

Polarimetry (Item No.: P2250200)

Curricular Relevance



Difficulty



Difficult

Preparation Time



10 Minutes

Execution Time



20 Minutes

Recommended Group Size



2 Students

Additional Requirements:

Experiment Variations:

Keywords:

Half-shade principle, optical rotatory power, optical activity, saccharimetry, specific rotation, reaction rate, Weber-Fechner Law.

Introduction

Overview

The rotation of the plane of polarisation through a sugar solution measured with a half-shade penumbra polarimeter and the reaction rate constant for the inversion of cane sugar determined.



Fig. 1: Experimental set-up for measuring the rotation of sugar solutions.

Equipment

Position No.	Material	Order No.	Quantity
1	Half-shade polarimeter, 230 V AC	35906-93	1
2	Immersion thermostat Alpha A, 230 V	08493-93	1
3	Balance OHAUS LG 311, 4 beams, 0...311 g	44007-31	1
4	Bath for thermostat, makrolon	08487-02	1
5	External circulation set f. thermostat Alpha A	08493-02	1
6	Stopwatch, digital, 1/100 s	03071-01	1
7	Hydrochloric acid 37 %, 1000 ml	30214-70	1
8	Water, distilled 5 l	31246-81	1
9	D (+)-Sucrose 100 g	30210-10	1
10	Crucible tongs, 200mm, stainl. steel	33600-00	1
11	Pipette with rubber bulb, long	64821-00	1
12	D(+)-Lactose, powder 100 g	31577-10	1
13	Graduated cylinder 100 ml, PP transparent	36629-01	2
14	Graduated vessel, 1 l, with handle	36640-00	1
15	Spoon, w. spatula end, 18 cm, plastic	38833-00	1
16	Beaker, 250 ml, low form, plastic	36013-01	2
17	Funnel, plastic, dia 100 mm	36891-00	1
18	Tubing connector, ID 6-10mm	47516-01	2
19	Glass rod, boro 3.3, l=300mm, d=8mm	40485-06	1

Tasks

1. To determine the specific rotation of cane sugar (sucrose) and lactose by measuring the rotation of various solutions of known concentration.
2. To determine the reaction rate constant when cane sugar is transformed into invert sugar.

Set-up and procedure

To measure optical activity we use a polarimeter in which light of wave-length 589 nm (sodium-D line) is first plane-polarised by a polariser and then studied by a second polariser to find its new plane of polarisation after passing through the sample substance.

In order to increase the accuracy of measurement, in one half of the field of view there is a Laurent's quartz plate which rotates the plane of polarisation through a further small angle. The analyser is now set to equal brightness on the two sides of the field of view: this gives a sharp setting and is easy to reproduce (Weber-Fechner Law).

1. First prepare a cane sugar solution of known concentration c . For example, a solution of concentration $c_0 = 0.24 \text{ g/cm}^3$ is made by dissolving 12 g of cane sugar and making up to a total volume of 50 cm³ with distilled water in the measuring cylinder.

The rotation of this solution is determined in the half-shade polarimeter by adjusting the two halves of the field to identical brightness. In all, four concentrations

$$c_0; c_0/2; c_0/4; c_0/8$$

are made by making up the solution remaining in the measuring cylinder each time with exactly the same volume of distilled water (it may be necessary to pour some off first).

The temperature of the solutions should be the same in all cases. The containing vessels are tempered in the water bath for approx. 5 minutes prior to each measurement.

The same procedure is used for measuring the rotation of lactose (concentration $c_0 = 0.30 \text{ g/cm}^3$, for example).

2. 12 g cane sugar are placed in the measuring cylinder and made up to 50 ml with distilled water ($c_0 = 0.24 \text{ g/cm}^3$).

When the sugar has dissolved completely (stir it), the measuring vessel is filled and heated in the water bath for approx. 5 minutes at test temperature T (approx. 30°C). This temperature must be maintained throughout the experiment. The measurement of the angle of rotation gives a value α_0 .

The solution remaining in the measuring cylinder is now made up to exactly double the volume with 2n HCl, stirred, placed in the measuring vessel and brought to the test temperature in the water bath. The stop clock is started at this time. The vessel is removed from the water bath after approx. 5 minutes and the angle of rotation measured. The series of measurements is discontinued after 40–50 minutes and the final angle of rotation determined. For this purpose, the solution is heated to 70°C in the water bath for approx. 5 minutes: this completes the inversion process very quickly. The vessel is then brought back to the test temperature and the final angle of rotation β determined.

Theory and evaluation

Optical activity is the ability of many substances to rotate the plane of vibration of a ray of plane-polarised light passing through them. It is caused by the two components of a plane-polarised ray of light travelling at different speeds through the asymmetric medium and, in so doing, undergoing a phase shift with respect to one another, this phase shift being indicated as a rotation of the plane of polarisation.

The specific rotation (rotatory power) of solutions of optically active substances is defined as the angle through which the plane of polarisation of a ray of sodium-D light (wave-length $\lambda = 589 \text{ nm}$) would be rotated by a column of solution 100 mm in length, containing 1 g of substance per cm^3 at 20°C . It is expressed by the symbol

$$[\alpha]_D^{20^\circ}$$

If we assume that the rotation is proportional to the concentration c , then the specific rotation can be determined from the rotation α at a known concentration:

$$[\alpha]_D^{20^\circ} = \frac{\alpha}{c} \text{ (related to a column length of 100 mm)}$$

If the measurement temperature starts at 20°C , the value obtained must be corrected in accordance with

$$[\alpha]_D^{20^\circ} = [\alpha]_D^\vartheta - 0.072(20^\circ - \vartheta)$$

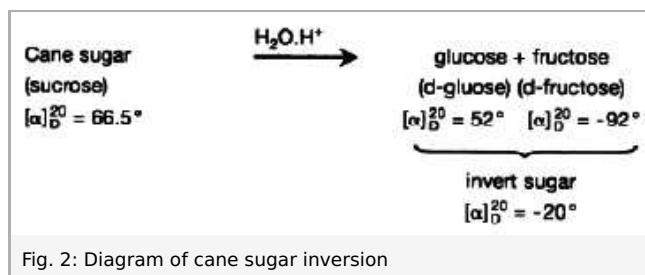
for lactose, or

$$[\alpha]_D^{20^\circ} = \frac{[\alpha]^\vartheta}{1 - 0.00037(\vartheta - 20^\circ)}$$

for sucrose.

The mean value found for the specific rotation of the lactose solution was $[\alpha]_D^{20^\circ} = 53.0^\circ$, with a standard deviation $s[\alpha]_D^{20^\circ} = 0.3^\circ$

The specific rotation of the cane sugar solution was $[\alpha]_D^{20^\circ} = 65.3^\circ$, with a standard deviation $s[\alpha]_D^{20^\circ} = 1.3^\circ$. In the presence of H^+ ions, which have a catalytic effect in this case, an aqueous solution of cane sugar breaks down into optically active glucose and fructose.



This is a first order reaction, i. e. the reaction rate is proportional to the concentration of the cane sugar.

Since the reaction rate is defined as the decrease in concentration per unit of time, the differential equation

$$-\frac{dc}{dt} = k \cdot c$$

can be used, from which

$$\ln \frac{c_0}{c(t)} = -kt \quad (1)$$

is obtained after integration, and where k is the reaction rate constant, c_0 the initial concentration at time $t = 0$ and $c(t)$ the concentration at time.

If $\alpha(t)$ is the rotation at time t , α_0 the rotation of the pure cane sugar solution and the rotation β of the solution which has been fully inverted, then

$$\frac{c_0}{c(t)} = \frac{\alpha_0 + |\beta|}{\alpha(t) + |\beta|}$$

With (1) we obtain

$$k = -\frac{1}{t} \ln \frac{\alpha_0 + |\beta|}{\alpha(t) + |\beta|}$$

We can now calculate the reaction rate constant k for each pair of values for t and $\alpha(t)$, and obtain the average value k , or determine it using the expression

$$t = A + B \cdot \ln \frac{\alpha_0 + |\beta|}{\alpha(t) + |\beta|}$$

from the slope β of the regression line, applying the relation

$$k = \frac{1}{\beta}$$

Evaluating the measured values shown in Fig. 3 ($c_0 = 0.24 \text{ g/cm}^3$, $\vartheta = 29.5 \text{ }^\circ\text{C}$) gives

$$k = 23.7 \pm 0.2 \cdot 10^{-3} \text{ min}^{-1}$$

Note

The detailed instruction manual of the polarimeter gives examples for several other materials which can be analyzed and will give reproducible results.

