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# HG

## Chemical Industry Standard of the People's Republic of China

HG/T 3278-2018

Replace HG/T 3278-2011

### Sodium humate

### 腐植酸钠

( English Translation )

( 报批稿 )

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## Foreword

SAC/TC 105/SC 7 is in charge of the English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

This standard is drafted in accordance with the rules given in the GB/T 1.1 *Directives for standardization — Part 1: Structure and drafting of standards*.

This standard replaces the HG/T 3278—2011 *Sodium humate for agricultural use* in whole. In addition to a number of editorial changes, the following technical deviations have been made with respect to the HG/T 3278—2011:

— The title of the standard has been changed from "sodium humate for agricultural use" to "sodium humate".

— The definition of "sodium humate"(see 3.1) has been added. The definition of "humic acid" (see 3.2,3.1 in 2011) and "soluble humic acid" (see 3.3, 3.7 in 2011) have been revised. "humic acid from organic mineral"(see 3.2 in 2011) , "humic acid from non-mineral biomass"(see 3.3 in 2011), "pyrotomalenic acid"(see 3.4 in 2011) , "hymatomelanic acid"(see 3.5 in 2011), "fulvic acid"(see 3.6 in 2011) and "water soluble humic acid"(see 3.8 in 2011) have been cancelled.

— "Table 1"(see 4.2 in 2011) has been revised. The limit requirements of "As, Cd, Pb, Cr, Hg" have been increased (see 4.3).

— The testing method of "The content of soluble humic acid" has been changed from "differential volumetric method" to "gravimetric method". "the residue method"(see 5.3 in 2011) has been removed. "testing of ash content"(see 5.6 in 2011) has been removed. The testing method of "content of As, Cd, Pb, Cr, Hg" has been increased (see 5.8).

This standard was proposed by China Petroleum and Chemical Industry Federation.

This standard was prepared by SAC/TC 105/SC 7 Subcommittee 7 on Humic-Acids Fertilizers of National Technical Committee 105 on Fertilizers and Soil Conditioners of Standardization Administration of China.

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This standard was issued in December 1987 as first edition, was first revised in December 2011. This is the second revised edition.

# Sodium humate

## 1 Scope

This standard specifies terms and definitions, technical requirements, test methods, inspection rules, marking, packaging, transport and storage of sodium humate.

This standard is applicable to sodium humate which is produced by reaction of sodium hydroxide with raw material of weathered coal, brown coal and peat under certain conditions.

## 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.

GB/T 191, *Packaging — Pictorial marking for handling of goods*

GB/T 6679, *General rules for sampling solid chemical products*

GB/T 8170, *Rules for rounding off for numerical values & expression and judgement of limiting values*

GB 18382, *Fertilizer marking — Presentation and declaration*

GB/T 23349, *Ecological index of arsenic, cadmium, lead, chromium and mercury for fertilizers*

GB/T 24891, *Determination of particle size for compound fertilizers*

HG/T 2843, *Chemical fertilizer products — standard volumetric, standard, reagent and indicator solutions for chemical analysis*

## 3 Terms and definitions

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

### 3.1

#### **sodium humate**

the products produced by reaction of sodium hydroxide with raw material of weathered coal, brown coal and peat under certain conditions

### 3.2

#### **humic acid**

humic substances(also called humic acid substances) are black or dark brown amorphous organic weak acid compounds which are high molecular weight, with characterizations of aromatic aliphatic series and multiple functional group structure, soluble in dilute alkaline solution and non-soluble in acid and water

## 3.3

**soluble humic acid**

ionic humic acid dissolved in aqueous solution of humic acid fertilizers and humate products from mineral sources. It is the main quality index of humic acid fertilizer and humate products.

## 4 Requirements

4.1 Appearance quality: Uniform black, dark brown particle or powder.

4.2 The technical specifications of the sodium humate products shall comply with the requirements in Table 1.

Table 1 — The requirements of technical specifications of sodium humate products

Items	Specifications			
	Excellent grade	Grade 1	Grade 2	Grade 3
Soluble humic acid content (as dry base) / %	≥60	≥50	≥40	≥30
Water insoluble content (as dry base) / %	≤5	≤10	≤20	≤25
Moisture / %	≤15		≤20	
Value of pH (1 % solution)	8~10		9~11	
Residue on sieve 1.00mm <sup>a</sup> / %	≤5			
Particle size(1.00 mm~4.75mm or 3.35 mm~5.60 mm) <sup>b</sup> / %	≥70			
<sup>a</sup> It is not required for granule products.				
<sup>b</sup> It is not required for powder products.				

4.3 The limits of arsenic, cadmium, lead, chromium and mercury shall comply with the requirements in Table 2.

Table 2 — The limits requirements of arsenic, cadmium, lead, chromium and mercury in sodium humate

Items	Specifications
Arsenic and its compounds (calculated by As) / %	≤0.005 0
Cadmium and its compounds (calculated by Cd) / %	≤0.001 0
Lead and its compounds (calculated by Pb) / %	≤0.020 0
Chromium and its compounds (calculated by Cr) / %	≤0.050 0
Mercury and its compounds (calculated by Hg) / %	≤0.000 5

## 5 Test method

The reagent, water and preparation of solution in this standard shall conform to HG/T 2843 when the specifications and preparation methods are not indicated.

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## 5.1 Appearance quality

Visual method.

## 5.2 Determination of soluble humic acid content (Gravimetry method)

### 5.2.1 Method summary

Extract soluble humate from the soluble humic acid with water under certain temperature, then acidify the extraction until the value of pH to 1 by a certain amount of acid, filter it by constant weight medium flow quantitative paper, wash the precipitate by HCl (pH = 1) many times till filtrate is colorless, dry the precipitate to constant weight in drying oven, weigh it, then calculate the content of soluble humic acid by subtracting ash content.

### 5.2.2 Reagents and materials

5.2.2.1 HCl:  $\rho = 1.18 \text{ g/mL}$ .

5.2.2.2 HCl solution:  $c(\text{HCl}) = 2.0 \text{ mol/L}$ .

Place 166 mL HCl along the wall of beaker slowly into water and stir, then cool to room temperature and dilute to 1000 mL.

5.2.2.3 HCl solution: pH = 1.

Add a certain amount of laboratory water into the beaker, drop the hydrochloric acid solution with  $c(\text{HCl}) = 2.0 \text{ mol/L}$ , measure the pH value of the solution with a pH meter till the value of pH to 1, and put it into a washing bottle for later use.

### 5.2.3 Apparatuses

5.2.3.1 General laboratory apparatus.

5.2.3.2 Electric thermostat drying oven: the temperature can be controlled at  $(110 \pm 5) \text{ }^\circ\text{C}$ .

5.2.3.3 Analytical balance: precision 0.000 1 g.

5.2.3.4 Centrifugal machine: 3 000 r/min~4 000 r/min.

5.2.3.5 pH meter: precision 0.01.

5.2.3.6 Adjustable hotplate: application of temperature is from room temperature to 400  $^\circ\text{C}$ .

5.2.3.7 Muffle furnace: rated temperature 1 200  $^\circ\text{C}$ .

## 5.2.4 Analysis procedures

### 5.2.4.1 Sample preparation

Take approximately 200 g through the reduction sampling of sodium humate, grind till it pass the mesh with 0.2 mm completely, place it in the clean and dry bottle, and store at room temperature.

### 5.2.4.2 Extraction

Weigh 1.0 g (accurate to 0.000 1 g) solid sample, transfer to 300 mL conical flask, add 100 mL distilled water, shake it to make the sample wet, put a small funnel on the flask, then place it in boiling bath (oil bath) to heat for 30 min, within the period shake it 3~4 times. Take out the flask and cool down to room temperature. Transfer all extract and residue into a 200 mL centrifugal cup, centrifuge it for 30 min at 3 000 r/min. With a dry constant weighed medium flow qualitative filter paper, filter supernatant by decantation, transfer the residue totally, wash it until the filtrate is colorless, place all filtrate in a conical flask and reserve.

### 5.2.4.3 Acidify precipitate

Add appropriate HCl (5.2.2.1) to the filtrate in the flask, then adjust till the value of pH stably to 1 with HCl (5.2.2.2), let stand for 30 min, dry-filter it with constant weight medium flow quantitative filter paper (avoid the precipitate scattering and try best to put it in the bottom of the paper). Wash the precipitate with HCl (pH = 1) (5.2.2.3) till the filtration is colorless, reserve the precipitate.

### 5.2.4.4 Drying of precipitate of humic acid

Place the constant weight medium flow quantitative filter paper ( $m_3$ ) and precipitate into drying oven, dry it for 4 h at  $(110 \pm 5) ^\circ\text{C}$  until constant weight, weigh the total mass of humic acid precipitate and quantitative filter paper ( $m_1$ ).

### 5.2.4.5 Ashing of humic acid precipitate

Place humic acid precipitate and quantitative filter paper (5.2.4.4) into a constant weight crucible which is combusted to constant weight in Muffle furnace, slightly shake it to flat, carbonize it at low temperature in a fume hood with an adjustable hot plate, the temperature is no more than  $100 ^\circ\text{C}$  in the beginning, and heat up to  $250 ^\circ\text{C} \sim 300 ^\circ\text{C}$  gradually till smokeless. Then move this crucible into Muffle furnace, raise the temperature slowly to  $500 ^\circ\text{C}$  in 30 min, and keep for 30 min. Continue to raise the temperature to  $(815 \pm 10) ^\circ\text{C}$  for 2 h. Take it out and cool in the air for 5 min, then transfer it to a desiccator to cool down to room temperature (appr.20min), weigh and calculate total ash mass ( $m_2$ ) of humic acid precipitate and quantitative filter paper, at the same time measure ash mass of quantitative filter paper ( $m_0$ ).

## 5.2.5 Expression of analysis results

The value of the mass fraction of the soluble humic acid content  $HA_{s,d}$  expressed as %, calculated according to formula (1):

$$HA_{s,d} = \frac{(m_1 - m_3) - (m_2 - m_0)}{m \times \left( \frac{100 - M_{ad}}{100} \right)} \times 100 \quad \dots\dots\dots(1)$$

where

- $m_1$  is the value of the total mass of soluble humic acid precipitate and quantitative filter paper after drying, in grams (g);
- $m_3$  is the value of the mass of constant weight quantitative filter paper, in grams (g);
- $m_2$  is the value of the total mass of soluble humic acid precipitate and quantitative filter paper, in grams (g);
- $m_0$  is the value of the mass of the ash of quantitative filter paper, in grams (g);
- $m$  is the value of the mass of sample taken for the determination, in grams (g);
- $M_{ad}$  is the moisture of test portion, expressed as %.

The result is reserved to one decimal place. The arithmetic mean value of the parallel measurement results is taken as the measurement results.

#### 5.2.6 Allowable deviation

The absolute difference of parallel determination results and different laboratory determination results shall conform to Table 3.

Table 3 — Allowable deviation of the analysis results of soluble humic acid content

Soluble humic acid content / %	The absolute difference of parallel determination results	The absolute difference of different laboratory determination results
<25	≤1.0	≤1.5
25~50	≤2.0	≤3.0
>50	≤3.0	≤4.0

### 5.3 Moisture (Gravimetric method)

#### 5.3.1 Method summary

Dry the test portion in an oven at  $(105 \pm 2) ^\circ\text{C}$  and the mass fraction of the loss compared with the test portion, is expressed as moisture.

#### 5.3.2 Apparatuses

##### 5.3.2.1 General laboratory apparatus.

##### 5.3.2.2 Electric thermostat drying oven: the temperature may be controlled at $(105 \pm 2) ^\circ\text{C}$ .

5.3.2.3 Ground-in stoppered weighing bottle:  $d$  50 mm×30 mm.

### 5.3.3 Analysis procedures

Weigh 1.0 g (accurate to 0.000 2 g) test portion with a pre-dried constant weight weighing bottle, open the stopper, place the bottle into the drying oven which is preheated to  $(105 \pm 2)$  °C for 2 h, take it out and put on the stopper, cool in the air for 2 min~3 min, then place it in a desiccator to cool down to room temperature (appr.20 min), weigh it. Then dry it for 30 min each time by checking the test portion mass until its difference is less than 0.001 g or increase. In the case of increase the former mass is used ( $m_4$ ).

### 5.3.4 Expression of analysis results

The mass fraction of the moisture of the sample  $M_{ad}$  expressed as %, calculated according to formula (2):

$$M_{ad} = \frac{m - m_4}{m} \times 100 \dots\dots\dots(2)$$

Where

$m$  is the value of the mass of test portion, in grams (g);

$m_4$  is the value of the mass of dried test portion, in grams (g).

The result is reserved to one decimal place. The arithmetic mean value of the parallel measurement results is taken as the measurement results.

### 5.3.5 Allowable deviation

The absolute difference of parallel determination results shall conform to Table 4.

Table 4 — Allowable deviation of the measure results of the moisture

Moisture (%)	The absolute difference of parallel determination results
<5	≤0.2
5~10	≤0.3
>10	≤0.4

## 5.4 Value of pH

### 5.4.1 Method summary

Measure the pH value of the 1 % sample solution by pH-meter.

### 5.4.2 Apparatuses

5.4.2.1 General laboratory apparatus.

5.4.2.2 pH meter, precision 0.01.

5.4.2.3 Electromagnetic stirrer.

5.4.3 Reagents

5.4.3.1 Phthalate standard buffer:  $c(\text{C}_6\text{H}_4\text{CO}_2\text{HCO}_2\text{K}) = 0.05 \text{ mol/L}$ ,  $\text{pH} = 4.003$ .

5.4.3.2 Phosphate standard buffer:  $c(\text{KH}_2\text{PO}_4) = 0.025 \text{ mol/L}$ ,  $c(\text{Na}_2\text{HPO}_4) = 0.025 \text{ mol/L}$ ,  $\text{pH} = 6.864$ .

5.4.3.3 Borate standard buffer:  $c(\text{Na}_2\text{B}_4\text{O}_7) = 0.01 \text{ mol/L}$ ,  $\text{pH} = 9.182$ .

5.4.4 Analysis procedures

Adjust pH meter according to the manual, then locate and calibrate with standard buffer. Weigh 1.0 g (accurate to 0.001 g) test portion into 150 mL beaker, add 100 mL water, place it on electromagnetic stirrer, stir it for 10 min under room temperature, then measure pH of the test portion solution with the adjusted, calibrated pH meter.

The arithmetic mean value of the parallel measurement results is taken as the measurement results.

5.4.5 Allowable deviation

The absolute difference in parallel determination results shall not exceed 0.2 pH.

5.5 Determination of water insoluble matter content

5.5.1 Method summary

Dissolve test portion with water and centrifuge it to receive water insoluble, the mass fraction of water insoluble mass compared with the mass of test portion is expressed as water insoluble content.

5.5.2 Apparatuses

5.5.2.1 General laboratory apparatus.

5.5.2.2 Electric thermostat drying oven: capable of being controlled at  $(110 \pm 5) \text{ }^\circ\text{C}$ .

5.5.2.3 Ground-in stoppered weighing bottle:  $d 70 \text{ mm} \times 35 \text{ mm}$ .

5.5.2.4 Medium flow quantitative paper.

5.5.2.5 Centrifugal machine: the lowest speed is 2 000 r/min, the volume of the centrifuge tube is greater than 150 mL.

5.5.2.6 Thermostat water bath: the controlled range of temperature is room temperature  $\sim 100 \text{ }^\circ\text{C}$ .

### 5.5.3 Analysis procedures

Weigh 1.0 g test portion (accurate to 0.000 2 g), place it into 250 mL conical flask, add 100 mL water, heat it in boiling water bath for 30 min, take it out and cool down to room temperature, then transfer the solution and residue totally to centrifuge tube and centrifuge it for 30 min at 2 000 r/min, decant the supernatant to receive the residue.

Dry quantitative paper and weighing bottle to constant weight in  $(110 \pm 5) ^\circ\text{C}$  drying oven, filter the residue in tube with the constant weight paper, wash it till the filtrate is colorless, then put the residue and paper into the weighing bottle, dry it in  $(110 \pm 5) ^\circ\text{C}$  drying oven for 2 h, take it out, cool for 2 min ~ 3 min at room temperature, then place it in desiccator to cool down to room temperature (appr.20min), weight it. Repeatedly dry, cool and weigh it until the difference between two successive weights is  $\leq 0.001$  g, take it as the mass of water insoluble ( $m_5$ ).

### 5.5.4 Expression of analysis results

The mass fraction of the content of water insoluble ( $W_d$  as dry basis) expressed as %, calculated according to formula (3):

$$W_d = \frac{m_5}{m \times \left( \frac{100 - M_{ad}}{100} \right)} \times 100 \dots\dots\dots(3)$$

Where

- $m_5$  is the value of the mass of water insoluble, in grams (g);
- $m$  is the value of the mass of test portion, in grams (g);
- $M_{ad}$  is the value of the moisture of test portion, expressed as %.

The result is reserved to one decimal place. The arithmetic mean value of the parallel measurement results is taken as the measurement result.

### 5.5.5 Allowable deviation

The absolute difference of parallel determination results and different laboratory determination results shall conform to Table 5.

Table 5 — Allowable deviation of the content of water insoluble results

the content of water insoluble / %	The absolute difference of parallel determination results	The absolute difference of different laboratory determination results
$\leq 20$	$\leq 1.0$	$\leq 1.5$
$> 20$	$\leq 2.0$	$\leq 3.0$

### 5.6 Determination of residue on sieve

### 5.6.1 Method summary

Pass the test portion through 1.00 mm aperture size sieve, the mass fraction of the residue on the sieve of test portion is the content of residue on sieve.

### 5.6.2 Apparatuses

#### 5.6.2.1 General laboratory apparatus.

#### 5.6.2.2 Testing sieve: aperture size 1.00 mm (with cover and receiver).

#### 5.6.2.3 Sieve shaker.

### 5.6.3 Analysis procedures

Weigh 100 g test portion (accurate to 0.1 g), place it on aperture size 1.00 mm sieve, cover it up and put it on shaker, clamp it, shake it for 5 min, or sieve it manually. Weigh the test portion which has not passed the 1.00 mm sieve ( $m_6$ ) (accurate to 0.1 g), including particles caught in the mesh of the sieve.

### 5.6.4 Expression of analysis result

The value of the residue on sieve which has not passed the 1.00 mm sieve  $\rho_{ad}$  expressed as %, calculated according to formula (4):

$$\rho_{ad} = \frac{m_6}{m} \times 100 \quad \dots\dots\dots(4)$$

Where

- $m_6$  is the value of the mass of test portion which has not passed the 1.00 mm sieve, in grams (g) ;
- $m$  is the value of the mass of test portion, in grams (g).

### 5.7 Particle size (sieving method)

According to GB/T 24891.

### 5.8 Determination of arsenic, cadmium, lead, chromium and mercury content

According to GB/T 23349.

## 6 Inspection rules

### 6.1 Products inspection and judgement by manufacturer

6.1.1 The products are inspected by quality supervision department of manufacturer, and the production enterprise shall guarantee that all the products for sale comply with the requirements in item 4.1, 4.2, 4.3 of

this standard. Each batch of products shall be accompanied by quality certificate, the contents in which shall be in accordance with the marking regulations.

6.1.2 When the production enterprise carries out the factory inspection, if all the inspection results are qualified, the product is judged to be qualified. In case of any unqualified items, double quantity of packaging bags shall be sampled from the same batch of products for re-inspection. When the re-inspection results comply with all the requirements of this standard, the product is judged as qualified. If there are still unqualified items, the product is judged as unqualified.

6.1.3 When all the type inspection items conform to the requirements, the batch of products are judged as qualified.

## 6.2 Factory inspection

6.2.1 The products are inspected according to batches, and the daily output is regarded as one batch. The maximum batch size is 500 t.

6.2.2 Each batch of products leaving the factory shall be attached with product certificate or quality certificate.

6.2.3 The factory inspection items are the specifications in 4.1, 4.2.

## 6.3 Type inspection

Type inspection items are the items specified in 4.1, 4.2 and 4.3.

Type inspection shall be proceeded in one of the following cases:

- a) when appraisalment of trial manufacture of new products or products produced by new plant is conducted;
- b) when significant changes in raw materials and process after the formal production, which is possible to impact the indicators of the products quality;
- c) when production is resumed after 6 months of production suspension;
- d) for normal production, type inspection shall be carried out periodically and at least once every 6 months;
- e) the state quality supervision institution require to carry out the type inspection.

## 6.4 Sampling plan

Carried out product sampling according to the requirements of GB/T 6679.

## 6.5 Judgment of qualified quality index

The "rounding off value comparison method" in GB/T 8170 is adopted to judge whether the quality index is qualified.

## 7 Marking

7.1 The product quality certificate shall specify the name of the production enterprise, the product name, the type of humic acid raw material (weathered coal, lignite, peat), grade, production date, technical specifications, inspection result and inspector.

7.2 The packaging bag of the product shall specify the name of the production or business enterprise, address, product name, grade, type of humic acid raw materials (weathered coal, lignite and peat), minimum marked value of soluble humic acid content, minimum marked value of water insoluble substance content, minimum marked value of moisture, pH value and standard number. Carry out others as specified in GB 18382.

## 8 Packaging

8.1 The products are packed in woven bags lined with polyethylene (polypropylene) bags or in three-in-one bags. The net content of each bag is  $(50 \pm 0.5)$  kg and  $(25 \pm 0.25)$  kg, and the average net content of each bag of each batch of products shall not be less than 50.0 kg and 25.0 kg.

8.2 Powder or ultra-fine powder products shall be sealed with aluminum-plastic composite bags with a heat sealing width of more than 15 mm.

8.3 When the buyer has special requirements for the net content of each bag, the supplier and the demander can reach an agreement and implement the agreement.

## 9 Transport and storage

9.1 The products shall be loaded or unloaded lightly during transportation, and the transportation tools and loading tools shall be clean, flat and free from protruding sharp objects.

9.2 The products shall be moisture-proof, wet-proof, sun-insolated-proof and breakage-proof during transport and storage. The warning instructions are implemented according to those specified in GB/T 191.