A 11 a - Particle Density, Occluded Air and Interstitial Air by Air Pycnometer

GEA NIRO® Method No. A 11 a

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1. Definition

Particle density g/ml is defined as the mass of particles having a total volume of 1 ml.
Occluded air ml/100g is defined as the difference between the volume of a given mass of particles and the volume of the same mass of air-free solids.
Interstitial air ml/100g is defined as the difference between the volume of a given mass of particles and the volume of the same mass of 100x tapped powder.

2. Scope

This method may to be used for all powders.

3. Principle

The true volume of a sample (the volume in g/cm3 enclosed by its outer surface an excluding its open pores) is determined by measuring the pressure change of helium in a calibrated volume.

4. Apparatus

a. Analytical balance, capable of weighing to 0.1 mg.
b. AccuPyc 1330 Pycnometer, Micromeritics (Fig. 1 and 2).
c. LC-20 Dot matrix printer, Star.

5. Reagents

Helium (g).

6. Procedure

1. Adjust the pressure of the helium to 2 bar on the gas flask.
2. Check the parameters by pressing the white button and button No. 2 on the keypad. Press 'Enter'.
   The parameters must be as follows:
   Number of purges: 3.
   Purge fill pressure: 19.5 psig.
   Number of runs: 3
   Run fill pressure: 19.5 psig
   Equilibration rate: 0.050 psig/min.

Use run precision: No.

3. Press 'Save' to store the information. The display should show 'Reload'.
4. Weigh out an amount of powder into the sample cup and remove excess powder on the sides of the cup.
5. Remove the chamber cap by turning it counter clockwise, then lifting up. Insert the sample cup in the cell chamber and put on the chamber cap again.
6. Press the white button and button No. 4.
7. Type sample identification followed by 'Enter' and the sample weight followed by 'Enter'.
8. To start the analysis press 'Enter'.
9. When the analysis stops (after approx. 10-12 min.) the results are printed.
10. Determine the moisture content (Method A 1 a), the fat content (Method A 9 a) and the 100x tapped powder bulk density (Method A 2 a).

7. Result

The particle density Dparticle is calculated as:

$$D_{particle} = \frac{W_{sample}}{V_{sample}} \quad [g/ml]$$

wsample = Weight of the sample in g. vsample =
Volume of the sample in ml.
An example of the print of the results is seen on Fig. 3.

The theoretical density of powder solids Dsolids in milk powder is calculated as:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF/1.52 + \%W} \quad [g/ml]$$

%F = fat content
%SNF = solid non-fat content
%W = moisture content

For whey powder of normal composition, the following formula can be used:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF(whey)/1.58 + \%W} \quad [g/ml]$$

%F = fat content
%SNF = solid non-fat content in whey powder
%W = moisture content

Occluded air content Voa is calculated as:

$$V_{oa} = \frac{100}{D_{particle}} - \frac{100}{D_{solids}} \quad [ml/100g]$$

Dparticle = particle density (from 7.1) Dsolids =
density of powder solids (from 7.2)

Interstitial air content is calculated as:

$$V_{ia} = \frac{100}{D_{powder}} - \frac{100}{D_{particle}} \quad [ml/100g]$$

Dpowder = Powder bulk density, tapped 100x (from 6.10) Dparticle = particle density (from 7.1)

8. **Reproducibility**
Particle density ± 0.02 g/ml

9. **Remarks**

Particle densities measured on powders produced by injection of inert gas(ses), e.g. CO2 and N2, into the feed will depend on the pressure used for the determination. The results will often not be the same as those measured with the Beckman Air Pycnometer. For baby food, produced with CO2 injection a pressure change to 10 psig on the AccuPyc will result in the same values as previously measured by the Beckman Air Pycnometer.

If there is any excess powder on the outside of the sample cup or around the O-ring, the analysis stops.

The analysis time is approx. 10-12 min.

If the analysis takes more than 1000 secs the analysis stops automatically.

If the deviation between determinations is >0.01 the pressure must be changed.

The temperature of the helium used must be the same as that of the instrument, in order to get correct results.

When calculating powder of different compositions, the density and the amounts of the constituents must be taken in consideration. For this purpose, the following values may be used:

Powder material, moisture free	**Density at 20°C**
Whole milk powder (28% fat)	1.28
Non-fat milk solids	1.52
Milk fat in powder	0.94
Ca-caseinate phosphate complex	1.39
Amorphus lactose	1.52
Beta-lactose	1.59
Alpha-lactose monohydrate	1.545
Anhydrous alpha-lactose	1.545

Spray dried whey powder	1.58
Residual whey components	1.8
Demineralized whey powder	1.525

10. **Literature**

GEA Niro Research Laboratory

Micromeritics: 'AccuPyc 1330 Pycnometer' operator's manual.

Buma T.J.: 'The true density of spray milk powder and of certain constituents' (Netherlands Milk and Dairy Journal, 1965, 19, pp. 249-265)

Fig. 1 AccuPyc 1330 Pycnometer

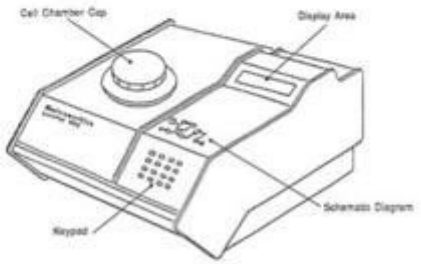

Fig. 2 Pycnometer Keys

Function	Keys	Used for
ZERO	0	Zero the pressure transducer
CALIBRATE	•	Calibrate the pycnometer
MANUAL	1	Manually control the valves. After pressing the MANUAL key, you may use the FILL EXPAND and VENT keys to open and close the valves

SET UP	2	Display or edit analysis parameters, report options, calibration volumes, data transmission parameters, unit types and operation language
TRANSMIT	3	Transmit analysis or calibration data over the serial line. If an automatic operation is in progress, transmit a partial report
ESCAPE	Clear	Delete all data entered in the current mode and return to display mode. If an automatic operation is in progress, cancel it
ANALYZE	4	Perform analysis
REVIEW	5	Review or edit completed analysis or calibration data
PRINT	6	Print analysis or calibration report. If an automatic operation is in progress, print partial report
FILL	7 (manual mode)	Open and close the fill valve. The indicator above the FILL key is on when the valve is open and off when the valve is closed
EXPAND	8 (manual mode)	Open and close the expansion valve. The indicator above the EXPAND key is on when the valve is open and off when the valve is closed
VENT	9 (manual mode)	Open and close the vent valve. The indicator above the VENT key is on when the valve is open and off when the valve is closed

Fig. 3 Example - print of results

ACCUPYC 1330 VI.03

Serial number: 529
Density and volume report Sample ID: 920268 Sample weight: 1.8702 g
Number of purges: 3

Equilibration rate: 0.0500 psig/min. Cell volume: 12.1664 cm^3 Expansion volume: 8.7231

Run	Volume cm^3	Deviation cm^3	Density g/cm^3	Deviation g/cm^3
1	1.4992	0.0001	1.2475	0.0001
2	1.5014	0.0023	1.2457	0.0019
3	1.4967	0.0024	1.2495	0.0020

Average volume: 1.4991 cm^3
Standard deviation: 0.0023 cm^3

Average density: 1.2475 g/ cm^3
Standard deviation: 0.0019 g/ cm^3