⟨311⟩ ALGINATES ASSAY

APPARATUS

The required apparatus (see *Figure 1*) contains a capillary metering valve, A, followed by a flowmeter, B, to control and monitor the flow of nitrogen through the system. Halogenated vinyl plastic tubing* and a rubber fitting, C, are used to connect the flowmeter to a sidearm of a reaction flask, D. Flask D is a 250-mL round-bottom, boiling flask, resting in a suitable heating mantle, E. Flask D is provided with a 225-mm Hopkins coil reflux condenser, F. The condenser terminates in a U-shaped trap, G, which contains two 25-g bands of 20-mesh zinc, the bands being bounded and separated by three 3-inch plugs of glass wool. The trap terminates in an adapter, H, that by means of a halogenated vinyl plastic tubing and a twistcock connector, I, connects with a 250-mL gas washing bottle, J. The inlet (bubbling) tube extends almost to the bottom of the gas washing bottle, and it terminates in a fritted disk having a coarse porosity. The size of all glass joints is $^{24}/_{40}$, except for the $^{45}/_{50}$ joint of the gas washing bottle.

* This type of tubing is commonly referred to as Tygon tubing. This note is added for clarity and it does not constitute USP's endorsement of this product.

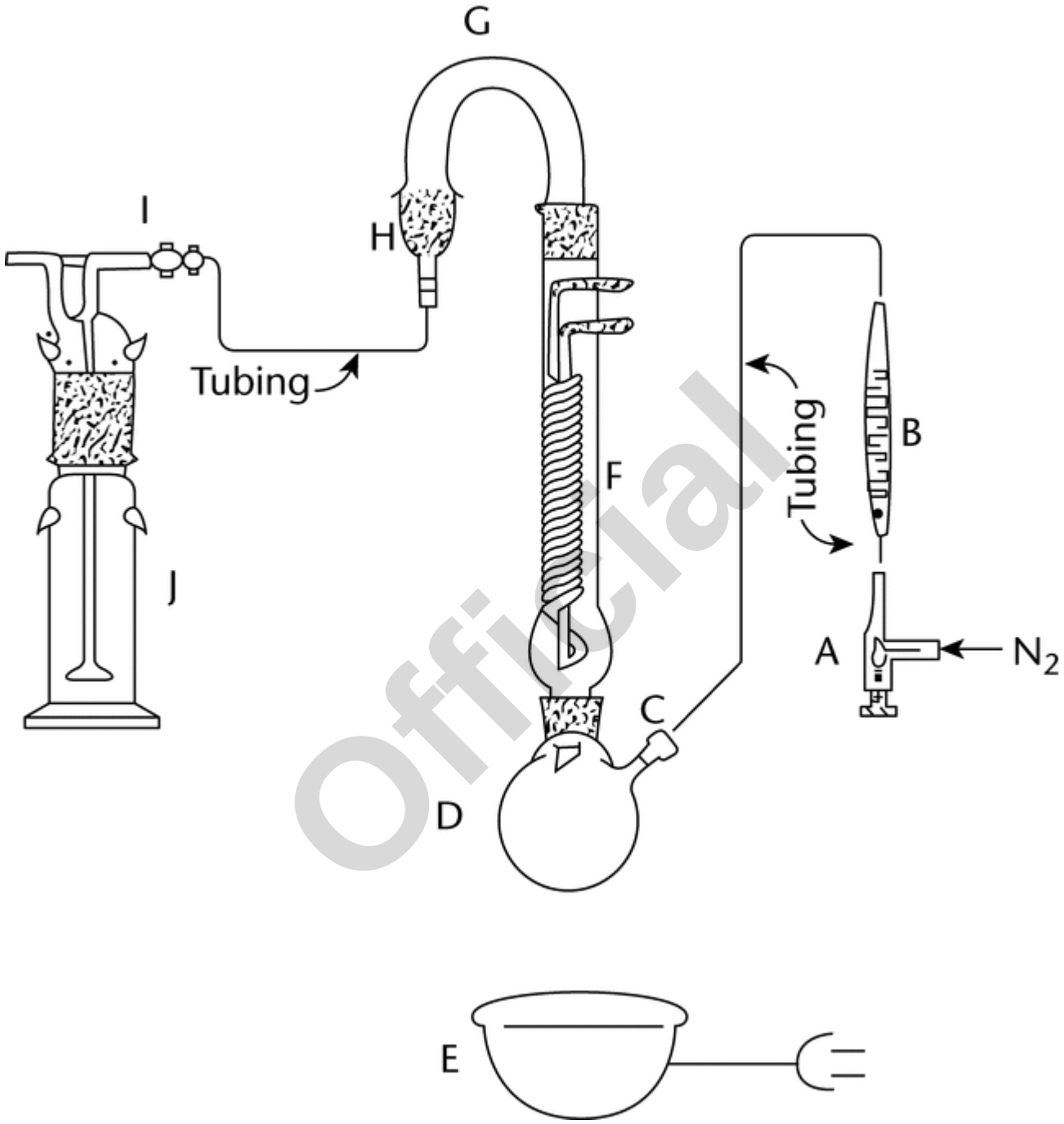

Figure 1. Apparatus for Alginates Assay

SYSTEM SUITABILITY

Using D-glucuronolactone as the standard, proceed as directed for *Procedure,* but do not perform the preboiling steps. The system is suitable if the following criteria are met: (1) a blank determination results in a net titration value, C, between 0.02 and 0.06 mEq, calculated as follows:

$$A_b - B_b$$

in which A_b is the number of mEq of 0.25 N sodium hydroxide in the 25 mL used, and B_b is the number of mEq of 0.1 N hydrochloric acid used in the blank titration; and (2) the percentage of carbon dioxide, CO_2, obtained from the standard is between 24.2% and 25.7%.

PROCEDURE

Unless otherwise directed in the individual monograph, transfer a specimen of about 250 mg, accurately weighed, into the reaction flask, D, add 50 mL of 0.1 N hydrochloric acid, insert several boiling chips, and connect the flask to the reflux condenser, F, using phosphoric acid as a lubricant. [NOTE—Stopcock grease may be used for the other connections.] Connect the nitrogen line to the sidearm of the flask, and adjust the flow of cooling water to about 2 L per minute.

[NOTE—The following preboiling steps, outlined in this paragraph, are optional and need only be performed when the presence of inorganic carbonates is suspected.] Maintain the flow of nitrogen through the apparatus at 90 to 100 mL per minute. Raise the heating mantle, E, to the flask, heat the specimen to boiling, and boil gently for 2 minutes. Turn the heat off, lower the mantle, E, and allow to cool for about 10 minutes.

Connect the empty gas washing bottle assembly, J, and sweep the system with nitrogen at a rate of 90 to 100 mL per minute for 5 minutes. Reduce the nitrogen flow to 60 to 65 mL per minute, add 10 drops of butyl alcohol, 25.0 mL of 0.25 N sodium hydroxide VS, and 50 mL of distilled water into the bottle, rinsing down the inside of the gas washing bottle, and replace the cap. Detach the rubber fitting, C, from sidearm, and add 46 mL of hydrochloric acid through the sidearm of the boiling flask. Reattach the nitrogen line, raise the heating mantle, and heat the reaction mixture to boiling. After 2 hours of boiling, increase the nitrogen flow to 90 to 100 mL per minute, discontinue the heating, and lower the mantle. Allow to cool for 10 minutes. Disconnect, and disassemble the gas washing bottle. Using a directed stream of distilled water, thoroughly rinse all parts of the bubbling tube and cap, collecting the washings in the gas washing bottle. Use nitrogen to gently force all water out of the bubbling tube. To the bottle immediately add 10 mL of 10% barium chloride solution and a stirring bar. Insert a tight stopper, and stir gently for 1 minute. Allow to stand for at least 5 minutes. Add three drops of phenolphthalein TS, and titrate with 0.1 N hydrochloric acid VS. Perform a blank determination (see *Residual Titrations* under *Titrimetry* ⟨541⟩). Calculate the percentage of carbon dioxide, CO_2, by the formula:

$$2200[(A - B) - C]/(1000W)(1 - D)$$

in which A is the number of mEq of 0.25 N sodium hydroxide in the 25 mL used; B is the number of mEq of 0.1 N hydrochloric acid used for the titration of the sample or the standard; C is the net titration value calculated in the blank determination; W is the weight, in g, of the sample or the standard taken; and D is the percentage expressed as a decimal (1 decimal place), obtained in the test for *Loss on drying* for the sample or for the standard.