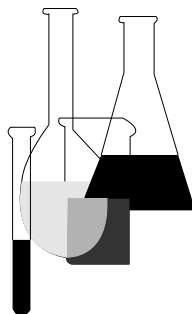




Product Properties Test Guidelines

OPPTS 830.7100 Viscosity



INTRODUCTION

This guideline is one of a series of test guidelines that have been developed by the Office of Prevention, Pesticides and Toxic Substances, United States Environmental Protection Agency for use in the testing of pesticides and toxic substances, and the development of test data that must be submitted to the Agency for review under Federal regulations.

The Office of Prevention, Pesticides and Toxic Substances (OPPTS) has developed this guideline through a process of harmonization that blended the testing guidance and requirements that existed in the Office of Pollution Prevention and Toxics (OPPT) and appeared in Title 40, Chapter I, Subchapter R of the Code of Federal Regulations (CFR), the Office of Pesticide Programs (OPP) which appeared in publications of the National Technical Information Service (NTIS) and the guidelines published by the Organization for Economic Cooperation and Development (OECD).

The purpose of harmonizing these guidelines into a single set of OPPTS guidelines is to minimize variations among the testing procedures that must be performed to meet the data requirements of the U. S. Environmental Protection Agency under the Toxic Substances Control Act (15 U.S.C. 2601) and the Federal Insecticide, Fungicide and Rodenticide Act (7 U.S.C. 136, *et seq.*).

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OPPTS 830.7100 Viscosity.

(a) **Scope**—(1) **Applicability.** This guideline is intended to meet testing requirements of both the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (7 U.S.C. 136, *et seq.*) and the Toxic Substances Control Act (TSCA) (15 U.S.C. 2601).

(2) **Background.** The source materials used in developing this harmonized OPPTS test guideline are OPP guideline 63–18 Viscosity (Pesticide Assessment Guidelines, Subdivision D: Product Chemistry, EPA Report 540/9–82–018, October 1982) and OECD guideline 114 Viscosity of Liquids.

(b) **Introductory information**—(1) **Prerequisite.** Density, for the rolling ball viscometer method.

(2) **Guidance information.** (i) Melting point/melting range.

(ii) Boiling point/boiling range.

(3) **Coefficient of variation.** Coefficients of variation appeared to be dependent on the chemicals tested. They are calculated from the mean values given by the participants of the OECD Laboratory Intercomparison Testing, part I, 1979, and their range is from 0.004 to 0.09, without referring to different methods applicable.

(4) **Qualifying statement.** The five methods listed are appropriate in principle for the investigation of Newtonian liquids. The measurement of non-Newtonian liquids is only possible with the rotational viscometer.

(5) **Additional comments.** These methods are capable of greater precision than is likely to be required for environmental assessment. The ranges are shown in the following table 1:

Table 1.—Measurement of Precision

Methods	Ranges
Capillary viscometer	0.5 mPa s to 10 ⁵ mPa s
Flow cup	8 mPa s to 700 mPa s
Rotational viscometer	10 mPa s to 10 ⁹ mPa s
Rolling ball viscometer	0.5 mPa s to 10 ⁵ mPa s
Drawing ball viscometer	0.5 mpa s to 10 ⁷ mpa s

(6) **Standard documents.** (i) The majority of the methods described is based on both international and national standards. The appropriate ISO Standards describing the concerned methods are cited within the text of this test guideline (see paragraph (f)(3) of this guideline).

(ii) A good summary of measuring instruments and measuring methods can be found under paragraph (f)(2) of this guideline.

(c) **Method**—(1) **Introduction, purpose, scope, relevance, application and limits of test.** (i) The viscosity of fluids is environmentally relevant owing to the penetration of fluids into the soil and the harmful effect on the ground water which may thus possibly be caused. From the point of view of this problem, surface tension as well as questions of wettability, miscibility or solubility play, a part in addition to viscosity, so that it is usually not sufficient to consider viscosity alone.

(ii) There is no environmentally relevant limit in the direction of low viscosities. The lower the viscosity the more easily a fluid seeps into the soil. The lowest dynamic viscosity of liquids occurring at room temperature is approximately 0.2 mPa s, that is to say one-fifth of the viscosity of water at 20 °C. A limit in the direction of high viscosities cannot be precisely quoted. Dynamic viscosities above approximately 10^7 mPa s are so high that penetration into the soil is no longer probable.

(iii) In the case of substances which have a yield value (pastes, ointments), the substance may still not penetrate into the soil, although the dynamic viscosity may be low after the yield value has been exceeded. If the substance is soluble in water or can be emulsified, environmental damage may occur despite the existence of a flow limit.

(2) **Definitions and units.** *Viscosity* is the property of a fluid substance of absorbing a stress during deformation which depends on the rate of the deformation. Similarly, the stress can be regarded as the cause which brings about a deformation rate.

(i) The shear stress, τ , and the shear rate, D , are related by the equation

$$\tau = \eta D$$

η is defined as the dynamic viscosity.

(ii) For Newtonian liquids, the viscosity is constant at all shear rates and depends only on the variables pressure and temperature.

(iii) For non-Newtonian liquids, the viscosity will vary with shear rate.

(iv) If the viscosity is measured with capillary viscometers without applied pressure, the measured quantity obtained is the ratio of dynamic viscosity to density, the so-called kinematic viscosity, ν .

(v) The SI unit of dynamic viscosity is the Pascal second, Pa s. For practical use a submultiple is more convenient; 1 mPa s = 10^{-3} Pa s (one centipoise [cP] in the obsolete cgs-system).

(vi) The SI unit of kinematic viscosity is the square meter per second, m^2/s . The normal sub unit derived from this is the square millimeter per

second, $\text{mm}^2/\text{s} = 10^{-6} \text{ m}^2/\text{s}$. (1 $\text{mm}^2/\text{s} = 1$ centistoke [cSt] in the obsolete cgs-system.)

(3) **Reference substances.** (i) The reference substances need not be employed in all cases when investigating a new substance. They are provided primarily so that calibration of the method may be performed from time to time and to offer the chance to compare the results when another method is applied.

(ii) The following list of reference substances has been extracted from that recommended by the IUPAC.

Table 2.—Recommended Reference Substances for Viscosity Measurements

Chemical Name	Certified Value and Accuracy	*COM022*Source ¹	Remarks
Series of mineral oils (hydrocarbons, partly natural, partly synthetic products).	1 to 27,000 mPa s (1.25 to 30,000 mm^2/sec at 20 °C. Uncertainty, $\pm 0.2\%$, above 4,000 mPa s $\pm 0.3\%$)	C	Newtonian liquids, determined by capillary viscometers with suspended level (Ubbelohde). Data also for other temperatures between 20 and 100 °C.
Type OS 2.5–2,000 (series of 10 liquids).	Certified for viscosity in mPa s and kinematic viscosity in mm^2/sec . Range for viscosity at 20 °C from 2 to 8,000	E	
Type 60H	60,000 mm^2/sec at 20 °C	E	
Type 200 H	200,000 mm^2/sec at 20 °C	E	
Mineral oil	11 to 1,000 mPa s $\pm 0.1\%$ at 20 °C	D	Newtonian liquid. Certified also for density and kinematic viscosity
Mineral oil	10^3 to 10^4 mPa s $\pm 0.5\%$ at 20 °C	D	Newtonian liquid. Certified also for density and kinematic viscosity.
Polyisobutylenes	10^4 to 10^5 mPa s $\pm 1.5\%$ at 20 °C		Newtonian liquid. Rotating cylinder viscometer method used.
Series of 11 mineral oils	Certified for viscosity in mPa s at 20 °C. Range from $1.503 \pm 0.1\%$ to $1,729 \pm 0.2\%$	G	Certified also for kinematic viscosity and density. Data also at 50 °C and 80 °C.
Series of 7 polyisobutylenes	Certified for viscosity in mPa s at 20 °C. Range from $4,170 \pm 1.3\%$ to $589 \times \pm 1.0\%$	G	Data also at 50, 80, and 100 °C.

¹ Units are given as reported by issuing laboratory. The letter references in column three indicating the countries reporting represent the following countries:

- C: Germany: The Physikalische-Technische Bundesanstalt, 33 Braunschweig, Bundesallee 100, Federal Republic of Germany.
- D: Hungary: National Office of Measures Nemerolgyi ut 37–39 sz. Budapest XII, Hungary.
- E: Japan: National Chemical Laboratory for Industry, Ministry of International Trade & Industry, 1–1 Honmachi, Shibuya-ku, Tokyo 151, Japan.
- G: Poland: Division of Physico-Chemical Metrology, National Board for Quality Control and Measures – 2, Elektoalna St., Warsaw, Poland.

(4) **Principle of the test method.** Viscosity measurements are carried out predominantly according to three measurement principles:

(i) The flow under gravity through a capillary, (capillary viscometer or flow cup).

(ii) Shearing of the fluid between concentric cylinders, coneplate and parallel plate (rotational viscometer).

(iii) (A) Dynamic viscosity can be measured by movement of a ball in a vertical or inclined liquid-filled cylindrical tube (e.g. a rolling ball viscometer by Hoppler, drawing ball viscometer, etc.)

(B) With the Hoppler viscometer the density must be known in order to calculate the dynamic viscosity.

(5) **Quality criteria.** The various methods of determining viscosity of liquids are compared as to application, measuring range and standardizability in the following table.

Table 3.—Comparison of the Methods

Method of Measurement	Viscosity		Measuring Range (mPa s or mm ² /s)	Standardization	Temperature Constancy required (°C)
	Dynamic (mPa s)	Kinematic (mm ² /s)			
Capillary viscometer		x	0.5–10 ⁵	ISO 3104 and 3105 ...	±0.1
Flow cup		x	8–700	ISO 3104 and 3105 ..	±0.5
Rotational viscometer	x		10–10 ⁹	ISO 3218.2	±0.2
Rolling ball viscometer	x		0.5–10 ⁵	no international standards, see DIN 53015.	±0.1
Drawing ball viscometer	x		0.5–10 ⁷	no international standards, see DIN 52007, part 2 (draft).	±0.1

(i) Possibility of standardization. See table 3 in paragraph (c)(5) of this guideline.

(ii) Possibility of automation. Yes.

(d) **Description of the test method—(1) Preparations-apparatus.**

(i) Capillary viscometer designs are described in: ISO 3104; ISO 3105; DIN 51550; DIN 51550; DIN 51562 part 1; DIN 51561; DIN 51366, DIN 51372; DIN 53177; ASTM D–1200–70; ASTM D–2393; ASTM D–914 part 25 to 37; ASTM D–88–56. Refer to paragraph (f) of this guideline for more information.

(ii) The standardization of rotational viscometers covers, with few exceptions, only general specifications concerning the flow pattern, range of shearing stresses to be used and velocity gradient as well as specification relating to specific substances. ISO 3219–1977; DIN 51398; DIN 51377; DIN 53214; DIN 53019 part 1; DIN 53229; DIN 52312 part 2; DIN 53921; ASTM D–562–55; ASTM D–3346–74; ASTM D-2983. Refer to paragraph (f) of this guideline for more information.

(iii) Forced ball viscometers are only standardized in such national standards as: DIN 53015, DIN 52007 part 2 and ASTM D–914 part 25 to part 37. Refer to paragraph (f) of this guideline for more information.

(2) **Test conditions.** Each determination of viscosity must be accompanied by the temperature at which the measurement was made. The determination should preferably be made at a temperature of 20 °C and at one other temperature approximately 20 °C higher (see table 1 in paragraph (c)(3)(ii) of this guideline for temperature control limits). At least two determinations should be made at each temperature.

(3) **Performance of the tests.** The measurement is carried out according to the specifications in the respective standards.

(e) **Data and reporting—(1) Treatment of the results.** (i) The evaluation of the viscosity measurement is to be carried out according to the standards in the case of capillary and forced ball viscometers. In the case of rotational viscometers, the specification of a viscosity is appropriate only for Newtonian fluids. For non-Newtonian fluids the results obtained are preferred in the form of flow curves which must be interpreted, assuming the validity of various laws of flow.

(ii) The uncertainties in the measurement are quoted in the standards for capillary, forced ball and rotational viscometers.

(2) **Test report (detailed conduct of test and evaluation).** The test report is to be drafted in accordance with the specifications in the standards. It must include individual and mean values at each temperature. Any variation from the standard method must be described in detail.

(f) **References.** The following references should be consulted for additional background material on this test guideline.

(1) Physicochemical Measurements: Catalogue of Reference Materials from National Laboratories, in: *Pure and Applied Chemistry*, IUPAC, vol. 48, pp. 513-514, Pergamon Press (1976).

(2) Wazer, W. et al. *Viscosity and Flow Measurement, Laboratory Handbook of Rheology*, Inst. Publ. New York - London (1963).

(3) International Organization for Standards, ISO. American National Standards Institute, Sales Department, 1430 Broadway, New York, NY 10018. Book of Standards (latest edition).

(4) Das 1st Norm., DIN. American National Standards Institute, Sales Department, 1430 Broadway, New York, NY 10018. Book of Standards (latest edition).

(5) American Society for Testing and Materials, ASTM, 1916 Race St., Philadelphia PA 19103. Annual Book of ASTM Standards (latest edition).

(6) *Organization for Economic Cooperation and Development*, Guidelines for The Testing of Chemicals, OECD 114 Viscosity of Liquids, OECD, Paris, France.