12. Iodometric determination of glucose

Glucose; C₆H₁₂O₆, also known as D-glucose, dextrose, or grape sugar) is a simple monosaccharide found in



plants. It is one of the three dietary monosaccharides, along with fructose and galactose, that are absorbed directly into the bloodstream during digestion. An important carbohydrate in biology, cells use it as the primary source of energy and a metabolic intermediate. Glucose is one of the main products of photosynthesis and fuels for cellular respiration.

Glucose solution can be used as a therapy in carbohydrate and fluid loss and also as a carrier solution for compatible drugs. Typical infusion solution is a mixture of physiological salt (0.9% w/v solution of sodium chloride in distilled water) with 5% glucose solution in volume ratio 2:1 (thus often denoted as "2:1").

Glucose content in blood is very important factor. Its content should be between 60 and 99 mg/L (3,4-5,5 mmol/L) in the morning, ca. 8 hours after last meal. A persistently high level is referred to as hyperglycemia; low levels are referred to as hypoglycemia.

In medicine, two major methods have been used to measure glucose. The first, still in use in some places, is a chemical method exploiting the nonspecific reducing property of glucose in a reaction with an indicator substance that changes color when reduced. Since other blood compounds also have reducing properties (e.g., urea, which can be abnormally high in uremic patients), this technique can produce erroneous readings in some situations (5 to 15 mg/dL has been reported). The more recent technique, using enzymes specific to glucose, is less susceptible to this kind of error. The two most common employed enzymes are glucose oxidase and hexokinase.

However, in our experiment, the sample will not contain reducing components different than glucose and the chemical method is sufficient. The good choice is iodometry.

Iodometry is a method of volumetric chemical analysis, a redox titration where the appearance or disappearance of elementary iodine indicates the end point. Iodine, I_2 , is almost insoluble in pure water, but addition of KI leads to formation of I_3^- ions ($I_2+I^- \rightarrow I_3^-$), and increased solubility of iodine.

Sometimes the solution of iodine in water+KI solvent is applied as titrant, where concentration of I_2 is known (direct titration). This is used if iodine is able to oxidize the analyzed sample, for example:

$$S^{2-} + 4I_2 + 4H_2O \rightarrow SO_4^{2-} + 8I^{-} + 8H^{+}$$

However, indirect or reverse titration is more common. The example is determination of copper. It is based on the oxidation of iodides to iodine by copper(II) ions, which get reduced to Cu^+ (see Task 13). The equivalent amount of elemental iodine is formed, which is next titrated using solution of thiosulfate:

 $2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^{-}$

lodometry is widely applied in analyzing organic reducing compounds, for example sugars which contain aldehyde groups –CHO, which can be easily oxidized to the carboxylic one –COOH. This is also the case of glucose. The reaction is quantitative in basic environment, because the carboxylic group is acidic and forms salts with bases.



Note the amount of NaOH needed to neutralize the gluconic acid formed. This base should be added, however, in excess, to ensure basic reaction.

Procedure

- 1. Dilute your sample in the volume flask to the mark. Shake few minutes.
- 2. Measure one pipette of the solution to Erlenmeyer flask with ground joint.
- 3. Add 20 mL of 0.1 M I_2 solution and 45 mL of 0.1 M NaOH solution.¹ Close the flask and leave it in dark place for 15 min.
- 4. Add 6 mL of 1 M HCl.
- Titrate the solution using 0.05 M sodium thiosulfate. When the liquid becomes light-yellow, add 1-2 mL of starch solution (the liquid becomes dark again) and titrate to colorless content.
- 6. Repeat points 2-5, preferably twice.

Report

- Calculate the mass of glucose in the initial sample, independent for the analyses made.
- Calculate the arithmetic average of the results obtained as well as the relative standard deviation (as the percent value of the mean).

Sources:

W.F. Goebel, "On the oxidation of glucose in alkaline solutions of iodine", J. Biol. Chem. 72 (1927) 801-807² Wikipedia

textbooks

¹ It is important to have pH during reaction (basic), but also proper pH during titration (acidic, but not too much). Thus, the volumes given above are calculated for exact concentrations. Check your data before performing the analysis and correct the volumes in the Procedure, if necessary.

² ON THE OXIDATION OF GLUCOSE IN ALKALINE SOLUTIONS OF IODINE, WALTHER F. GOEBEL: "To 10 cc. of a 0.9 per cent glucose solution were added 20 cc. of 0.1 N iodine-potassium iodide solution and to this were added 2 equivalents of 0.1 N sodium hydroxide (40 cc. + 5 cc. to neutralize the gluconic acid formed). When the alkali was added over an interval, it was run from a burette at constant rate. When added "immediately" the required amount was quickly poured from a cylinder into the well stirred glucose-iodine solution. The whole operation required about 3 seconds. After standing a total of 15 minutes, when the reaction had come to completion, the mixture was acidified with 6 cc. of 1.0 N hydrochloric acid and was titrated with 0.05 N sodium thiosulfate, using starch as an indicator. The method is accurate to about 0.15 per cent. The temperature variation was not greater than 0.25°. The sodium hydroxide used in these and subsequent experiments was exactly 0.1 N and was prepared from carbonate-free sodium hydroxide from sodium. The iodine-potassium iodide solution contained 25.0 gm. of the potassium iodide per liter."