II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE

of 25 April 1984

adapting to technical progress for the sixth time Council Directive 67/548/EEC on the approximation of laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances

(84/449/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 67/548/EEC of 27 June 1967 on the approximation of laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances (1), as amended for the sixth time by Council Directive 79/831/EEC (2), and in particular Articles 19, 20 and 21 thereof,

Whereas Article 3 (1) of Directive 79/831/EEC provides that the physico-chemical properties, toxicity and ecotoxicity of the substances and preparations shall be determined according to the methods specified in Annex V;

Whereas Article 19 of Directive 79/831/EEC of 18 September 1979 provides that Annex V comes under the procedure, involving the Committee for Adaptation to Technical Progress; whereas due regard should be given, in particular, to any methods recognized and recommended by competent international organizations;

Whereas the provisions of this Directive are in accordance with the opinion of the Committee for the Adaptation to Technical Progress of the Directives on the Removal of Technical Barriers to Trade in Dangerous Substances and Preparations,

HAS ADOPTED THIS DIRECTIVE:

Article 1

The text of Annex V to Directive 67/548/EEC is hereby replaced by the text of the Annex to this Directive.

Article 2

Member States shall adopt and publish before 1 July 1985 the measures needed to comply with this Directive and immediately inform the Commission thereof. They shall apply these measures by 1 July 1986 at the latest.

Article 3

This Directive is adressed to the Member States.

Done at Brussels, 25 April 1984.

For the Commission

Karl-Heinz NARJES

Member of the Commission

(1) OJ No L 196, 16. 8. 1967, p. 1.
(2) OJ No L 259, 15. 10. 1979, p. 10.

ANNEX

The Annex sets out test methods for the determination of physicochemical, toxicological and ecotoxicological properties listed in Annexes VII and VIII to Directive 79/831/EEC. The methods are based on those recognized and recommended by competent international bodies (in particular OECD).

When such methods were not available, national standards or scientific consensus methods have been adopted. Generally, tests should be performed with the substance as marketed. Attention should be given to the possible influence of impurities on the test results.

When the methods of this Annex are inappropriate for the investigation of a certain property, the notifier must justify the alternate method used.

Animal tests and studies shall be conducted in accordance with national regulations and shall take into account humane principles and international developments in the field of animal welfare.

Among equivalent testing methods, the method using the minimum number of animals is chosen.

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PART A: METHODS FOR THE DETERMINATION OF PHYSICO-CHEMICAL PROPERTIES

A. 1. MELTING POINT/MELTING RANGE

1. METHOD

The methods described are based on the OECD Test Guideline (1).

1.1. Introduction

The methods and devices described are to be applied for the determination of the melting point of chemical substances, without any restriction in respect to their degree of purity.

The selection of the method is dependent on the nature of the substance to be tested.

In consequence the limiting factor will be according to whether the substance can be pulverized easily, with difficulty, or not at all.

For some substances, the determination of the freezing or solidification point is more appropriate and the standards for these determinations have also been included in the guideline.

1.2. Definitions and units

The melting point is defined as the temperature at which the phase transition from solid to liquid state at normal atmospheric pressure takes place.

This temperature ideally corresponds to the temperature at the solidification point or the freezing point.

As the phase transition of many substances takes place over a large temperature range, it is often described as the melting range.

Conversion of units (K to °C):

$t = T - 273{,}15$

with t in °C, T in K.

1.3. Reference substances

Reference substances do not need to be employed in all cases when investigating a new substance. This should primarily serve to calibrate the method from time to time and to allow a comparison of results when another method is applied.

Some calibration substances are listed in the references (2).

1.4. Principle of the test method

The temperature (temperature range) of the phase transition from the solid to the liquid state is determined. In practice while heating a sample of the test substance at atmospheric pressure the temperatures of the initial melting and the final stage of melting are determined. Three types of methods are described, namely capillary method, hot stages method and freezing point determinations.

1.4.1. *Capillary method*

1.4.1.1. Melting point devices with liquid bath

A small amount of the finely ground substance is charged in a capillary tube and packed tightly. The tube is heated together with a thermometer and the temperature rise is adjusted to less than about 1 K/min during the actual melting. The initial and final melting temperatures are determined.

1.4.1.2. Metal block

As described under 1.4.1.1, except that the capillary tube and the thermometer are situated in a heated metal block, and can be observed through holes in the block.

1.4.1.3. Photocell detection

The sample in the capillary tube is heated automatically in a metal cylinder. A beam of light is directed through the substance, by way of a hole in the cylinder, to a precisely calibrated photocell. The optical properties of most substances change from opaque to transparent when they are melting. The intensity of light reaching the photocell increases and sends a stop signal to the digital indicator reading out the temperature of a platinum resistance thermometer located in the heat chamber. This method is not suitable for some highly coloured substances.

1.4.2. *Hot stages*

1.4.2.1. Kofler hot bar

The Kofler hot bar uses two pieces of metal of different thermal conductivity, heated electrically, with the bar designed so that the temperature gradient is almost linear along its length. The temperature of the hot bar can range from 283 to 543 K with a special temperature-reading device including a runner with a pointer and tab designed for the specific bar. In order to determine a melting point, the substance is laid in a thin layer directly on the surface of the hot bar. In a few seconds a sharp dividing line between the fluid and solid phase develops. The temperature at the dividing line is read by adjusting the pointer to rest at the line.

1.4.2.2. Melt microscope

Several microscope hot stages are in use for the determination of melting points with very small quantities of material. In most of the hot stages the temperature is measured with a sensitive thermocouple but sometimes mercury thermometers are also used. A typical microscope hot stage melting point apparatus has a heating chamber which contains a metal plate upon which the sample is placed on a slide. The centre of the metal plate contains a hole permitting the entrance of light from the illuminating mirror of the microscope. When in use, the chamber is closed by a glass plate to cut off air from the working area.

The heating of the sample is regulated by a rheostat. For very precise measurements, polarized light may be used for optically anisotropic substances.

1.4.2.3. Meniscus method

This method is specifically used for polyamides.

Determination of the temperature at which the displacement of a meniscus of silicone oil, enclosed between a hot stage and a cover-glass supported by the polyamide test specimen, is observed visually.