

KES

Analysis of Nitrogen in Lubrication

KES T – M161

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1. Scope of Application

This standard provide for the method of quantitative analysis of nitrogen element contained in crude oil and petroleurn.

2. General Subject

General subject applied for chemical analysis commonly follows KS M 0001 and for concluding method of numerical value depend on KS A 0021.

3. Kind of Method of Examination

Kind of Method of Examination is like table 1.

Table 1 Kind of Method of Examination

Kind of Method of Examination	Special Quality(Reference)
Macro Kjeldahl'method	It is wet on analytical method of nitrogen on organic matter and is the method adjust well known Kjeldahl partially. It is an organic law to this standard because the amount of sample used a test is much by 0.2 ~ 2gr, it is long time to need to dissolve sample. (7 ~ 10hour) The quantitative method of nitrogen make neutral proper method.
Micro kjeldahl'method	It is the method miniaturizing macro Kjeldahl method. The amount of sample used test is smaller than that of macro Kjeldahl method (0.1 ~ 0.4gr). It is short time to need to dissolve sample (3 ~ 5 hour). The quantitative method of nitrogen make extinction brightness method.
Propriety law of very small amount and whole quantity	Because it is the analytical law of machinery and tools can get quickly nitrogen portion. But it need to get collection rate (Collection calculation) of nitrogen using standard material.

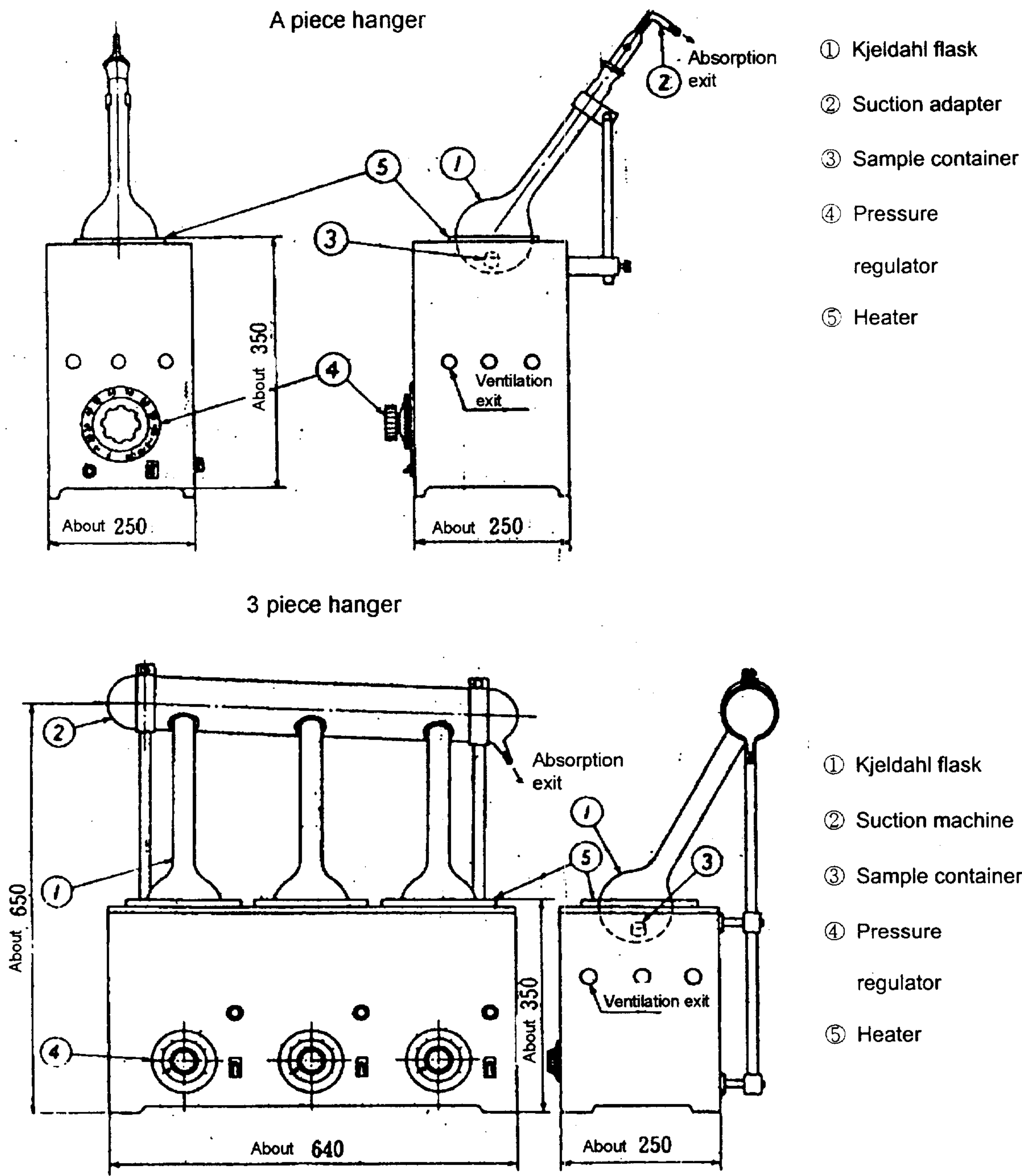
* In case that test result is a doubt depend on macro Kjeldahl method.

* In case of examining the accuracy of test result it is desirable for using the standard sample of nitrogen portion and the statistical examination method of accuracy refer to KS A 0004.

The picture 1 shows an example of electric heating form.

Note (1) It is proper for electric capacity(per one piece) 750w.

Unit : mm



Picture 1 Macro Kjeldahl sample dissolving equipment(Ex. of electric heating form)

4. Method of Examination

4.1 macro Kjeldahl method

4.1.1 Outline of test method

Among dark sulfuric acid putting catalyst heat and dissolve sample and in the samples get nitrogen changed with ammonium sulfate. In this place add strong alkali and distill steam. Make arising ammonia absorbed in boracic acid solution, with sulfuric acid standard solution neutralize and get proper and get nitrogen portion.

* This test method can't be applied for samples that put adding medicine contained nitrogenous compound have nitrogen-nitrogen union and nitrogen-oxygen union.

4.1.2 Macro Kjeldahl method testing machine

It is made up (1) ~ (5) and shows one example in picture 1 and picture 2. Also the electric insulation ability of testing machine must be over 5 MΩ when in the condition of closing the electric circuit using 500 V insulation resistance provided insulation resistance between sources of electricity terminal and an upper box for KS C 1301 [Insulation resistance world (Electric generator form)] or KS C 1302 [Insulation resistance world (Electric form)]. However in case of containing electric heat circuit it is desirable over 0.5 MΩ.

(1) Kjeldahl flask : As Kjeldahl flask in the use of chemical analysis use capacity 500 ml or 1000 ml

(2) Sample Container : Picture 208 provided for KS M 267
(Glass tool in use of testing petroleum class)

(3) Suction adapter and suction machine

Suction adapter is picture 20g provided for KS M 2670.

Suction machine is used picture provided for KS M 2670

(4) Macro Kjeldahl Sample dissolving equipment

It is gas or electric heating form and, what heat and maintain contents of Kjeldahl

flask by 360 ~ 380 °C

(5) Macro Kjeldahl Steam distilling apparatus : As what can be steam distilling flask and refrigerator and so on and the quality of glass part must be used hardened one grace snow example of its construction in the picture 2.

Unit : mm

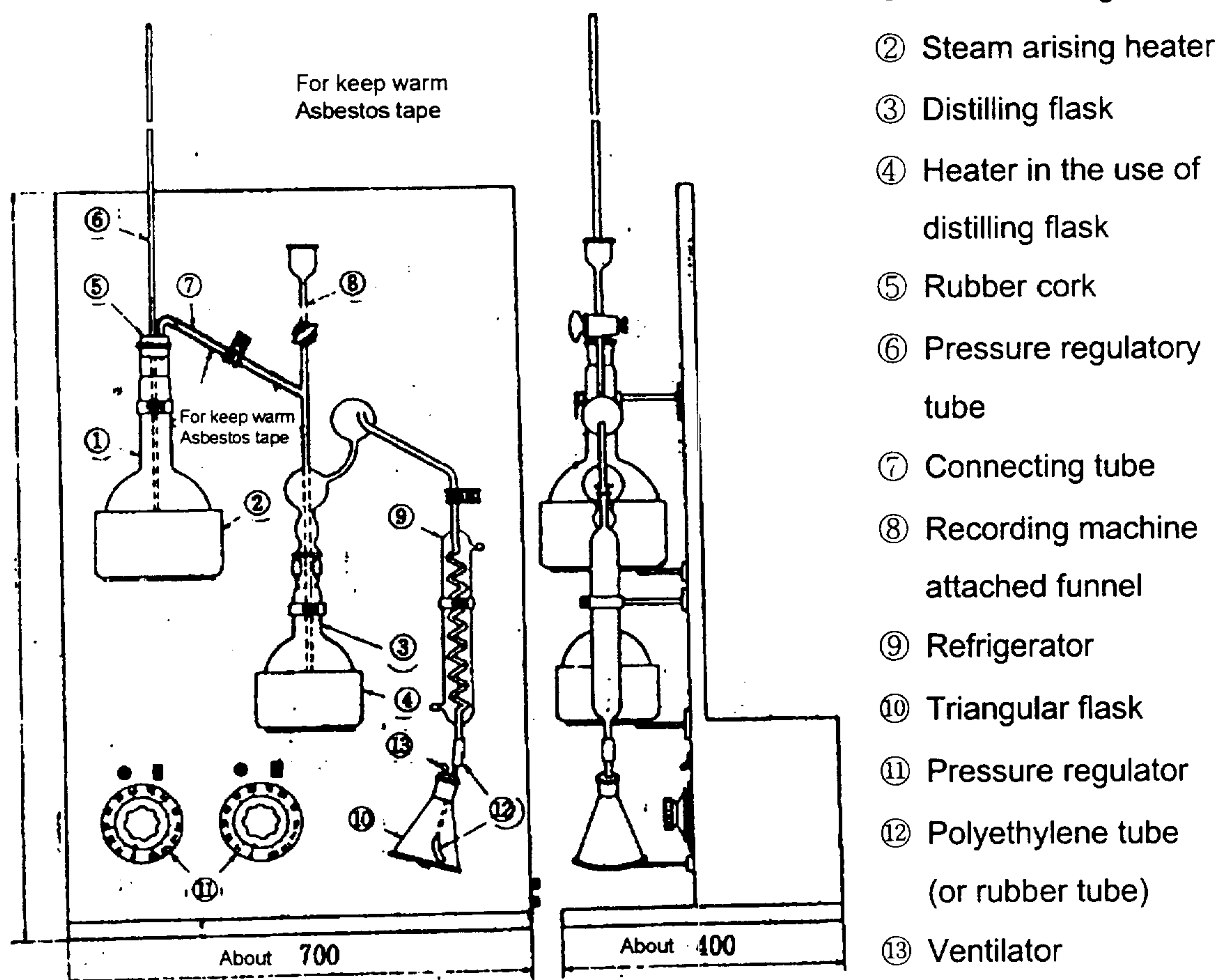


Fig. 2 Macro Kjeldahl steam distilling apparatus

Remark : As for the glass part of this apparatus it is desirable for compounding following KS standardized goods and using it.

(a) Steam arising flask : As for round bottom flask what capacity 1000 ml or 2000 ml.

- (b) Distilling flask : As for short head and round bottom flask what is capacity is 1000 mL.
- (c) Recording machine attached funnel : Picture 211 provided for KS M 2670
- (d) Refrigerator : Picture 212 provided for KS M 2670
- (e) Connecting tube : Picture 213 provided for KS M 2670

4.1.3 Reagent : Follow test using in the macro Kjeldahl method

- (1) Sulfuric acid : Use what provided for KS M 8103 [Sulfuric acid (Reagent)]
- (2) Sucroso : Use test grade
- (3) Sodium hydroxide solution (50 weight %) : Use what take water 500 mL get expelled cold up to real temperature in the container made stainless steel, dissolve sodium hydroxide 500g provided for KS M 8116 [Sodium hydroxide (Reagent)]

Note (2) Wear protective gloves and protective glasses in case of the preparing of solution and dealing.

- (4) Sodium hydroxide solution (0.04 weight %)

After dissolving sodium hydroxide 500g provided for KS M 8116 [Sodium hydroxide (Reagent)] in the water make total amount 1000 mL with water.

- (5) Catalyst : Mix copper sulfate 5 hydrate provided for KS M 3124 [Sulfuric acid copper (Reagent)] and potassium sulfate with the ratio 1 to 25 (Weight ratio) and use it using potassium sulfate remove what shatters proper amount to self-control melting pot, put it in the electric circuit, make it raised slowly from 100 °C to 500 ~ 600 °C and heat for 12 hours. After heating use what expelled cold in the deccigater.
- (6) Boiling stone : After boiling carborandom particle and minute porcelain and so on in sulfuric acid for thirty minute removing nitrogenous compound wash it with water and dry.
- (7) Boric acid solution (0.4 weight %) : Dissolve boric acid 4.0g provided for KS M 8118 [Boric acid (Reagent)] in the water 1000 mL.

(8) According to 0.01N sulfuric acid standard solution KS M 8006 (Basic data concerning propriety in the test of content) Prepare and express 0.1 N sulfuric acid standard solution and use what accurately dilute this ten times.

(9) Mixing indicator : Use what mix following the kinds of solution with similar amount.

Must preserve this mixing indicator in brown bottle. Not have to use what passe over a week.

(a) Methyl Red (Reagent) : Dissolve 0.125g in the ethyl alcohol (95) 100 ml provided for KS M 8101 [Ethyl alcohol (Reagent)]

(b) Dissolve methylene Blue (2water base) 0.0083g provided for KS M 8274 [Methylene Blue/ 2water base, 3water base, 4water base (Reagent)] in the ethyl alcohol 100 ml provided for KS M 8101.

Note ⁽³⁾ May use methyl purple 0.1 % solution instead of mixing indicator.

(10) Zinc : Use zinc (Reagent)

4.1.4 Order of examination : It is as follows examination order of macro Kjeldahl Method

(1) Extraction of sample : Measure sample amount showed in the table 2 in the sample container up to 1 mg and extract it. Put it in acid coping with sample amount, again put boiling stone 3 ~ 5 piece knowing the weight, shake it and mix.

Table 2 Extraction amount of sample and sulfuric acid amount

Nitrogen portion (weight %)	Extraction amount of sample (g)	Sulfuric acid amount (ml)
Less than 0.05	2	50
0.05 ~ 0.1	1	40
0.1 ~ 0.25	0.5	30
Surplus 0.25	0.2	30

(2) Resolution

- (a) Put Kjeldahl flask on the equipment resolving macro Kjeldahl sample and connect with suction adapter or after connecting with suction machine heat to be sucked.
- Remark :** If uses suction machine fulfill glass cotton between the neck part of Kjeldahl flask and the gap of connecting part of suction machine to raise suction efficiency.
- (b) Be careful that bubble doesn't arise strongly in the contents of Kjeldahl flask and slowly elevate heating. When bubble arise strongly is weakened heating. Although is weakened heating when bubble arise strongly add sulfuric acid 10 ~ 15 ml.
- (c) If strong bubble dries down regulate heating to condense and return arising sulfuric acid steam from the mouth of Kjeldahl flask to $\frac{1}{2} \sim \frac{3}{4}$ situation of its neck part and the carbide attached the neck part of Kjeldahl flask is washed and down totally according to reflux liquid until contents will become brown suction heat continually.
- (d) Stop heating, leave Kjeldahl flask as it is for ten minutes and put a catalyst 25g in it. Heat again sulfuric acid reflux in the neck part of Kjeldahl flask until contents will become colorlessness and clearness solution again heat for 30 ~ 60 minutes and resolve.
- (e) After finishing resolution expel cold up to real temperature and measure the weight of Kjeldahl flask being contents. Output final catalyst rate according to following form and verify that it is the scope of 1.0-1.5.
- If it is less than 1.0 heat again and evaporate sulfuric acid.
- If it is more than 1.5 do again from(1), in this time add sulfuric acid amount to 5 ml excessively more than that of provided for table 2.

$$R = \frac{1.8 C}{S} = \frac{1.8 C}{A - (C + F + B + V)}$$

In this place, R : Final catalyst rate

C : Adding amount of catalyst (= 25) (g)

S : Serving sulfuric acid amount (g)

A : Weight of Kjeldahl flask contained contents after finishing
resolution (g)

F : Weight of empty Kjeldahl flask (g)

B : Weight of boiling stone

V : Weight sample vessel (g)

(f) Subsequently refrigerate Kjeldhal flask in the ice cooking in a double boiler, add slowly water 300 ml and dissolve salt extracted.

(3) Steam distillation

(a) Remove only solution is in the Kjeldahl flask to distilling flask wash inner Kjeldahl flask with water 100 ml and add solution washed. In this place add zinc 1g.

(b) Put boric acid solution (0.4 weight %) 50 ml in 500 ml triangular flask, and construct macroKjeldahl steam distilling apparatus (Reference picture 2)

Send cooling water to refrigerator and add in to the distilling flask in the distilling flask in the use of a funnel sodium hydroxide solution indicated a double numerical valve of surviving sulfuric acid amount as it is by ml.

Note (4) This operation make suction from motive engine attached the cork of 500 ml triangular flask.

(c) Adjust heating not to boil suddenly the flask arising steam and distilling flask.

If solution in the 500 mL triangular flask becomes 100 ~ 150 mL pull out a cork, lift up the front end of polyethylene tube (or rubber tube) doesn't to be sunk in the 500 mL triangular flask solution, continue steam distillation for one minute and stop heating. Wash the front end of polyethylene tube (Rubber tube) with a little water.

Note ⁽⁵⁾ If the solution amount of 500 mL triangular flask gets to near to 150 mL as salt is extracted in the distilling flask and easy to boil suddenly need to adjust heating for a bounded drop not to be mixed among the effluence.

- (4) Propriety : Add mixing indicator 0.5 mL in the 500 mL triangular flask solution is proper in use of 0.01 N sulfuric acid standard solution and make the point that the solution discolours from right green to purple color final point.

Note ⁽⁶⁾ If the decision of final point is difficult put boric acid solution (0.4 weight %) 50 mL in other 500 mL triangular flask, add water here and make same amount with proper solution. Add mixing indicator 0.5 mL and solution hydroxide (0.04 weight %) 1 ~ 2 drop and make green color. Compare this solution with the color of final point neutralize and is proper with 0.01 N boric acid solution.

Note ⁽⁷⁾ In case of using metal purple make the point discolored from grayish blue to purple color the final point.

- (5) Base Test : Operate (1) ~ (4) in the use of sucroso instead of sample.

Note ⁽⁸⁾ The amount of using of sucroso is selected from table 3 according to sulfuric acid amount used resolving sample. And catalyst amount becomes 25g for anything.