

## Alginates Assay (Carbon Dioxide Determination by Decarboxylation)<sup>1</sup>

This method can also be used for gellan gum.

### Apparatus

The apparatus required is shown in Figure 1. It consists of a capillary metering valve, A, followed by a flow meter, 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 flow meter 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 contain 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 twist cock 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 terminated 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.

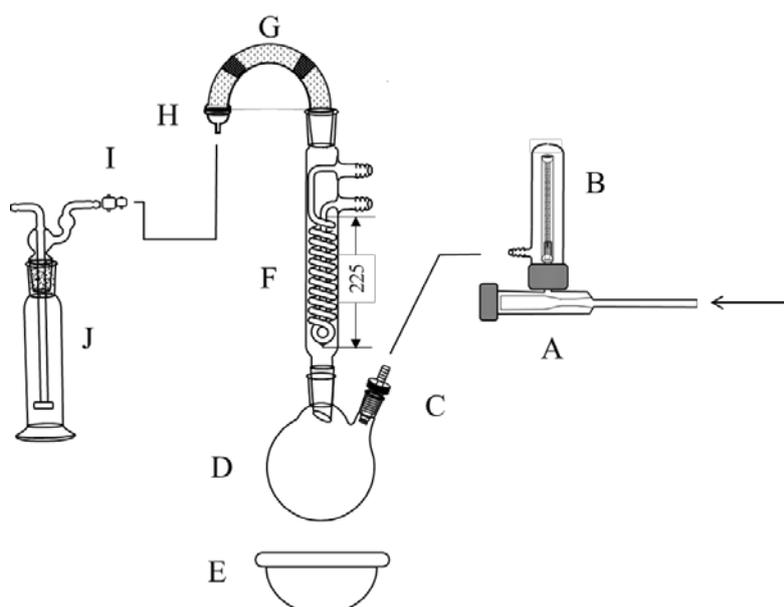


Figure 1. Apparatus

### System suitability

Using D-glucuronolactone as the standard, proceed as directed for procedure, but do not perform the preboiling steps. Calculate the value for system suitability using the formula. The value for system suitability should be between 0.02 and 0.06.

$$\text{Value for system suitability} = C_{\text{NaOH}} \times V_{\text{NaOH}} - C_{\text{HCl}} \times V_{\text{blank}}$$

Where

$C_{\text{NaOH}}$  is the concentration of sodium hydroxide solution added, mol/l

$V_{\text{NaOH}}$  is the volume of sodium hydroxide solution pipetted, ml

$C_{\text{HCl}}$  is the concentration of hydrochloric acid, mol/l

$V_{\text{blank}}$  is the volume of hydrochloric acid used for the titration of the blank, ml

### Procedure

<sup>1</sup> Adapted from USP 37-NF32 with permission. Copyright 2013. The United States Pharmacopeia Convention

Weigh accurately about 0.25 g of the sample into the reaction flask, D. Add 50 ml of 0.1 mol/l hydrochloric acid, insert several boiling chips and connect the flask to the reflux condenser, F, using syrupy 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/min. Maintain the flow of nitrogen through the apparatus at 90 to 100 ml/min. Raise the heating mantle, E, to the flask, heat the sample to boiling and boil gently for 2 min. Turn the heat off, lower the mantle, E, and allow to cool for about 10 min. Connect the empty gas washing bottle assembly, J, and sweep the system with nitrogen at a rate of 90 to 100 ml/min for 5 min. Reduce the nitrogen flow to 60 to 65 ml/min, add 10 drops of 1-butanol, pipette 25.0 ml of 0.25 mol/l sodium hydroxide solution and add 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 3 hours of boiling, increase the nitrogen flow to 90 to 100 ml/min, discontinue the heating and lower the mantle. Allow to cool for 10 min. 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 magnetic stirring bar. Insert a tight stopper and stir gently for 1 min. Allow to stand for at least 5 min. Add three drops of phenolphthalein TS and titrate with 0.1 mol/l hydrochloric acid. Perform a blank determination.

Calculate the percentage of carbon dioxide from;

$$\begin{aligned} \text{Carbon dioxide (\%)} &= \frac{22 [(C_{\text{NaOH}} \times V_{\text{NaOH}} - C_{\text{HCl}} \times V_{\text{sample}}) - (C_{\text{NaOH}} \times V_{\text{NaOH}} - C_{\text{HCl}} \times V_{\text{blank}})]}{1000 W (1 - 0.01 \text{ LD})} \times 100 \\ &= \frac{22 C_{\text{HCl}} (V_{\text{blank}} - V_{\text{sample}})}{1000 W (1 - 0.01 \text{ LD})} \times 100 \end{aligned}$$

Where

$C_{\text{NaOH}}$  is the concentration of sodium hydroxide solution, mol/l

$V_{\text{NaOH}}$  is the volume of sodium hydroxide solution pipetted, ml

$C_{\text{HCl}}$  is the concentration of hydrochloric acid, mol/l

$V_{\text{sample}}$  is the volume of hydrochloric acid used for the titration of the sample, ml

$V_{\text{blank}}$  is the volume of hydrochloric acid used for the titration of the blank, ml

LD is the loss on drying obtained, %

W is the weight of the sample, g

Each ml of 1 mol/l sodium hydroxide is equivalent to 22 mg of carbon dioxide.