

ICS 65.100.20
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GB

National Standards of the People's Republic of China

GB XXXX - XXXX

Pendimethalin Technical

(Draft for Approval)

(Draft completion date: December 2006)

Issue Date: 200X – XX -XX

Implementation Date: 200X –XX -XX

**Issued by the General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China and the Standardisation
Administration of the People's Republic of China**

Foreword

Articles 3 and 5 of this standard are mandatory, while the rest are recommended.

This standard is proposed by the China Petroleum and Chemical Industry Association

This standard is under the jurisdiction of the National Pesticide Standardisation Technical Committee (**CSBTC/TC133**).

The organisation in charge of drafting this standard was: Shenyang Chemical Industry Research Institute.

The organisations which participated in the drafting of this standard were: Shandong Huayang Technology Co., Ltd and Jiangsu Longdeng Chemical Co., Ltd.

The main drafters of this standard were: Zhang Pulong, Hou Chunqing, Zhu Fengxia, Feng Xiuzhen and Li Peng.

The interpretation of this standard is entrusted to the secretariat of the National Pesticide Standardisation Technical Committee.

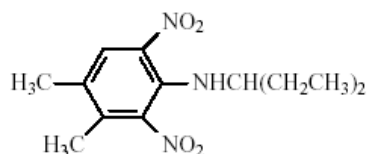
Pendimethalin Technical

Other names of this product legitimately containing the ingredient pendimethalin, its structure and basic physical and chemical parameters are as follows:

ISO common name: Pendimethalin

CIPAC numerical code: 357

Chemical name: *N*-(1 - ethylpropyl) - 2, 6 - nitro -3, 4 - dimethylaniline



Molecular formula: C₁₃H₁₉N₃O₄

Relative molecular weight: 281.3 (by 2001 the international atomic relative mass)

Biological activity: weeding

Melting point: 54 • to 58 •

Vapour pressure (25 •): 4.0 × 10⁻³Pa

Solubility (26 • , g/l): water 3.0 × 10⁻⁴ (20 •), acetone 700, corn oil 148, isopropanol 77, xylene 628, heptane 138, soluble in benzene, toluene, chloroform and dichloromethane, slightly soluble in petroleum ether and petrol

Stability: stable in 5 • to 130 • , DT₅₀ < 21d in water, stable in acid and alkali, DT₅₀ = 30d to 90d in soil, slowly decomposed under light.

1 Scope

This standard sets the requirements, test methods, marking, labelling, packaging and transportation methods for pendimethalin technical.

This standard applies to pendimethalin and pendimethalin technical composed by production impurities.

2 Normative References

The provisions in the following documents become provisions of this standard after being referenced. For dated reference documents, all later amendments (excluding corrigenda) and versions do not apply to this standard; however, the parties to the agreement are encouraged to study whether the latest versions of these documents apply. For undated reference documents, the latest versions apply to this standard.

GB/T 1600	Testing method of water in pesticides
GB/T 1601	Testing method for the pH value of pesticides
GB/T 1604	Acceptance regulations for commercial pesticides
GB/T 1605-2001	Sampling method for commercial pesticides
GB 3796	General rule for packing of pesticides

3 Requirements

3.1 Composition and Appearance:

This product should be a brown-yellow to orange crystal powder without any visible foreign ingredients or added modifier.

3.2 Pendimethalin technical should meet the requirements set out in Table 1.

**Table 1 – Control Items and Guidelines
for Pendimethalin Technical**

Item	Guideline
Pendimethalin mass fraction /% •	95.0
Water /% •	0.5
pH range	4.0 to 8.0
Acetone insoluble /% ^{a)} •	0.5
a) Under normal production conditions, insoluble-acetone should be tested at least every 3 months.	

4 Test Method

4.1 Sampling

Sampling should be carried out in line with the Commodity Pesticide Technical Sampling Method set out in GB/T 1605-2001. A random sampling method should be used to determine which package shall be used as the sample, and the final sample quantity should not be less than 100g.

4.2 Identification Test

4.2.1 Liquid chromatography: this identification test can be carried out at the same time as the pendimethalin content test. Under the same chromatographic operation conditions, the relative difference between the retention time of certain chromatographic peaks in the sample solution and the retention time of the chromatographic peak of pendimethalin in the standard solution should be less than 1.5%.

4.2.2 Gas chromatography: this identification test can be carried out at the same time as the pendimethalin content test. Under the same chromatographic operating conditions, the relative difference between the retention time of certain chromatographic peaks in the sample solution and the retention time of the chromatographic peak of pendimethalin in the standard solution should be less than 1.5%.

4.3 Pendimethalin mass fraction test

4.3.1 Liquid chromatography (Arbitration method)

4.3.1.1 Summary of Method

Dissolve the sample in acetonitrile solution. Apply acetonitrile + water as the mobile phase; use a stainless steel column with ODS (C18) as filler and an ultraviolet detector. Conduct the reversed phase chromatographic isolation and measurement of pendimethalin in the sample.

4.3.1.2 Reagent and Solution

Acetonitrile: chromatographic pure;
Water: secondary distilled water;
Standard pendimethalin: known content • 99.0%.

4.3.1.3 Instruments

Liquid chromatography: variable ultraviolet wavelength detector and quantitative sampling valve;
Chromatographic data processor or chromatography workstation;
Chromatographic column: 200 mm × 4.6mm (i.d.) stainless steel column with Hypersil ODS 5 • m filler (or other reversed phase columns with the same column effect);
Filter: membrane pore diameter about 0.45 • m;
Quantitative sampling tube: 5 • l;
Micro dosing unit: 50 • l;
Ultrasonic cleaner.

4.3.1.4 Liquid chromatography operating condition

Mobile phase: • (acetonitrile: water) = 60:40;
Mobile phase flow volume: 1.0 ml/min;
Column temperature: room temperature (the temperature fluctuation should not exceed 2 •);
Detecting wavelength: 240 nm;
Sampling volume: 5 • l;
Retention time: 13.5 min for pendimethalin.

The above are typical operating parameters. The given operating parameters may be adjusted appropriately according to the characteristics of different instruments, so as to obtain the best result. A typical high performance liquid chromatogram of pendimethalin technical is shown in Figure 1.



1 – Pendimethalin

Figure 1 - High performance liquid chromatogram of pendimethalin technical

4.3.1.5 Test Procedure

4.3.1.5.1 Preparation of standard solution

Take 0.1g standard pendimethalin (accurate to 0.0002 g) and place in a 50ml measuring flask then dissolve with acetonitrile, diluting the solution to the scale, and shake evenly. Using a pipette, take 5ml of the above solution and place in another 50ml measuring flask. Dilute to the scale by mobile phase and shake evenly.

4.3.1.5.2 Preparation of sample solution

Take a 0.1g sample of pendimethalin (accurate to 0.0002 g) and place in a 50ml measuring flask. Dissolve with acetonitrile, dilute the solution to scale and shake evenly. Using a pipette, take 5ml of the above solution and place in another 50ml measuring flask. Dilute to scale by mobile phase and shake evenly.

4.3.1.5.3 Test

Under the above operating conditions and after the baseline of the instrument is stabilised, continuously syringe several doses of the standard solution, calculating the repetition of the corresponding response value of each dose until the pendimethalin peak area's variation of two adjacent doses is less than 1.2% and then measure in sequence the standard solution, sample solution, sample solution and standard solution.

4.3.1.6 Calculation

The mass fraction ω_1 (%) of pendimethalin in the sample is calculated using formula (1):

$$\omega_1 = \frac{A_2 \cdot m_1 \cdot \omega}{A_1 \cdot m_2} \dots\dots\dots (1)$$

In the formula:

A_1 - average peak area of pendimethalin in the standard solution;

A_2 - average peak area of pendimethalin in the sample solution,

m_1 – mass of the standard pendimethalin, unit expressed in grams (g);

m_2 - mass of the sample, unit expressed in grams (g);

w – mass fraction of the standard pendimethalin, %;

4.3.1.7 Tolerance

The difference between the results from two parallel tests should not exceed 1.0%. Take the calculated average as the test result.

4.3.2 Gas chromatography

4.3.2.1 Summary of Method

Dissolve the sample in chloroform solution. Apply dipentyl phthalate as the internal standard; use a glass chromatographic column with 5% OV-101/Chromosorb W AW DMCS, 180 • m to 250 • m as filler and a hydrogen flame ionisation detector. Conduct the gas chromatographic isolation and measurement of pendimethalin in the sample.

4.3.2.2 Instrument

Gas chromatography: hydrogen flame ionisation detector;

Chromatographic data processor or chromatography workstation;

Chromatographic column: 1m × 3.2mm (i.d.) glass column with 5% OV-101/Chromosorb W AW DMCS, 180 • m to 250 • m filler;

Micro dosing unit: 10 • l.

4.3.2.3 Reagent and Solution

Chloroform;

Pendimethalin sample: known content • 99.0%;

Dipentyl phthalate: without any impurity to interfere the analysis;

Internal standard solution: take 5g dipentyl phthalate and place in a 1,000ml measuring flask. Dissolve in chloroform, dilute the solution to scale and shake evenly.

4.3.2.4 Gas chromatography operating condition

Temperature (•): column temperature 175, gas chamber 200, detector chamber 200;

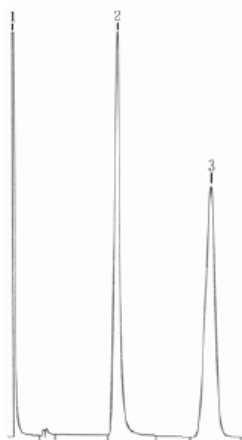
Gas flow (ml / min): carrier gas (N₂) 40, hydrogen 35, air 350;

Sampling volume (• l): 0.5;

Retention time (min): about 9.4 for pendimethalin, about 14.0 for the internal standard substance.

The above are typical operating parameters. The given operating parameters can be adjusted appropriately according to characteristics of different

instruments, so as to obtain the best result. A typical gas chromatogram of pendimethalin technical and internal standard substance is shown in Figure 2.



- 1 - Solvent;
- 2 - Pendimethalin;
- 3 – Internal standard substance (dipentyl phthalate).

Figure 2 - Gas chromatogram of pendimethalin technical and internal standard substance

4.3.2.5 Test Procedure

4.3.2.5.1 Preparation of standard solution

Take 0.1g standard pendimethalin (accurate to 0.0002 g) and place in a 10ml bottle with a plug. Using a pipette, add 10ml internal standard solution and shake evenly, so as to dissolve it.

4.3.2.5.2 Preparation of sample solution

Take a 0.1g sample pendimethalin (accurate to 0.0002 g) and place in a 10ml bottle with a plug. Using the same pipette as indicated in 4.3.2.5.1, add 10ml internal standard solution and shake evenly.

4.3.2.5.3 Test

Under the above operating conditions and after the baseline of the instrument is stabilised, continuously syringe several doses of the standard solution, calculating the repetition of the peak area ratio of the pendimethalin and the internal standard substance; when the variation of the peak area ratio of the pendimethalin and the internal standard substance of two adjacent doses becomes less than 1.2%, measure in sequence the standard solution, sample solution, sample solution and standard solution.

4.3.2.6 Calculation

The mass fraction \bullet_1 (%) of pendimethalin in the sample is calculated using formula (2):

$$\omega_1' = \frac{\gamma_2 \cdot m_1 \cdot \omega}{\gamma_1 \cdot m_2} \dots \dots \dots (3)$$

- ₁ - average ratio between the pendimethalin and internal standard peak areas in the standard solution;
- ₂ - average ratio between the pendimethalin and internal standard peak areas in the sample solution;
- m₁ – mass of the standard pendimethalin, unit expressed in grams (g);
- m₂ – mass of the sample, unit expressed in grams (g);
- - mass fraction of the standard pendimethalin, %;

4.3.2.7 Tolerance

The difference between the results from two parallel tests should not exceed 1.0%. Take the calculated average as the test result.

4.4 Water content test

The water content test should be carried out according to the Karl Fisher method as set out in GB/T 1600.

4.5 pH value test

Take a sample according to the requirements stipulated in GB / T 1601. Dissolve with a solution of • (acetone: water) = 65:35 and take measurement using the pH value test method for pesticides in GB / T 1601.

4.6 Acetone insoluble test

The acetone insoluble test should be carried out as according to GB/T 19138.

4.7 Product inspection and acceptance

Product inspection and acceptance should conform to the provisions set out in GB/T 1604. Limiting values should be processed using the rounding off comparison method.

5 Marking, labelling, packaging and transportation

5.1 The marking, labelling and packaging of pendimethalin technical should conform to the provisions set out in GB 3796.

5.2 Pendimethalin technical should be packed in plastic barrels or steel barrels with plastic lining, without direct contact with the metal. The net content of each barrel should be 25kg, 50kg or 200kg.

5.3 Other forms of packaging may be used in accordance with the requirements of the customer or contrasting orders, but should in all cases meet the provisions set out in GB 3796.

- 5.4 Pendimethalin technical packs should be stored in well-ventilated and dry warehouses.
- 5.5 During transportation, strict precautions should be taken against dampness and direct sunlight; the product should not be placed together with food, seeds or feedstuff, and contact with the skin and eyes and inhaling into the mouth and nose should be avoided.
- 5.6 **Safety:** pendimethalin falls into the category mildly toxic. It is toxic when swallowed or inhaled and can be infiltrated through the skin. Protective goggles, rubber gloves and other necessary protective clothing should be worn when dealing with this product. In case of contact with the skin or eyes it should be rinsed immediately with plenty of water; in case it is eaten by mistake, the person affected should be sent to hospital immediately for emergency treatment.
- 5.7 **Acceptance period:** the acceptance period for pendimethalin technical is one month. Quality acceptance should be completed within one month from delivery and each guideline should meet the requirements of this standard.
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