grams will differ by more than 0.01 g/cm^3.

APPENDIX C
EFFECT OF DRYING TIME ON DENSITY

DRYING	MEDIUM VOLATILE BITUMINOUS COAL		SUBBITUMINOUS COAL	
(HOURS)	WEIGHT (grams)	DENSITY (g/cm^3)	WEIGHT (grams)	DENSITY (g/cm^3)
1	20.47	1.353	23.41	1.388
2	20.44	1.357	23.20	1.395
4	20.44	1.354	23.05	1.390
6	20.43	1.353	22.96	1.387
8	20.38	1.355	22.90	1.387
20	20.37	1.349	22.84	1.390
24	20.36	1.357	22.81	1.391

Two coal samples of different ranks, were oven dried at 110°C for varying lengths of time. After each additional interval of drying, the densities were determined. The conclusion is that 1 hour is the minimum drying time although 4 hours is a suggested time for establishing constant density determinations of bituminous and subbituminous coal samples. Longer periods of drying appear not to affect the density determination significantly. It is only important that the sample is dried to eliminate most of the moisture and 4 hours is a reasonable time.

APPENDIX D

DAILY DENSITY CALCULATION OF A REFERENCE SAMPLE

ZERO COR-RECTION	HUMIDITY (% rel.)	TEMP. (° F)	WEIGHT (grams)	UNCORRECTED VOLUME MEASUREMENTS 1	2	3	STANDARD DEVIATION OF VOLUME	CORRECTED AVERAGE VOLUME	ZERO CORRECTED AVERAGED DENSITY
-.14	48	70	26.74	18.96	19.02	18.93	0.0458	18.97	1.410
-.10	44	72	26.78	19.54	19.10	19.06	0.2663	19.23	1.392
-.12	41	72	26.67	19.12	18.95	18.96	0.0954	19.01	1.403
-.10	44	72	26.72	19.04	18.98	19.00	0.0306	19.01	1.403
-.13	38	70	26.65	19.04	18.95	18.88	0.0802	18.96	1.406
-.18	31	71	26.64	19.38	18.88	18.81	0.3109	19.02	1.406
-.12	36	69	26.52	18.69	18.75	18.75	0.0346	18.73	1.416
-.14	36	73	26.54	18.80	18.74	18.74	0.0346	18.76	1.415
-.14	38	72	26.48	18.56	18.75	18.66	0.0950	18.66	1.419
-.15	36	73	26.46	18.56	18.76	18.65	0.0608	18.66	1.418
-.12	40	75	26.42	18.66	18.68	18.66	0.0100	18.67	1.415
-.09	46	73	26.48	18.78	18.78	18.78	0.0000	18.78	1.412
-.11	42	71	26.43	18.77	18.69	18.69	0.0062	18.72	1.412
-.04	62	75	26.43	18.72	18.69	18.71	0.0153	18.71	1.413
-.13	48	75	26.30	18.61	18.57	18.55	0.0306	18.58	1.416
-.17	50	70	26.20	18.65	18.63	18.59	0.0306	18.62	1.407
-.02	48	69	26.22	18.50	18.51	18.56	0.0321	18.52	1.416
-.05	50	76	26.60	18.65	18.72	18.77	0.0603	18.71	1.421
-.04	60	71	26.27	18.52	18.55	18.55	0.0173	18.54	1.417
+.13	65	72	26.22	18.70	18.65	18.63	0.0361	18.66	1.405
-.03	75	69	26.18	18.31	18.50	18.51	0.1069	18.44	1.420
+.21	70	70	26.20	18.61	18.75	18.66	0.0709	18.67	1.413
+.08	70	75	26.39	18.88	18.76	18.68	0.1007	18.77	1.406
+.16	75	80	26.27	18.44	18.56	18.56	0.0693	18.52	1.418
+.08	82	75	26.52	18.62	18.64	18.66	0.0200	18.64	1.423
						mean =	0.0664	mean =	1.412
								standard deviation =	0.007

The density of a medium volatile bituminous coal sample was determined daily over a period of 26 working days. The humidity and temperature were recorded before each determination. The volume of the empty sample container was determined before each sample was analyzed. The difference between the volume reading and 0.00 was recorded as a zero correction additive to be added to the subsequent sample volume measurement. High and low humidity was found to affect the zero correction and the effects are believed to be minimal at about 50 - 60 percent relative humidity (fig. D-1). However, because the appropriate zero corrections are used to calculate the density, humidity does not affect the density determination (fig. D-2).

The precision for volume measurements is calculated as 0.0664 cm for this reference sample, which is the mean of the volume standard deviations (ASTM,1981a). The precision for the calculated density is 0.007 g/ cm^3, which is the standard deviation of the zero corrected averaged densities.

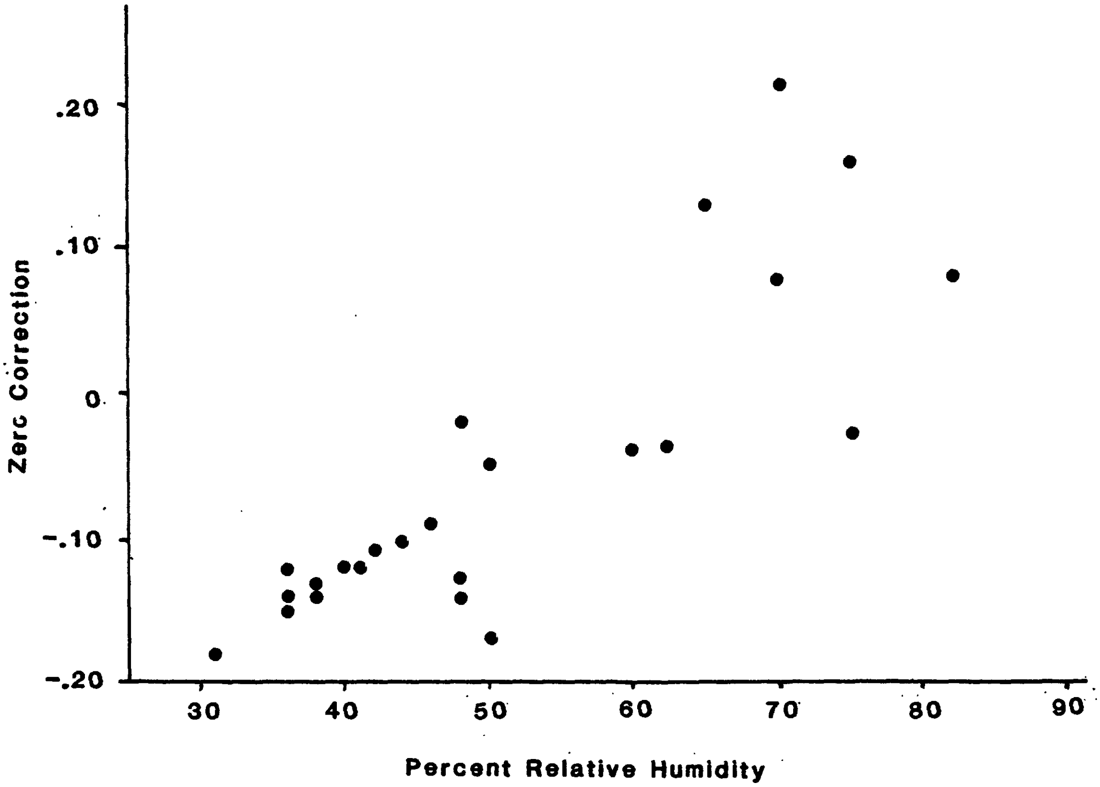

Figure D-1 - Zero Correction versus Percent Relative Humidity

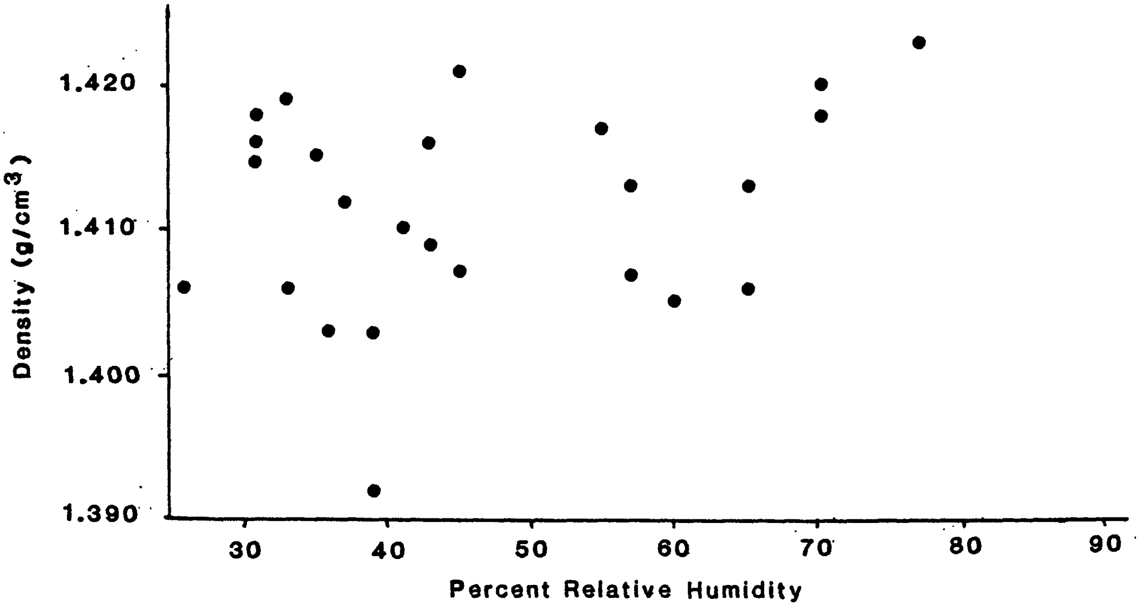

Figure D-2 - Sample Density using appropriate zero correction versus Percent Relative Humidity