Letter to the Editor

Mass Basis Karl Fischer Titration Equation for Moisture Determination

Sirs:

In two papers (1,2) in which techniques developed to apply automatic Karl Fischer titration efficiently to the determination of moisture in solid materials (including grain) were described, equations used to calculate moisture content were presented. Various quantities were expressed on a volume basis to take advantage of the precision and convenience of using pipets and calibrated syringes for measuring the quantity of solvent used for extraction, the aliquot of solvent-extracted water mixture, and the quantity of the solvent blank. In a paper applying these techniques to the determination of moisture in sunflowerseed (3) and other papers in the offing, some analysts prefer to express quantities on a mass basis rather than on a volume basis. It is the purpose of this letter to develop and present a moisture content equation on a mass basis. The quantities, symbols and units used in the derivation of the equation are listed in Table I.

In the application of the titration techniques (1,2), water is extracted from m_g grams of solid (grain, seed, etc.) into M grams of solvent (usually methanol). An aliquot of m_s μg of the solvent-extracted water mixture or m_b μg of solvent blank is introduced into the reaction vessel of the automatic Karl Fischer titrator. The indication of the titrator, in ml of Karl Fischer reagent used or in μg of water titrated, is A_s for the aliquot or A_b for the blank. In some cases (oily seeds, for example), m_o g of extraneous substance such as oil are present in the solvent-extracted water mixture and must be accounted for in the equation.

Moisture content (mc), on a wet basis, of a solid sample is defined as the ratio of the mass of water in the sample to the mass of the sample, expressed as percentage. The mass of water in a sample of mass m_g is equal to $m_g \times (mc/100)$; the mass of water in the solvent used for extraction of water from the solid sample is $MC \times (A_b/m_b)$ where C is the standardization factor for the titrator. The mass of water in the mixture of solvent and extracted water is equal to:

$$MC \times (A_b/m_b) + m_g \times (mc/100)$$

The total mass of the mixture, m_x, is equal to:

$$M + m_o + m_g \times (mc/100)$$

The ratio of the mass of water in the mixture to the total mass of the mixture is thus equal to:

$$[MC \times (A_b/m_b) + m_g \times (mc/100)]/[M + m_o + m_g \times (mc/100)]$$

This ratio is also equal to the ratio of the mass of water in the aliquot of the mixture to the mass of the aliquot: $C \times (A_s/m_s)$.

The following equation results from setting the last two expressions equal and rearranging:

$$mc, \% = \left\{(MC/m_g) \times [A_s/m_s - A_b/m_b] + (m_oC/m_g) \times (A_s/m_s)\right\} \times 100/[1 - C(A_s/m_s)] \quad [1]$$

Equation 1 is the desired equation, including the mass of extraneous substance if present, with quantities expressed on a mass basis. As indicated in the earlier papers (1,2), m_o could be estimated by heating m_a g of the solvent-extracted water mixture until a "dry" residue of m_r g remained. The mass of extraneous substance, m_o, in the total mass of mixture, m_x, could then be calculated as $m_o = m_x \times (m_r/m_a)$.

TABLE I

Quantities, Symbols and Units Used in Derivation of Moisture Content Equation

Symbol	Quantity	Units
A_b	Titrator indication for solvent blank	μg of H_2O, or ml of Karl Fischer reagent
A_s	Titrator indication for solvent-extracted H_2O mixture	Same as A_b
C	Standardization factor for titrator	μg of H_2O/indicated μg of H_2O, or μg of H_2O/ml of Karl Fischer reagent used
M	Mass of solvent used for extraction	g
m_a	Mass of solvent-extracted H_2O mixture heated to estimate m_o	g
m_b	Mass of solvent blank	μg
m_g	Mass of sample of solid	g
m_o	Mass of extraneous substance in total mass of mixture, m_x	g
m_r	Mass of "dry" residue from m_a	g
m_s	Mass of aliquot of solvent-extracted H_2O mixture	μg
m_x	Total mass of solvent-extracted H_2O mixture	g
mc	Moisture content, defined in the text	percent

REFERENCES

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3. Robertson, J.A., and W.R. Windham, JAOCS 60:1773 (1983).

Frank E. Jones
Center for Chemical Engineering
National Bureau of Standards
Gaithersburg, MD 20899

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