

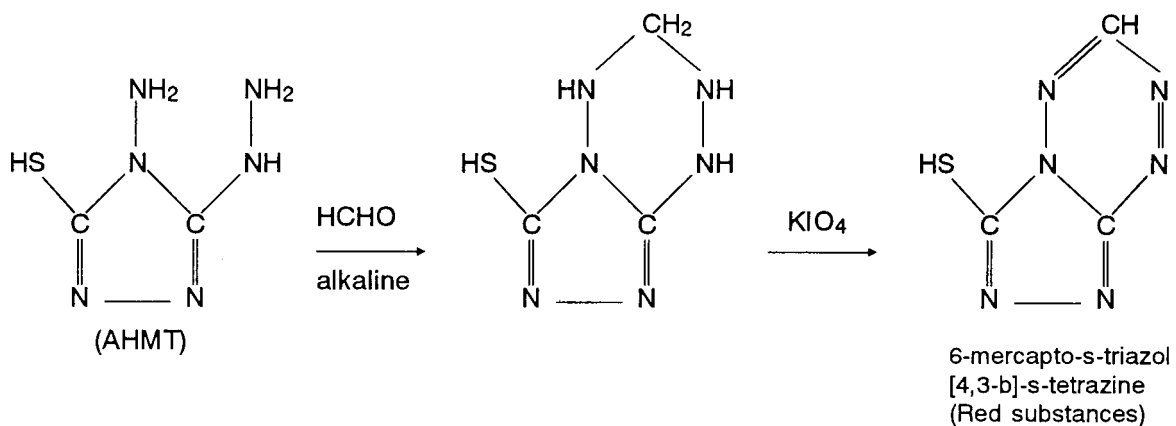
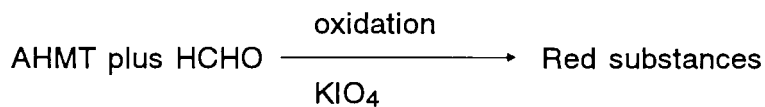
DETERMINATION OF FORMALDEHYDE BY 4-amino-3-hydrazino-5-mercapto-1,2,4-triazol (AHMT) METHOD (Colorimetric method)

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INTRODUCTION

In this method the formaldehyde undergoes oxidation when reacted with AHMT and KIO_4 to yield violet compounds. The intensity of the compounds is proportional to the concentration of formaldehyde present. This AHMT method is more sensitive than the chromotropic acid method and the acetylacetone method. AHMT is also suitable for determination of residual formaldehyde in bisulfite treated shrimps.

Principle of AHMT Method



APPARATUS

1. Distillation unit (Fig. 1)
2. Conical flasks, 250 ml
3. Volumetric flasks, 200 ml
4. Test tubes with glass stoppers, 20 ml
5. Pipettes, 2 ml, 5 ml, 10 ml
6. Spectrophotometer (550 nm)

REAGENTS

1. AHMT solution

Weigh 0.5 g of AHMT and dissolve in 100 ml of 0.5N HCl solution. Store in a cool, dark place.

2. Potassium periodate (KIO₄) solution

Weigh 0.75 g of KIO₄ and dissolve in 100 ml of 0.2N KOH solution using a water bath.

3. Formaldehyde(HCHO) standard solution

Accurately weigh about 1 g of formalin (35%, stock solution) into weighing bottle with 5 ml distilled water and make up to 100 ml with distilled water in a volumetric flask. Pipette 10 ml of this solution into a conical flask and accurately add 50 ml of 0.1N iodine solution. Add 20 ml of 1N KOH solution then stand for 15 min at room temperature. Add 15 ml of 10% H₂SO₄ solution. Titrate against 0.1N sodium thiosulphate (Na₂S₂O₃) solution, using 1 ml of starch solution as indicator. Blank should be done using 10 ml distilled water instead of the formaldehyde solution.

Determination of the actual concentration of stock formaldehyde

Formaldehyde (HCHO) content, C(%) = $1.501 \times (V_B - V)F/W$

- Where
- | | | |
|----------------|---|--|
| V | : | Titration volume (ml) of 0.1N Na ₂ S ₂ O ₃ solution (Main test) |
| V _B | : | Titration volume (ml) of 0.1N Na ₂ S ₂ O ₃ solution for blank using distilled water instead of formaldehyde |
| F | : | Factor of 0.1N Na ₂ S ₂ O ₃ solution |
| W | : | Sample weight of formalin (stock solution) used |

Preparation of standard formaldehyde solutions

Accurately weigh 200/C (g) of formalin (35%, stock solution), dissolve with distilled water and make up to 100 ml. This contains 20,000 mg formaldehyde per litre. Make a 10 times dilution (10 → 100) of this and repeat 4 times to obtain a formaldehyde solution where 1 ml contains 2 ug HCHO.

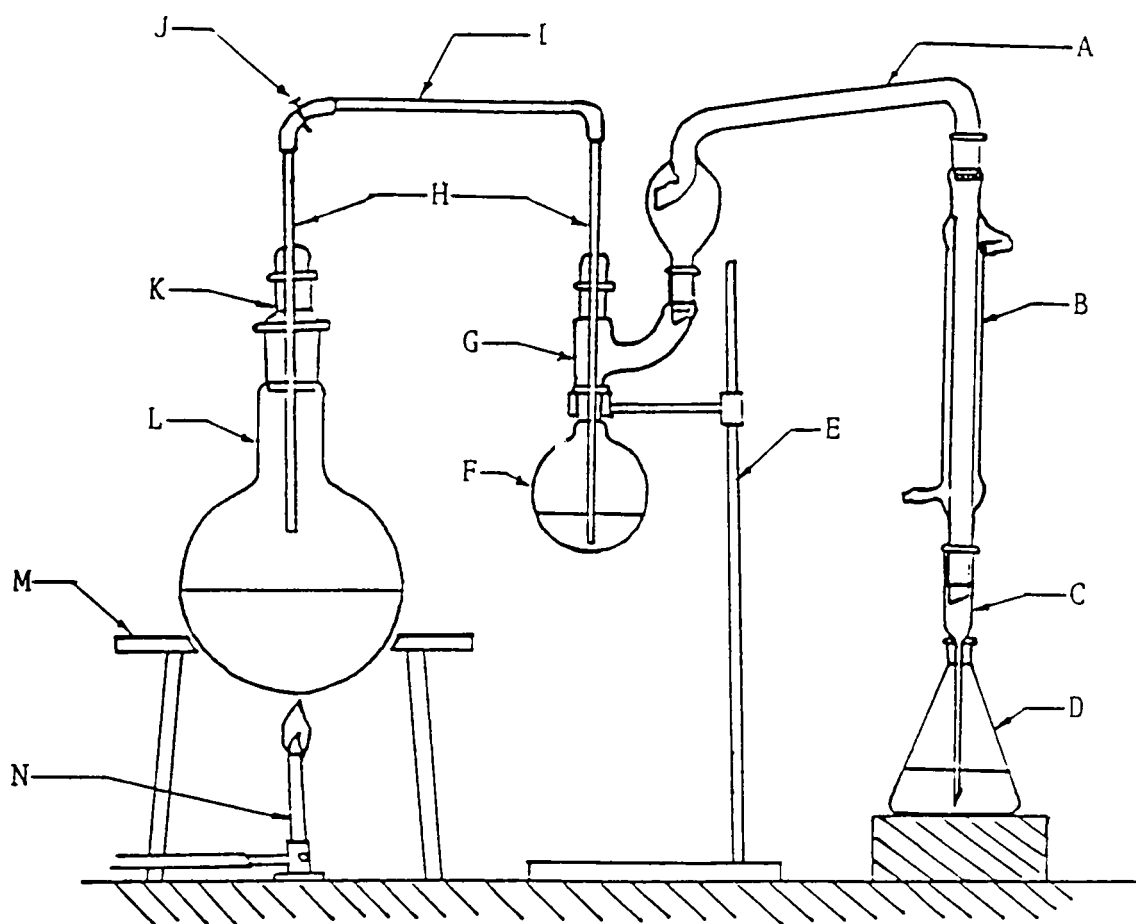
PROCEDURE

Sample preparation (Extraction by distillation)

1. Accurately weigh 1 - 10 g of solid sample or pipette 10 ml of liquid sample (100 - 500 ug as HCHO) into flask.
2. Add 5 - 10 ml of distilled water and 1 ml of 20% H₃PO₃ solution.
3. Add 5 - 10 ml of distilled water into receiver and make sure the edge of the condenser is below the distilled water.
4. Start the steam distillation (distillation speed : 6 - 10 ml/ min).
5. When about 200 ml of distillate is collected in the receiver, stop the steam distillation.
6. Make up to 200 ml with distilled water in a volumetric flask.

Analytical Procedure

1. Pipette 2.0 ml of sample solution(distillate) into test tube with stopper.
2. Add 2.0 ml of 5N KOH solution followed by 2.0 ml of AHMT.
3. Mix gently and stand for 20 mins at room temperature.
4. Add 2.0 ml of KIO₄ solution and mix gently till the bubbles fade away.
5. Measure its absorption at the wavelength of 550 nm. HCHO concentration (ug/ml) is calculated according to HCHO standard curve (calibration curve).



- | | | | | | |
|---|---|------------------------------|---|---|--------------------------|
| A | : | Splash head | H | : | Steam inlet tube |
| B | : | Liebig condenser | I | : | Glass tubing |
| C | : | Straight delivery adapters | J | : | Clip |
| D | : | Conical flask | K | : | Reduction adaptors |
| E | : | Retort stand | L | : | Round bottomed flask, 2L |
| F | : | Round bottomed flask, 250 ml | M | : | Tripod stand |
| G | : | Two neck multiple adaptor | N | : | Bunsen burner |

Fig. 1. Steam distillation apparatus

Preparation of standard curve

1. Pipette 0.5, 1.0, 1.5 and 2.0 ml of HCHO standard solution each into test tubes with stoppers. Prepare one empty test-tube with stopper.
2. Make up to 2.0 ml with distilled water.
3. Repeat as in Analytical Procedure.

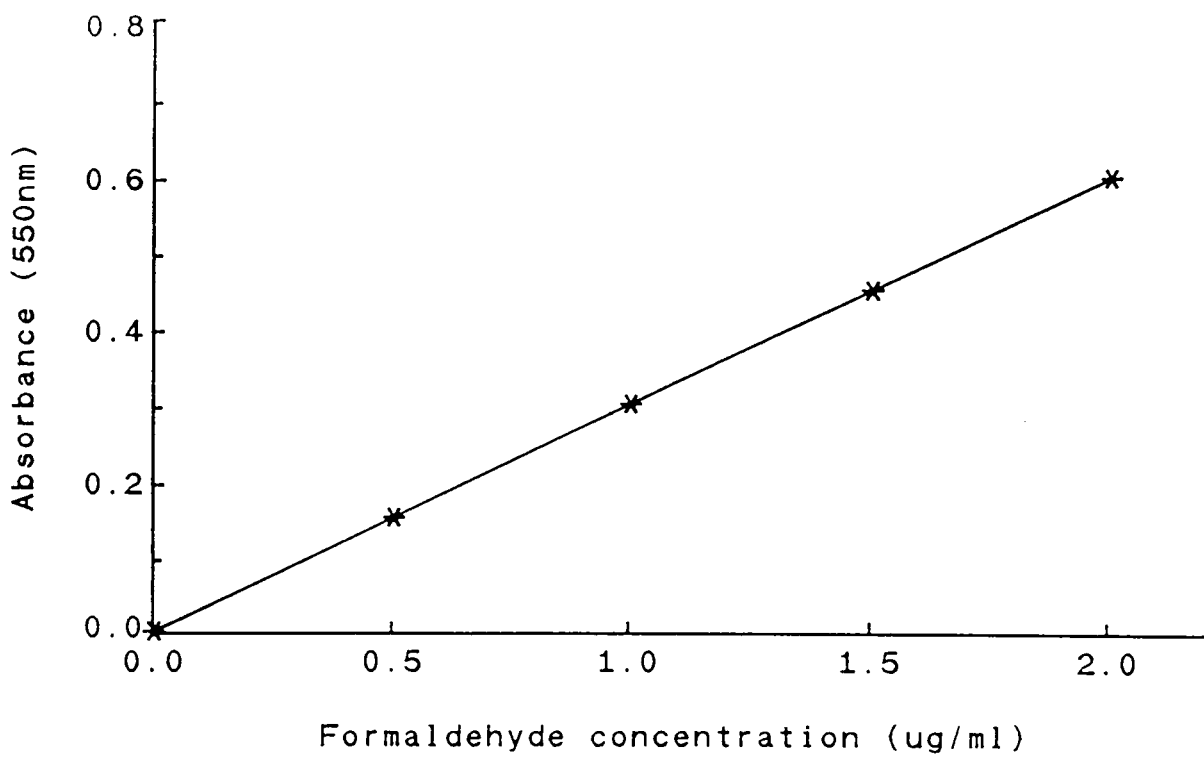


Fig 2. Standard curve for formaldehyde (AHMT Method)

CALCULATION

$$\begin{aligned}\text{Formaldehyde (ug/g)} &= \frac{A}{\text{Vol. of filtrate used}} \times \frac{\text{Total vol. of filtrate}}{\text{Wt. of sample}} \\ &= \frac{A}{2} \times \frac{200}{10} \\ &= A \times 10\end{aligned}$$

where A = Reading from standard curve (ug)

REMARKS

Detection limit of this method is 0.1 ppm.

REFERENCE

Standard Methods of Analysis for Hygiene Chemists - With Commentary - authorized by the Pharmaceutical Society of Japan, Kimbara Publishing Co., Ltd. p. 110. (1990).

Mimura, H., Kaneko, M., Nishiyama, N., Fukui, S., and Kanno, S. (1976). Determination of Formaldehyde by the 4-Amino-3-hydrazino-5-mercapto-1,2,4-triazol Method (AHMT Method). The Journal of Hygiene Chemistry. 22(1), p. 39-41.