
**Phenolic, amino and condensation
resins — Determination of free-
formaldehyde content**

*Résines phénoliques, aminiques et de condensation — Dosage du
formaldéhyde libre*



Reference number
ISO 11402:2004(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11402 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 11402:1993), as well as ISO 9020:1994 and ISO 9397:1995. The three standards have been combined into one, the sulfite procedure being taken from ISO 9020 and the hydroxylamine hydrochloride procedure from ISO 9397.

Phenolic, amino and condensation resins — Determination of free-formaldehyde content

1 Scope

This International Standard specifies three methods for determining the free-formaldehyde content in the following:

- a) **Phenolic resins**, by potentiometric titration in aqueous or organic solution (hydroxylamine hydrochloride procedure). The method is applicable to resins with free-formaldehyde contents up to and including 15 % by mass. For free-formaldehyde contents between 15 % by mass and 30 % by mass, it may be necessary to adjust the concentrations of the standard volumetric solutions used accordingly.
- b) **Amino resins and furan resins** (sulfite procedure). The method is applicable to resins resulting from the polycondensation of urea and melamine with formaldehyde and to furan resins resulting from the polycondensation of furfuryl alcohol with formaldehyde without further modification.
- c) **Condensation resins** (KCN procedure), including urea resins, furan resins, melamine resins and phenolic resins, as well as combinations and modifications of these resins.

The purpose of this International Standard is to establish recognized and useful determination procedures (for instance in order to check whether official regulations on the handling of hazardous workplace materials are observed). As far as possible, all formaldehyde-containing resins are to be included in this connection.

The free-formaldehyde content determined in formaldehyde condensation resins using this International Standard represents the actual content at the time of the determination. The value bears no quantitative relationship to the free-formaldehyde content during or after processing.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 free formaldehyde
formaldehyde that is present in unbound form as formaldehyde, formaldehyde dihydrate (dihydroxymethylene) or polyoxymethylene in a condensation resin

4 Test procedures

4.1 General

The choice of test procedure for the determination of free formaldehyde in condensation resins is made in accordance with Table 1.

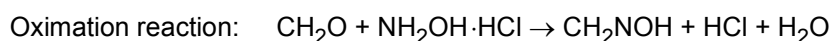
Table 1 — Selection of procedure

Procedure	Suitable for testing of
Hydroxylamine hydrochloride procedure	Phenolic resins, furan resins ^a (unmodified with urea or melamine resin)
Sulfite procedure	Urea resins, melamine resins, furan resins ^a , urea-melamine resins, furan-urea resins
KCN procedure ^b	Melamine-phenolic resins, urea-phenolic resins, urea-melamine-phenolic resins
^a See Annex A, Clause A.2.	
^b See Annex A, Clause A.3.	

4.2 Hydroxylamine hydrochloride procedure

4.2.1 Principle

The formaldehyde is converted to the oxime with hydroxylamine hydrochloride. The hydrochloric acid formed during this reaction is determined by potentiometric back-titration, using sodium hydroxide solution.



4.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

4.2.2.1 Hydroxylamine hydrochloride, 10 % by mass solution, the pH of which has been adjusted to 3,5 by the addition of sodium hydroxide solution.

4.2.2.2 Sodium hydroxide, standard volumetric solutions, $c(\text{NaOH}) = 1 \text{ mol/l}$ and $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

4.2.2.3 Hydrochloric acid, standard volumetric solutions, $c(\text{HCl}) = 1 \text{ mol/l}$ and $c(\text{HCl}) = 0,1 \text{ mol/l}$.

4.2.2.4 Methanol, free of aldehydes and ketones.

4.2.2.5 Propan-2-ol, free of aldehydes and ketones.

4.2.3 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

4.2.3.1 Balance, accurate to 0,1 mg.

4.2.3.2 pH-meter, sensitive to 0,1 pH-units, equipped with a glass indicating electrode and a standard calomel reference electrode.

4.2.3.3 Magnetic stirrer.

4.2.3.4 Graduated burettes, of capacity 10 ml and 25 ml, the latter being for use if the formaldehyde content is likely to be greater than 5 % by mass.

4.2.4 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

4.2.5 Procedure

4.2.5.1 Test temperature

Carry out the test at $(23 \pm 1) ^\circ\text{C}$.

4.2.5.2 Test portion

Weigh, to the nearest 0,1 mg, into a 250 ml beaker, a test portion of mass from 1 g to 5 g, depending on the assumed formaldehyde content (see Table 2).

Table 2 — Mass of test portion

Assumed formaldehyde content % by mass	Mass of test portion g
< 2	$5,0 \pm 0,2$
2 to 4	$3,0 \pm 0,2$
> 4	1 to 2

4.2.5.3 Determination

Add 50 ml of methanol (4.2.2.4), or 50 ml of a mixture of 3 volumes of propan-2-ol (4.2.2.5) and 1 volume of water, to the contents of the beaker, switch on the magnetic stirrer (4.2.3.3) and stir until the resin has dissolved and the temperature has stabilized at $(23 \pm 1) ^\circ\text{C}$.

Introduce the electrodes of the pH-meter (4.2.3.2) into the solution and, using the 0,1 mol/l solution of hydrochloric acid (for neutralized resins) or the 1 mol/l solution (for highly alkaline resins) (see 4.2.2.3), adjust the pH to 3,5.

Pipette into the solution approximately 25 ml of hydroxylamine hydrochloride solution (4.2.2.1) at $(23 \pm 1) ^\circ\text{C}$.

Stir for (10 ± 1) min.

Titrate rapidly, using the 1 mol/l solution of sodium hydroxide (or the 0,1 mol/l solution for low formaldehyde contents) (see 4.2.2.2) contained in a suitable-capacity burette (4.2.3.4), until the pH is 3,5.

4.2.5.4 Blank test

Conduct a blank test in parallel with the determination, by the same procedure, using the same reagents as in the determination, but omitting the test portion.

4.2.6 Expression of results

Calculate the free-formaldehyde content $w(\text{CH}_2\text{O}, \text{free})$, expressed as a percentage by mass, using the equation:

$$w(\text{CH}_2\text{O}, \text{free}) = \frac{3c(V_1 - V_0)}{m}$$

where

c is the actual concentration, in moles per litre, of the solution of sodium hydroxide (4.2.2.2) utilized;

V_0 is the volume, in millilitres, of the solution of sodium hydroxide (4.2.2.2) utilized for the blank test (see 4.2.5.4);

V_1 is the volume, in millilitres, of the solution of sodium hydroxide (4.2.2.2) utilized for the determination (see 4.2.5.3);

m is the mass, in grams, of the test portion (see 4.2.5.2).

4.2.7 Precision

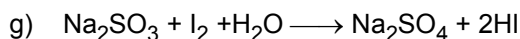
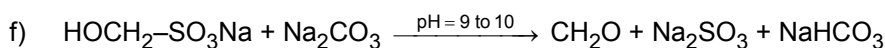
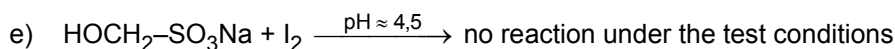
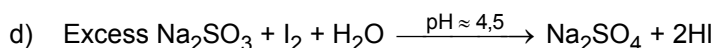
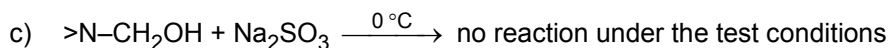
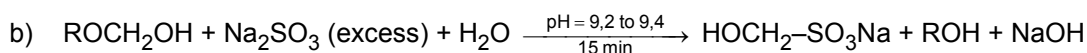
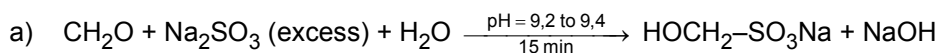
Repeatability: 0,2 % by mass formaldehyde;

Reproducibility: 0,4 % by mass formaldehyde.

4.3 Sulfite procedure

4.3.1 Principle

The method is based on the following reactions:



Free formaldehyde and alcohol formaldehyde semiacetals in a test portion are reacted with excess sodium sulfite solution at a temperature of 0 °C to form hydroxymethane sulfonate. The excess sodium sulfite is titrated with iodine solution. The hydroxymethane sulfonate is decomposed with sodium carbonate solution and the sodium sulfite liberated is titrated with iodine solution.

4.3.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

4.3.2.1 Sodium sulfite solution, $c(\text{Na}_2\text{SO}_3) = 1 \text{ mol/l}$.

4.3.2.2 Acetic acid, $c(\text{CH}_3\text{COOH}) = 1 \text{ mol/l}$.

4.3.2.3 Sodium carbonate solution, $c(\text{Na}_2\text{CO}_3) \approx 100 \text{ g/l}$.

4.3.2.4 Buffer solution.

Dissolve 12,37 g of boric acid in water in a 1 000 ml volumetric flask, add 100 ml of 1 mol/l sodium hydroxide solution, dilute to the mark with water and mix well.

Before use, cool the solution to 0 °C.

4.3.2.5 Iodine, standard volumetric solution, $c(\text{I}_2) = 0,05 \text{ mol/l}$, i.e. 12,690 g/l. If necessary, standardize the solution against sodium thiosulfate standard reference solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$.

4.3.2.6 Dichloromethane, neutral (pH = 7).

Before use, cool the dichloromethane to 0 °C.

4.3.2.7 Starch, dissolved in hot water to give a 10 g/l solution, or **powdered starch**, soluble in cold water (so-called Zulkovsky starch is suitable).

4.3.2.8 Water containing ice, prepared from water of at least grade 3 purity as defined in ISO 3696.

4.3.2.9 Ice, finely divided, prepared from water of at least grade 3 purity as defined in ISO 3696.

4.3.3 Apparatus

Ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1 (burettes) or ISO 648 (pipettes), together with the following:

4.3.3.1 High-speed mixer.

4.3.3.2 Magnetic stirrer.

4.3.3.3 Ice bath.

4.3.3.4 Burettes or, preferably, **microburettes**, of suitable capacity.

4.3.3.5 Pipettes, of capacity 10 ml and 25 ml.

4.3.4 Procedure

4.3.4.1 General

Carry out the determination in duplicate.

4.3.4.2 Test portion

By reference to Table 3, select the appropriate test-portion mass. If the free-formaldehyde content cannot be predicted, take a test portion of about 1 g and carry out a preliminary determination.

Weigh, to the nearest 0,001 g, the test portion into a 600 ml beaker.

Table 3 — Mass of test portion

Expected free-formaldehyde content % by mass	Approximate mass of test portion g
up to 0,5	3,0
from 0,5 to 1	1,5
from 1 to 2	1,0
from 2 to 3	0,5
from 3 to 5	0,25

4.3.4.3 Determination

Ensure that the temperature of the contents of the beaker is not higher than 0 °C during the whole determination. If necessary, add some finely divided ice (4.3.2.9) to the mixture.

In the case of water-soluble products, dissolve the test portion immediately in a mixture of 150 ml of water containing ice (4.3.2.8), about 10 g of finely divided ice (4.3.2.9) and 25 ml of buffer solution (4.3.2.4). In the case of products that do not form clear solutions with water, dissolve the test portion immediately in 50 ml of dichloromethane (4.3.2.6). Then add a mixture of 150 ml of water containing ice (4.3.2.8), about 20 g of finely divided ice (4.3.2.9) and 25 ml of buffer solution (4.3.2.4) and emulsify with the high-speed mixer (4.3.3.1) for 10 s. Withdraw the mixer and rinse it with a small volume of water containing ice (4.3.2.8) to remove adhering liquid. Collect the rinsings in the test solution.

Place the beaker in the ice bath (4.3.3.3) and stir the contents of the beaker, using the magnetic stirrer (4.3.3.2). Whilst continuously stirring, add, by means of a burette (4.3.3.4), 2 ml of sodium sulfite solution (4.3.2.1). Continue stirring for 15 min and then add 10 ml of acetic acid (4.3.2.2) and either 50 mg of powdered starch or 3 or 4 drops of starch solution (4.3.2.7). Titrate with iodine solution (4.3.2.5) until a greyish-blue or violet coloration is obtained that is stable for at least 10 s. Then add 30 ml of sodium carbonate solution (4.3.2.3). Titrate the liberated sodium sulfite with iodine solution until a blue coloration is obtained that is stable for at least 1 min. Record the volume V of iodine solution required for the titration of the liberated sodium sulfite.

4.3.5 Expression of results

Calculate the free-formaldehyde content $w(\text{CH}_2\text{O}, \text{free})$, expressed as a percentage by mass, using the equation:

$$w(\text{CH}_2\text{O}, \text{free}) = \frac{V \times 1,5 \times 0,1 \times f}{m}$$

where

V is the volume, in millilitres, of iodine solution (4.3.2.5) used;

f is an adjustment factor taking into account any difference in the concentration of the iodine solution (4.3.2.5) before standardization, $c(I_2)$ nominal, and after standardization, $c(I_2)$ actual, given by:

$$f = \frac{c(I_2) \text{ actual}}{c(I_2) \text{ nominal}};$$

m is the mass, in grams, of the test portion;

1,5 is the mass, in milligrams, of formaldehyde corresponding to 1,00 ml of iodine solution, $c(I_2) = 0,05 \text{ mol/l}$;

0,1 is the conversion factor necessary to convert milligrams to grams and to express w as a percentage.

If the two results (duplicates) differ by more than the value indicated in 4.3.6.1, repeat the procedure described in 4.3.4.

Calculate the mean of two valid results (replicates) and report the final result to the nearest 0,1 % by mass.

4.3.6 Precision

4.3.6.1 Repeatability, r

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method may be expected to lie with a 95 % probability is:

0,06 % by mass (absolute) for products having free-formaldehyde contents up to 1 % by mass;

6 % (relative) for products having free-formaldehyde contents above 1 % by mass.

4.3.6.2 Reproducibility, R

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method may be expected to lie with a 95 % probability is:

0,1 % by mass (absolute) for products having free-formaldehyde contents up to 1 % by mass;

10 % (relative) for products having free-formaldehyde contents above 1 % by mass.

4.4 KCN procedure

WARNING — Potassium cyanide is a very toxic material. It must be handled very carefully. In contact with acids, the toxic gas HCN is evolved. Work in a hood. Avoid contact with skin, eyes and clothing.

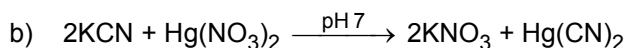
4.4.1 Principle

A test portion of the resin to be tested is dissolved or dispersed in water (if appropriate with the aid of N,N' -dimethylformamide), and the free formaldehyde quantitatively converted to cyanohydrin with excess potassium cyanide. The excess of potassium cyanide is subsequently back-titrated with mercury(II) nitrate solution using diphenylcarbazone as indicator.

4.4.2 Reactions

The analytical procedure is based on the following reactions:





4.4.3 Reagents

4.4.3.1 *N,N'*-dimethylformamide.

4.4.3.2 Nitric acid, $c(\text{HNO}_3) = 2 \text{ mol/l}$.

4.4.3.3 Potassium cyanide solution, $c(\text{KCN}) = 0,1 \text{ mol/l}$.

The concentration of the potassium cyanide solution shall be checked weekly against a standard reference solution of mercury(II) nitrate (4.4.3.6).

4.4.3.4 Phosphate buffer solution.

Dissolve 348 g of K_2HPO_4 and 136 g of KH_2PO_4 in water and make up to 1 000 ml.

4.4.3.5 Borate buffer solution.

Dissolve 76,4 g of $\text{K}_2\text{B}_4\text{O}_7 \cdot 4\text{H}_2\text{O}$ in water and make up to 1 000 ml.

4.4.3.6 Mercury(II) nitrate solution, $c[\text{Hg}(\text{NO}_3)_2] = 0,05 \text{ mol/l}$.

A dilute solution of formaldehyde of known concentration (e.g. 3,6 g/l) is used to determine the formaldehyde equivalent of the mercury(II) nitrate solution. Use 10 ml of this solution (corresponding to 36 mg of formaldehyde) for the determination. See Annex A for information on disposal of the analytical residues.

4.4.3.7 Indicator solution: 1 % diphenylcarbazone in methanol.

4.4.4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

4.4.4.1 Analytical balance, accurate to 0,1 mg.

4.4.4.2 Burettes, capacity 25 ml.

4.4.4.3 Graduated pipette, capacity 5 ml.

4.4.4.4 Transfer pipettes, capacity 10 ml and 20 ml.

CAUTION — Pipetting by mouth is prohibited.

4.4.4.5 Conical flask, capacity 500 ml, with ground-glass neck and stopper.

4.4.4.6 Stopwatch.

4.4.5 Procedure

4.4.5.1 Determination

Weigh into the 500 ml conical flask (4.4.4.5), to the nearest 0,1 mg, a test portion of not more than 1 g containing 10 mg to 40 mg of free formaldehyde. Dissolve or disperse the test portion in 150 ml of water [dissolve test portions having poor miscibility with water in 10 ml of *N,N'*-dimethylformamide (4.4.3.1) prior to addition of the water]. Add 40 ml of borate buffer solution (4.4.3.5) with a graduated cylinder, and immediately thereafter 20 ml of potassium cyanide solution (4.4.3.3) with a transfer pipette (4.4.4.4); then swirl the flask briefly. Close the flask and allow the mixture to react at room temperature for 2 min. Then add 5 ml of

phosphate buffer solution (4.4.3.4) with the graduated pipette (4.4.4.3) and 8 drops of diphenylcarbazone solution (4.4.3.7). Adjust the pH to 7 by adding nitric acid (4.4.3.2) from a burette until the mixture becomes almost colourless, then immediately titrate the solution with mercury(II) nitrate solution (4.4.3.6) to a light violet coloration.

4.4.5.2 Blank test

Check the blank value of the potassium cyanide solution once a week, using the same procedure and the same quantities of all reagents but omitting the test portion.

4.4.5.3 Control

In the case of resins containing a complexing agent for mercury(II) (Hg^{2+}) ions, determine, in addition, a control value by titration with mercury(II) nitrate solution (4.4.3.6), following the procedure described in 4.4.5.1 but without addition of potassium cyanide solution (4.4.3.3).

4.4.6 Expression of results

Calculate the free-formaldehyde content $w(\text{CH}_2\text{O}, \text{free})$, expressed as a percentage by mass, using the equation:

$$w(\text{CH}_2\text{O}, \text{free}) = \frac{(V_0 - V_1 + V_2) \times \rho(\text{CH}_2\text{O})}{10m_0}$$

where

V_0 is the volume, in millilitres, of mercury(II) nitrate solution (4.4.3.6) required for titration of 20 ml of potassium cyanide solution (4.4.3.3) in the blank test (4.4.5.2);

V_1 is the volume, in millilitres, of mercury(II) nitrate solution (4.4.3.6) required for titration of the test portion;

V_2 is the volume, in millilitres, of mercury(II) nitrate solution (4.4.3.6) required for titration of the test portion in the control test (4.4.5.3), if one is carried out;

m_0 is the mass, in grams, of the test portion;

$\rho(\text{CH}_2\text{O})$ is the formaldehyde equivalent, in milligrams per millilitre of mercury(II) nitrate solution, calculated from the equation:

$$\rho(\text{CH}_2\text{O}) = \frac{m_1}{V_0 - V_3}$$

m_1 being the mass, in milligrams, of formaldehyde used for the determination of the formaldehyde equivalent,

V_3 being the volume, in millilitres, of mercury(II) nitrate solution required.

4.4.7 Precision

4.4.7.1 General

The precision of this procedure was determined in a round-robin test in which nine laboratories took part.

4.4.7.2 Repeatability, r

At a free-formaldehyde content < 2 % by mass, $r = 0,08$ % by mass (absolute).

At a free-formaldehyde content ≥ 2 % by mass, $r = 4$ % (relative).

4.4.7.3 Reproducibility, R

At a free-formaldehyde content < 2 % by mass, $R = 0,16$ % by mass (absolute).

At a free-formaldehyde content ≥ 2 % by mass, $R = 8$ % (relative).

5 Test report

The test report shall include the following information:

- a) all details necessary to identify the product tested;
- b) a reference to this International Standard (ISO 11402);
- c) the sampling date;
- d) the procedure used;
- e) the free-formaldehyde content $w(\text{CH}_2\text{O}, \text{free})$ (individual and average values);
- f) any deviation from the test method specified;
- g) the date of the test.

Annex A (informative)

Suitability of the procedures for different types of resin

A.1 Carbonyl compounds

If a condensation resin contains aldehydes and/or ketones, adducts thereof (hemiacetals, hydrates) or dimers or oligomers thereof, these compounds may be partially or wholly determined in addition to formaldehyde.

A.2 Furan resins

Furan resins are condensation products of furfuryl alcohol and formaldehyde. They can be modified by chemical transformation or by physical distribution of the modification agent. They can contain up to 90 % of free furfuryl alcohol. If urea and melamine resins are used for modification, only the sulfite procedure is suitable (see also Clause A.3). The hydroxylamine hydrochloride procedure can be used for phenolic-resin-modified furan resins.

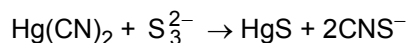
A.3 KCN procedure

At present, this is the only procedure suitable for mixed resins containing both phenol and urea or melamine. Urea resins hydrolyse partially when the hydroxylamine hydrochloride procedure is used; phenolic resins give secondary reactions when the sulfite procedure is used. Straight-chain phenolic resins, furan resins and completely etherified urea and melamine resins may be analysed using this procedure, but because of the toxicity of KCN the procedure should be limited to these resin types (UF/PF, MF/PF, UF/MF/PF) and only used when it is really necessary to obtain results with a high repeatability or reproducibility.

Annex B (informative)

Removal of Hg^{2+} and CN^- from mercury and cyanide residues

B.1 Reaction



B.2 Preparation of detoxification solution

Dissolve 30 g of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ in about 100 ml of water. Add 8 g of finely powdered sulfur, and bring the suspension to the boil in a covered vessel. The sulfur will dissolve after 30 min to 60 min. Cool, and dilute the solution to about 1 l. The solution contains approximately 0,125 mol/l of Na_2S_3 .

B.3 Treatment of titration solutions

Add approximately 10 ml of the detoxification solution to each titration solution — including those from the blank (4.4.5.2) and control (4.4.5.3) determinations — in a sealable collection vessel. The following day, the solution should be clear and it should not be possible to detect either Hg^{2+} or CN^- in the solution. Filter the solution or decant off the supernatant liquid, and discard into the sewage system.

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