vapor diffused rather uniformly through the entire surface of the capsule.

CONCLUSIONS

The water content of gelatin capsules may be subject to desorption in the presence of hygroscopic materials. Relative hygroscopicity of the gelatin capsules and the filling materials determines the direction of vapor phase transfer of water in the closed capsule system. Gelatin capsules offer little protection to a hygroscopic content from atmospheric water vapor. Certain coatings may enhance this protective function, and the extent of protection may be evaluated in terms of prolonged diffusion lag time.

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Notes

Determination of Calcium Carbonate Moisture Content by a Radioisotope Technique

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The transmission of beta particles through calcium carbonate samples with different moisture contents was studied. It was found that the transmission was linearly related to the moisture content.

In many pharmaceutical manufacturing processes the moisture content of various materials is important. Conventional methods for moisture analysis are time consuming and delay further processing and subsequent production steps. Thus, a definite need exists for a rapid, simple method for moisture analysis. The objective of this investigation was to study the feasibility of determining the moisture content of pharmaceutical products using the beta-particle transmission technique. Although this technique has been used for determining the moisture content of certain materials such as wood (1) and paper (2), no reference to its use for moisture analysis of pharmaceutical products has been reported.

Calcium carbonate was selected for this study because it is a component of many pharmaceutical products. The transmission of the beta particles from a SrY-90 source through calcium carbonate samples with known moisture contents was measured. It was found that the transmission was linearly related to the moisture content.

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EXPERIMENTAL

Radiation Counting Equipment and Radioactive Source.—A conventional decade scaler, a Geiger-Müller tube with a 1.4 mg./cm.^{2} end window, and a lead shield with a Lucite tube mount were used for the counter. The radioactive source was a $^3/_{16}$-in. diameter deposit of SrY-90 in the center of a $1^1/_4$-in. aluminum planchet having five concentric circular grooves on the bottom. The activity of the deposit was 218,000 c.p.m. at a distance of 2.4 cm. from the detector window. The deposit was covered with a thin coat of label glaze. The planchet was attached to an aluminum planchet holder which, in turn, was placed in the Lucite tube mount of the lead shield.

Preparation and Measurement of Samples.—Stock samples of wetted calcium carbonate were prepared from calcium carbonate powder (A.R. grade) and distilled water. For each stock sample, 100 Gm. of calcium carbonate was mixed with the calculated amount of water with a mechanical mixer. The samples were stored in screw-cap glass jars and the actual moisture contents were determined by gravimetric analysis using 2- to 3-Gm. samples dried to constant weight in weighing bottles at 100°.

Preliminary experimentation showed that the sample layer must be as uniformly dense as possible in order to get reproducible results. Slugs having convex surfaces were made from the stock samples with a 1-in. diameter punch and die set and a Carver laboratory press. The appropriate weight of sample, determined from a mass absorption curve, was added to the die, and a pressure of 5000 pounds was gradually applied to the sample. The moisture content of the slugs was limited to a range of 0.5–10%, since below 0.5% the powder was too dry to press into slugs and above 10% water droplets were pressed out of the powder. Each slug was placed in a geometrically reproducible position over the SrY-90 source under the Geiger-Müller tube in the lead shield. The distance from the top of the slug to the thin window of the tube was 1.8 cm. The beta-particle transmission was determined during 2 minutes.

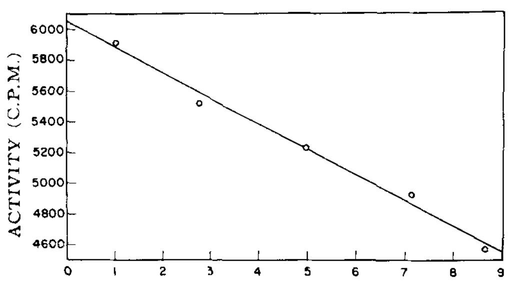

Fig. 1.—SrY-90 beta-particle transmission through wetted calcium carbonate using a constant dry sample weight.

RESULTS

Five stock samples with moisture contents ranging from 1.04 to 8.65% were studied using a constant dry-sample weight of 2.75 Gm. The beta-particle transmissions of four slugs from each stock sample were measured and the average transmission was calculated. The moisture contents and average transmissions of the five stock samples were: 1.04%, 5,913 ± 131 c.p.m.; 2.79%, 5,521 ± 73 c.p.m.; 4.97%, 5,229 ± 47 c.p.m.; 7.13%, 4,930 ± 61 c.p.m.; and 8.65%, 4,568 ± 72 c.p.m. Figure 1 is a least squares plot of these data.

Four stock samples with moisture contents ranging from 1.04 to 7.13% were studied using a constant wet-sample weight of 2.89 Gm. The beta-particle transmissions of four slugs from each stock sample were measured and the average transmission was calculated. The moisture contents and average transmissions of the four stock samples were: 1.04%, 4,857 ± 88 c.p.m.; 2.79%, 5,033 ± 88 c.p.m.; 4.97%, 5,162 ± 41 c.p.m.; 7.13%, 5,294 ± 41 c.p.m. Figure 2 is a least squares plot of these data.

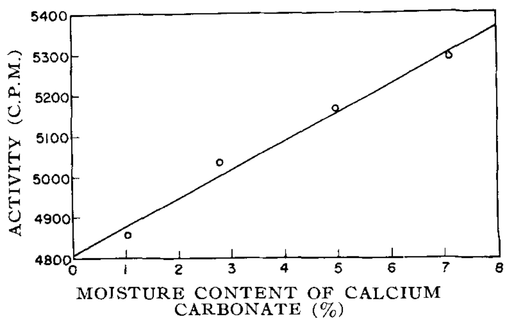

Fig. 2.—SrY-90 beta-particle transmission through wetted calcium carbonate using a constant wet sample weight.

DISCUSSION

The precision of the determinations could be improved by reducing the counting error. The standard deviations of the four determinations for each moisture content range from ±0.80 to ±2.21%. However, the expected counting error for the 2-min. counts is about 1%. The counting error could be reduced by either taking longer counts or using a more intense beta source. If a more intense beta source is used, a scintillation counter with an anthracene crystal would be desirable to avoid coincidence corrections.

The beta-particle transmission technique should be useful in routine moisture analyses of materials when moisture content is the only variable. Calibration curves relating beta-particle transmission to moisture content would be required for each material.

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