

that contains the lead equivalent of the heavy metals limit of the test.

**§ 436.209 Melting range or temperature.**

(a) *Apparatus.* Melting range apparatus consists of a glass container for a bath of colorless fluid, a suitable stirring device, an accurate thermometer, and a controlled source of heat. Any apparatus or method of equal accuracy may be used. The accuracy should be checked periodically by use of melting point standards, preferably those that melt near the expected melting range of the product to be tested. The bath fluid is selected with a view to the temperature required, but light paraffin is used generally and certain liquid silicones are well adapted to the higher temperature ranges. The fluid is deep enough to permit immersion of the thermometer to its specified immersion depth so that the bulb is still 2 centimeters above the bottom of the bath.

(b) *Sample preparation.* If necessary, reduce the sample to a fine powder and store it in a desiccator over sulfuric acid for 24 hours. If a method for loss on drying is included in the section for the antibiotic to be tested, a sample dried by that method may be used.

(c) *Test procedure.* Use a capillary glass tube about 10 centimeters long and 0.8 to 1.2 millimeters internal diameter with the wall 0.2 to 0.3 millimeter in thickness. Charge the tube with a sufficient amount of the dry power to form a column 2.5 to 3.5 millimeters high from the sealed end when packed down as closely as possible by moderate tapping on a solid surface. Heat the bath until a temperature  $10^{\circ} \pm 1^{\circ}$  C. below the expected melting range is reached, then introduce the charged tube, and heat at a rate of rise of  $3^{\circ} \pm 0.5^{\circ}$  C. per minute until melting is completed. The temperature at which the column of the sample is observed to collapse definitely against the side of the tube at any point is defined as the beginning of melting, and the temperature at which the sample becomes liquid throughout is defined as the end of melting or the melting point.

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**§ 436.210 Specific rotation.**

(a) *Test procedure.* The appropriate solvent, test concentration, and polarimeter tube length are specified in the section for each antibiotic to be tested. Accurately weigh the sample to be tested in a glass-stoppered volumetric flask, dissolve in the appropriate solvent, and dilute to the specified test concentration at 25° C. Maintain the solution at 25° C. and transfer to the appropriate polarimeter tube. Determine the angular rotation of both solvent and sample solution in a suitable polarimeter, using a sodium light source or a white light source with a 589.3-millimicron filter. The zero correction is the average of the blank readings and is subtracted from the average observed rotation of the sample solution if the two figures are of the same sign, or is added if they are opposite in sign, to give the corrected angular rotation of the sample solution. The determination must be completed within one-half hour from the time the solution is prepared.

(b) *Calculations.* Determine the specific rotation,  $[\alpha]$ , by the following formula:

$$[\alpha]_t = \frac{100a}{lc}$$

where:

*a*—The corrected angular rotation of the sample solution in degrees at temperature *t* using a light source of a wavelength of *x* millimicrons;

*l*—The length of the polarimeter tube in decimeters;

*c*—The concentration of the solution expressed as number of grams of substance in 100 milliliters of solution.

**§ 436.211 Identity test by infrared spectrophotometry.**

(a) *Apparatus*—(1) *Spectrophotometer.* A suitable spectrophotometer capable of recording the infrared absorption spectrum in the 2 to 15 micron range.

(2) *Hydraulic press.* A 30-ton hydraulic press with 12-inch square platens.

(b) *Sample preparation methods.* Use the sample preparation method specified in the individual section for each antibiotic.

(1) *Potassium bromide discs.* Quantities of materials specified are for a 13-millimeter die. Appropriate adjustments