1. Rationale

The pol % extract is calculated from the Brix % extract as determined in Method 1.3 and the saccharimeter reading which is determined as described below.

2. Principle

After digestion a portion of the sample is reacted with lead sub-acetate powder for clarification and used to determine the pol of a solution. When calculating the final pol of the sample the Brix of the solution is always needed.

3. Apparatus

3.1 Saccharimeter and 200 mm pol tube
3.2 Stoppered bottle (225 cm$^3$)
3.3 Stemless funnel (100 mm φ)
3.4 Tall form beaker (250 cm$^3$)
3.5 Watch glass (100 mm φ)
3.6 Filter paper, Whatman 91 or equivalent (185 mm φ)

4. Reagents

4.1 Lead sub-acetate powder

Lead sub-acetate trihydrate \([\text{Pb(OAc)}_2 \cdot 3\text{H}_2\text{O}]\), also called basic lead acetate, is poisonous and will accumulate in the human body. Direct contact through the skin, inhalation (powder dust) or swallowing must be avoided. Wear a dust mask, gloves and safety glasses during use.

The lead sub-acetate should conform to the following specifications:

- Basic lead (as PbO) > 33%
- Moisture at 105°C < 1.5%
- Insoluble in dilute acetic acid < 0.02%
- Insoluble in water < 1.0%
- Chloride (Cl) < 0.003%
- Nitrate and nitrite (NO₃⁻) < 0.003%
- Copper (Cu) < 0.002%
- Substances not precipitated by H₂S (as sulphates) < 0.30%
- Iron < 0.002%

Refer to Method 11.2 for the determination of the total and basic lead content of lead sub-acetate.
5. Procedure

Shake the contents of the 450 cm³ bottle prepared in Method 1.1, and transfer approximately 150 cm³ to a 225 cm³ stoppered bottle, taking the precaution of first rinsing the smaller bottle and cap with some of the extract and discarding the rinsings.

To the 150 cm³ extract add sufficient lead sub-acetate for clarification. The amount added must be the minimum required for clarification as over-leading will introduce errors. 1.0 g lead sub-acetate per 150 cm³ sample is usually sufficient for cane extract whilst considerably less is necessary for bagasse extract (0.3 g). Put the lid back on the bottle.

Vigorously shake the leaded extract and then allow it to stand for about 30 seconds to permit flocculation of the precipitate.

Place a fluted filter paper in the stemless filter funnel.

Place the filter funnel in the mouth of the beaker so that the funnel is supported by the rim of the beaker.

Pour the leaded extract, in one operation, into the filter funnel, taking care not to overflow the upper edge of the filter paper. Cover with a watch glass.

Rinse the beaker with the first 25 cm³ of filtrate and discard this portion of filtrate. Do not return this filtrate to the filter funnel. If the subsequent filtrate is still not clear, use a further portion for rinsing.

Before polarizing, visually check the clarity of the filtrate. If there is any sign of haziness, prepare a new sample using extract from the 450 cm³ bottle and pay particular attention to the amount of lead used.

Swirl the filtrate in the beaker and then rinse the saccharimeter tube three times with a portion of filtrate.

Fill the tube with the remaining filtrate and take the saccharimeter reading.

If a flow-through pol tube is used, a minimum of 40 cm³ of filtrate must be poured into the funnel in one operation, while holding the top of the beaker against the side of the funnel to ensure that no air bubbles enter the tube.

6. Calculations

The pol % extract is obtained from the saccharimeter reading and the Brix of the extract. The basic formula for the calculation is:

\[
\text{pol} \% \text{ extract} = \frac{\text{normal mass} \times \text{saccharimeter reading}}{\text{mass (g) of 100 cm}^3 \text{ of solution}}
\]

where (i) the normal mass = 26.000g when the saccharimeter is calibrated according to the International Sugar Scale.

(ii) the mass in g of 100 cm³ of solution is equal to 99.718 × apparent specific gravity at 20°C/20°C of the solution. This can be calculated from the Brix.

In practice the pol is found from Schmitz formula:

\[
\text{pol} = \frac{\text{polarimeter reading}}{0.0000576 \times \text{Brix}^2 + 0.014752 \times \text{Brix} + 3.83545}
\]
7. Precision

The tolerance associated with the pol analysis is ± 0.05°Z

8. Example

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brix of the extract</td>
<td>6.13°Bx</td>
</tr>
<tr>
<td>Saccharimeter reading</td>
<td>20.55°Z</td>
</tr>
<tr>
<td>Pol of the extract</td>
<td>5.23°Z</td>
</tr>
</tbody>
</table>

9. References