






MANUAL PROSEDUR
PEMERIKSAAN IODIUM DALAM GARAM
LABORATORIUM GAKI
FAKULTAS KEDOKTERAN
UNIVERSITAS DIPONEGORO

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| Kode Dokumen | : | SPMI-UNDIP/MP/04.06/08 |
| Revisi ke | : | - |
| Tanggal | : | 15 Januari 2018 |
| Disiapkan oleh | : | Ketua Laboratorium GAKI |
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| FAKULTAS KEDOKTERAN UNIVERSITAS DIPONEGORO | MANUAL PROSEDUR Pemeriksaan Iodium dalam Garam SPMI-UNDIP/MP/04.06/08 | Disetujui oleh  Dekan |
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|  | | MANUAL PROSEDUR Pemeriksaan Iodium Dalam Garam | Disetujui oleh:  |
| Revisi ke - | Tanggal 15-01-2018 | SPMI-UNDIP/MP/04.06/08 | |

Tujuan

1. Melakukan pemeriksaan iodium secara kuantitatif dengan metode titrasi iodometri.
2. Menentukan kadar iodium pada garam dapur beberapa merek.

Dasar Teori

Penetapan kadar iodium suatu bahan pangan diperlukan untuk mengetahui kandungan iodium yang terdapat dalam bahan pangan. Dengan mengetahui kandungan iodium dalam bahan pangan tersebut nantinya akan digunakan untuk mengukur tingkat kecukupan iodium sehari dari konsumsi bahan pangan tersebut. Garam beriodium merupakan solusi bagi kebutuhan iodium untuk masyarakat. Perlu dilakukan kontrol apakah produk garam beriodium sudah memenuhi standar minimal kadar iodium, yaitu 30 ppm.

Definisi

1. Garam adalah suatu zat berbentuk padat, kristal, dan berwarna putih yang merupakan hasil dari laut.
2. Iodium adalah suatu elemen non metal, diperlukan manusia untuk sintesis hormon tiroid, sebagai unsur paling penting dalam proses tumbuh kembang manusia.
3. Iodometri (disebut pula analisis iodometrik) adalah titrasi redoks yang melibatkan titrasi iodin yang diproduksi dalam reaksi dengan larutan standar natrium tiosulfat.

RUANG LINGKUP

Laboratorium GAKI

Referensi

Monitoring Universal Salt Iodization Programmes, Published by PAMM/MI/ICCIDD, 1995

Prosedur

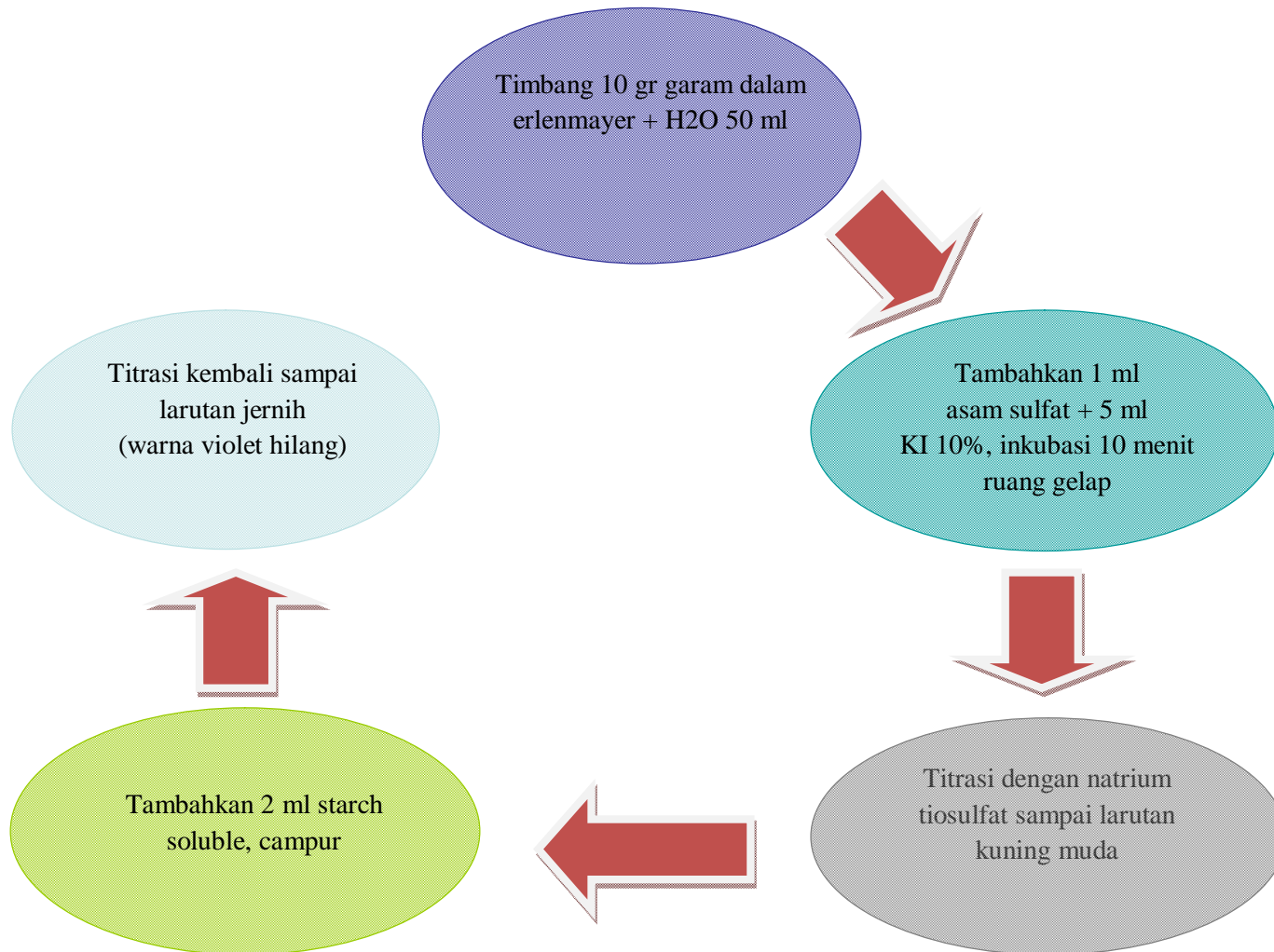
1. Ditimbang 10 gr garam dimasukkan dalam Erlenmeyer 250 ml
2. Ditambahkan 50 ml aqua
3. Ditambahkan 1 ml H₂SO₄ 2N
4. Ditambahkan 5 ml KI 10%, inkubasi 10 menit diruang gelap

5. Dititrasi dengan Natrium tiosulfat sampai larutan kuning muda
6. Ditambahkan starch soluble 2 ml, campur.
7. Dititrasi kembali sampai larutan jernih seperti semula

Lampiran

1. Manual kerja titrasi iodometri
2. Manual pembuatan reagensia
3. Table Iodine

**BAGAN ALIR MANUAL PROSEDUR
Pemeriksaan Iodium Dalam Garam**



Lampiran 1.

Figure 11-1: Weighing salt sample



Procedural Steps

Step 1. Weigh 10g of the salt sample into a 250mL Erlenmeyer flask with a stopper.

Step 2. Add approximately 30mL water, swirl to dissolve salt sample.

Step 3. Add water to make volume up to 50mL.

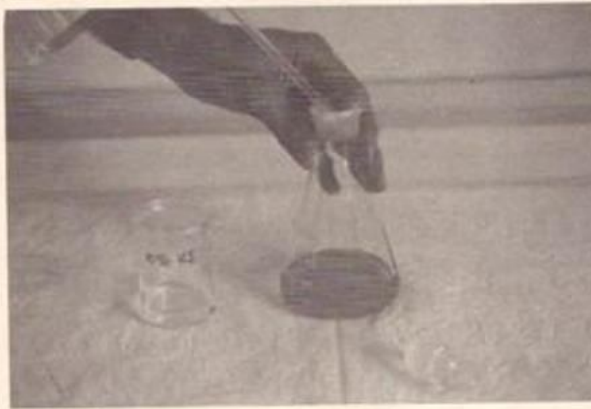
Step 4. Add 1mL 2N H_2SO_4 .

CAUTION - Do not pipette by mouth.

Step 5. Add 5mL 10% KI. The solution should turn yellow if iodine is present.

CAUTION - Do not pipette by mouth.

Figure 11-2: Addition of 10% potassium iodide solution



Step 6. Stopper the flask and put in the dark (cupboard or drawer) for 10 minutes.

Step 7. Rinse and fill burette with 0.005M $\text{Na}_2\text{S}_2\text{O}_3$ and adjust level to zero.

Figure 11-3: Filling the burette with sodium thio-sulfate solution



Step 8. Remove flask from drawer, and add some $\text{Na}_2\text{S}_2\text{O}_3$ from the titration burette until the solution turns pale yellow (Flask B shown in Figure 11.4).

Step 9. Add approximately 2mL of starch indicator solution (the solution should turn dark purple) and continue titrating until the solution becomes pink, and finally colourless. (Colour sequence of titration is shown in flasks C, D and E, figure 11.4)

Figure 11-4: This photo shows the various color changes that will be seen during the titration.

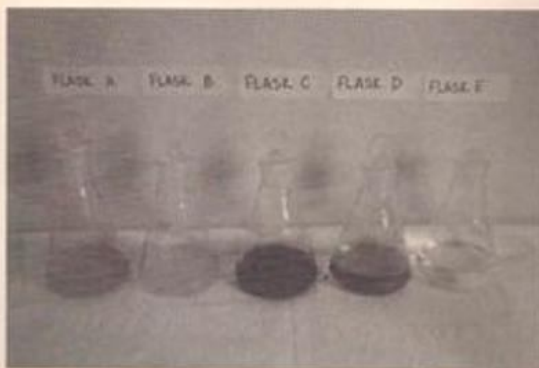
Flask A - after addition of KI (Step 5);

Flask B - just prior to addition of starch (Step 8);

Flask C - after starch has been added (Step 9);

Flask D - just prior to titration end-point (Step 9);

Flask E - titration end-point (step 9).



Step 10. Record the level of thiosulfate in the burette and convert to parts per million (ppm) using the conversion table in Appendix 11-3.

NOTE: Analysis time is approximately 20 minutes per sample.

Precautions

- The reaction mixture should be kept in the dark before titration because a side reaction can occur when the solution is exposed to light that causes iodide ions to be oxidized to iodine.
- Inaccurate results may occur if starch solution is used while still warm.
- If starch indicator is added too early, a strong iodine-starch complex is formed, which reacts slowly, and gives falsely elevated results.
- The reaction should be performed at mild room temperature ($<30^\circ\text{C}$), since the iodine is volatile, and the indicator solution loses sensitivity when exposed to high temperatures.

Lampiran 2

REAGENT PREPARATION

Water Requirements for Reagent Preparation

Water required for this method should be boiled, distilled water, which requires provision of a distillation unit. As a simpler alternative, regular tap water treated with a mixed bed deionizing resin can be used, thus avoiding the need for an expensive distillation unit. (See Appendix 11-2 for further details on preparation of this water.

- **0.005M Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$):** Dissolve 1.24g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in 1000mL water.

Store in a cool, dark place. This volume is sufficient for 100–200 samples, depending on the iodine content of samples. The solution is stable at least 1 month, if stored properly.

- **2N Sulfuric acid (H_2SO_4):** Slowly add 6mL concentrated H_2SO_4 to 90mL water. Make to 100mL with water. This volume is sufficient for 100 samples. The solution is stable indefinitely.

Note: Always add acid to water, **not** water to acid, to avoid excess heat formation and spitting of acid. Stir solution while adding acid.

- **10% Potassium iodide (KI):** Dissolve 100g KI in 1000mL water. Store in a cool, dark place. This volume is sufficient for 200 samples. Properly stored the solution is stable for six months.

- **Starch indicator solution:** Make 100mL of a saturated NaCl solution, by adding NaCl to approximately 80mL water in a beaker, with stirring and/or heating, until no further solid will dissolve. This solution is stable for at least one year. Weigh 1g soluble starch into a 100mL beaker, add 10mL water, heat to dissolve. Add saturated NaCl solution to the hot starch solution to make up to 100mL. Store in a cool, dark place. This volume is sufficient for 50 samples. The solution is stable for up to one month, and should be heated (not boiled) each day it is used to re-suspend any solids.

Lampiran 3

APPENDIX 11-3

CONVERSION TABLE : IODINE CONTENT IN PARTS PER MILLION

| BURETTE READING | PARTS PER MILLION (ppm) | BURETTE READING | PARTS PER MILLION (ppm) |
|--------------------|----------------------------|--------------------|----------------------------|
| 0.0 | 0.0 | 5.0 | 52.9 |
| 0.1 | 1.1 | 5.1 | 54.0 |
| 0.2 | 2.1 | 5.2 | 55.0 |
| 0.3 | 3.2 | 5.3 | 56.1 |
| 0.4 | 4.2 | 5.4 | 57.1 |
| 0.5 | 5.3 | 5.5 | 58.2 |
| 0.6 | 6.3 | 5.6 | 59.2 |
| 0.7 | 7.4 | 5.7 | 60.3 |
| 0.8 | 8.5 | 5.8 | 61.4 |
| 0.9 | 9.5 | 5.9 | 62.4 |
| 1.0 | 10.6 | 6.0 | 63.5 |
| 1.1 | 11.6 | 6.1 | 64.5 |
| 1.2 | 12.7 | 6.2 | 65.6 |
| 1.3 | 13.8 | 6.3 | 66.7 |
| 1.4 | 14.8 | 6.4 | 67.7 |
| 1.5 | 15.9 | 6.5 | 68.8 |
| 1.6 | 16.9 | 6.6 | 69.8 |
| 1.7 | 18.0 | 6.7 | 70.9 |
| 1.8 | 19.0 | 6.8 | 71.9 |
| 1.9 | 20.1 | 6.9 | 73.0 |
| 2.0 | 21.2 | 7.0 | 74.1 |
| 2.1 | 22.2 | 7.1 | 75.1 |
| 2.2 | 23.3 | 7.2 | 76.2 |
| 2.3 | 24.3 | 7.3 | 77.2 |
| 2.4 | 25.4 | 7.4 | 78.3 |
| 2.5 | 26.5 | 7.5 | 79.4 |
| 2.6 | 27.5 | 7.6 | 80.4 |
| 2.7 | 28.6 | 7.7 | 81.5 |
| 2.8 | 29.6 | 7.8 | 82.5 |
| 2.9 | 30.7 | 7.9 | 83.6 |
| 3.0 | 31.7 | 8.0 | 84.6 |
| 3.1 | 32.8 | 8.1 | 85.7 |
| 3.2 | 33.9 | 8.2 | 86.8 |
| 3.3 | 34.9 | 8.3 | 87.8 |
| 3.4 | 36.0 | 8.4 | 88.9 |
| 3.5 | 37.0 | 8.5 | 89.9 |
| 3.6 | 38.1 | 8.6 | 91.0 |
| 3.7 | 39.1 | 8.7 | 92.0 |
| 3.8 | 40.2 | 8.8 | 93.1 |
| 3.9 | 41.3 | 8.9 | 94.2 |
| 4.0 | 42.3 | 9.0 | 95.2 |
| 4.1 | 43.4 | 9.1 | 96.3 |
| 4.2 | 44.4 | 9.2 | 97.3 |
| 4.3 | 45.5 | 9.3 | 98.4 |
| 4.4 | 46.6 | 9.4 | 99.5 |
| 4.5 | 47.6 | 9.5 | 100.5 |
| 4.6 | 48.7 | 9.6 | 101.6 |
| 4.7 | 49.7 | 9.7 | 102.6 |
| 4.8 | 50.8 | 9.8 | 103.7 |
| 4.9 | 51.9 | 9.9 | 104.7 |

Titration Methods for Salt Iodine Analysis