

Summary of comments to the 1st Draft International Recommendation
“Newtonian Viscosity Standard Liquids for the Calibration and Verification of Viscometers”
TC17/SC5 Secretariat: Russian Federation

<i>Document Paragraph</i>	<i>Country</i>	<i>COMMENTS</i>	<i>Response</i>
General	USA	The document discusses requirements on the liquids in detail, but the requirements on the instruments and procedures are said to be discussed only when they deviate from ISO 3104 and ISO 3105. This is appropriate if indeed there are no duplicated details concerning the instruments and procedures.	
General	USA	References should be numbered and listed in the order they are referred to in the text. Text references should be to the reference number. Many references are too old and should be updated. Reference in OIML documents should be to international standards or documents, reference to national or regional standards should be avoided, unless they contain unique information necessary for the recommendation not available in international standards. This applies especially to WECC document 19, where a reference to the GUM would be adequate. Numbering system	accepted
General	USA	The discussion about uncertainty in Appendix 4 is not balanced. The general discussion in the first part is incomplete, and it would be better to simply to refer the reader to the Guide to the expression of uncertainty in measurement and shorter summaries such as EA-4/02 (1999). The example is hard to follow. Using subheadings and tables would help. An equation that adds all of the contributions would help. The use of 20% for "undetected systematic errors" is not supported. Why not 200%?	accepted
General	Poland	According to ISO 31-0:1992, it is required to write “mPa · s” instead of “mPa·s” or “mPas” and in all numbers there are required spaces (e.g. there is $1 \cdot 10^5$, it should be $1 \cdot 10^5$), e.t.c.	accepted
General	Bulgaria	We should like to recommend the publication EA-4/02 “Uncertainty expression of Measurement in calibration” to replace WECC-Doc 19 when the uncertainty of measurement is calculated	accepted

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1 Scope and field of application	USA	<p>Please use terminology of D17, primary and secondary standards, instead of first and second order, to avoid confusion. Perhaps primary and secondary standards should be defined in this context in the terminology section. We would like a definition of first and second order if they are necessary to be used at all.</p> <p>Questions for clarification: Is a first order viscometer calibrated from the primary viscosity standard (water at 20 °C and associated step-up procedure. Is a second order viscometer calibrated from direct comparison of a first order viscometer using transfer liquids? Is this what we might determine as a primary standard viscometer and a secondary standard viscometer?</p>	Accepted partly D17 classifies liquids as “reference” and “working”
2 nd paragraph	USA	"as traceable to National Standards of Viscosity national standards of viscosity according to ISO 9001 17025 (1999) subclause 5.6.2.1.1." ISO 17025 is more appropriate for this activity.	accepted

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		<p>VSS may be better and more intuitive than SSp. And 1st and 2nd in place of 1-th and 2-th.</p> <p>According to the CIPM SI Brochure in 1 m²/s the 1 is unnecessary. Consider adding density.</p> <p>Range of kinematic viscosity and temperature is ok. Range of pressure questionable for kinematic viscometers. The relative deviation table looks ok, as does the temperature requirements and timing requirements. We question the 100-fold pressure range. The text that follows in this document is limited to ISO 3104, ISO 3105 and ASTM D 2162 viscometers, all of which are at atmospheric pressure. D 2162 does note and cite the buoyancy applicable to the step-up range. However, except for the step-up procedure, the buoyancy correction is frequently considered not to add significantly to the final uncertainty. In the second paragraph reference is made to D17, this Recommendation should be complete and contain all the relevant information. Please include D17 specifications. Viscosity should be the first column of the first table. Please number the tables according to the OIML directives. Please consider expressing the relative deviation in percent, but in any case, be consistent throughout. Table interpretation; does it mean if there are four steps in going from 1 to 50 mm² s⁻¹, the relative deviation of the fourth step would have a relative deviation of 4 x 0.05%, or 0.2%?</p> <p>Section is well thought out, in "Requirements d) to j)" There is no j). proofed</p> <p>The facilities necessary for the preparation of the final mixture depend on the amount of liquid (mostly usually between 5 and 50 l) Typical mixing times may be between 24 and 100 h. [Is such a long mixing time necessary for only 5 liters?]</p>	
Clause 2.2	The Netherlands	Replace "1-th" by 1 st	accepted

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Clause 4	Slovakia	There are stated specification for temperature control (temperature change during a measurement = $<0,003^{\circ}\text{C}$ and $<0,007^{\circ}\text{C}$. On the other hand in point "9.1 as suitable thermometer for temperature control a mercury in glass thermometer with $0,01^{\circ}\text{C}$ scale interval is stated. I consider that these two requirements are in contradiction.	accepted
	Japan	1) The range of dynamic viscosity is not consistent with OIML-DI No.17. 2) On the standard specimens, the reason for up to 10 MPa is not clear. 3) “From the kinematic viscosity values” instead of “From the viscosity values”. 4) On the standard viscometer, descriptions of concrete information are necessary. 5) The values of temperature change and temperature gradient of the thermostatic bath should be also changed by the temperature range (between -40 and $+150$ □). In practice, a temperature change during a measurement of 1-st order SSp (≤ 0.003 □) is difficult to realized at various temperatures (usually between of -40 and $+150$ □).	accepted
Clause 5, p 6	Poland	It should be "behaviour" instead "behavior"	accepted
	Netherlands	According to our opinion: "The fulfillment of requirements a) to c)....given in clause 7." should be replaced by"..... clause 8." And "Requirements d) to J) may be" should be replaced by "Requirements d) to i) may be..."	accepted partly
	Japan	"Requirements d) to i) instead Requirements d) to j)"	accepted
	Bulgaria	Should be replaced by "viscosity, irreversible during thermal stability testing"	accepted
Clause 6	Poland	It should be " containers" instead "containments"	accepted
Clause 8	Japan	8.1 $7 \cdot 10^{-3}$ shall be changed to $4 \cdot 10^{-3}$. 8.2 Information on the criterion about the influence of duration of measurement shall be added.	accepted

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	Bulgaria	8.1 The term "aging coefficient" is not clearly defined. The last sentence of the paragraph should be more precise 8.2 c) There is no assessment of the measurement in reference to this point. It is not quite clear what is the influence of the specific measurement over the thermal stability	accepted partly
Clause 9	Netherlands	9.1 Replace "1-th" by "1 st " and replace "2-th" by "2 nd " Replace " $K_2Cr_2O_7$ " by " $K_2Cr_2O_7$ " 9.4 In both clauses 9.4 and 10 it is mentioned that the temperature coefficient U_γ is stated in the calibration certificate, but this is missing in Appendix 5.	accepted
	Japan	9.1 Isn't it necessary for evaluating the effect of the change in the kinematic viscosity of the order of SSp on the value of Equation (1) 9.2 More precise information about the kinematic energy correction ("Hagenbach'-correction) shall be added. 9.3 "pyknometers" instead "pycnometers", An oscillation U-tube density meter shall be added as an instrument to measure the density for a viscosity of less than 700 mPa s.	accepted
	Bulgaria	9.4 The second part of the sentence should be abolished.	Don't understand

	USA	<p>9.1 <u>Standard viscometers</u></p> <p>for the calibration of 1-th order SSs: [Calibration of 1st order VSS] [Use] two primary or secondary standard viscometers of approximately the same viscometer constant; [.] [The ($k=2$)] uncertainty of viscometer constant [.] including the uncertainty of the kinetic energy correction [.] between [must not exceed] 0.1% at low and 0.8% at high values of the constant.</p> <p>for the calibration of 2-th order SSs: [Calibration of 2nd order VSS] [Use] two reference standard viscometers of approximately the same viscometer constant; [.] [The ($k=2$)] uncertainty of viscometer constant [.] including the uncertainty of the kinetic energy correction [.] [must not exceed] 0.2% at low and 1% at high values of the constant.</p> <p>Suitable thermometers are calibrated platinum-resistance thermometers or mercury in glass thermometers with [a] 0.01°C scale interval. It may be necessary to apply corrections according to the calibration certificate. The depth of immersion shall be the same during calibration and use. [Mercury-in-glass thermometers with a scale interval of 0.01 °C are unusual. It's better to state the required uncertainty of the thermometer and not the technology employed.]</p> <p>If both viscosity determinations differ by more than the repeatability of the flow-time measurement, the uncertainty shall be increased accordingly. The relative difference shall not exceed $0.7 \cdot 10^{-3}$. [Move this paragraph to the end of the Measurement section.]</p> <p><u>Measurement</u></p> <p>Handling viscometers like filling and cleaning shall be done [The handling of viscometers, such as filling and cleaning, must be done] in accordance with ISO 3104 and 3105.</p>	<p>accepted</p> <p style="text-align: right;">6 of 9</p>
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		<p>9.1 When cleaning the standard viscometers with sulphochromic acid (saturated aqueous solution of $K_2Cr_2O_7$ [$K_2Cr_2O_7$] mixed with H_2SO_4 in a ratio of about 1:1) the temperature should not exceed 30 °C and the cleaning time 2 h. This acid shall only be used [only] for cleaning heavily contaminated viscometers.</p>	accepted
		<p>9.2 The viscometers should be designed such that the Hagenbach, or kinetic energy correction, or any other correction should be minimized. In the context of this document, the g/g' would be equal to unity by definition, since all work would be done at the same location. The MS thesis by Bell (Cannon) showed that at low Reynolds numbers, the kinetic correction coefficient, “m”, decreases toward zero, rather than approach a value of one regardless the shape of the entrance and exit geometries at very low Reynolds numbers. The previous document implied a capillary length of 300 to 400 mm. This version suggests 90 mm for standard size capillary viscometers. Does this apply to 1st and / or 2nd order viscometers? Section might be more explicit.</p>	accepted
		<p>9.3 A suitable method for measurement of density is the use of two calibrated pycnometers of typically 25 or 50 ml of volume. The relative uncertainty of mass determination taking into account the air buoyancy correction can be kept below $2 \cdot 10^{-5}$ using routine laboratory methods. [Routine laboratory methods that correct for air buoyancy can keep the relative uncertainty of mass determination below $2 \cdot 10^{-5}$.] For liquids that meet the stability requirements listed in clause 8, the density is not significantly changed by ageing. Therefore, it is only necessary to determine the density of each batch of reference liquid [only] once.</p>	Accepted
		<p>9.4 Note: From the [A] great number of viscosity-temperature equations [are available. The] the use of the Vogel-equation for determining U_v is given as an example in Appendix 3.</p>	
		<p>9.5 It is recommended to calculate the uncertainty of viscosity measurement according to the method described in the WECC document No 19 and in a more general way in the “Guide to the expression of uncertainty in measurement”. [It is recommended that the uncertainty of the measurements be calculated according to the <i>Guide to the expression of uncertainty in measurement</i>]. Convenient summaries can be found in [] and [.]</p>	accepted

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		<p>The coverage factor should be $k=2$ corresponding to an interval of confidence of 95%. An example is given in Appendix 4.</p> <p>Generally, the [The] main contribution to the uncertainty [usually] arises from the uncertainty u_C of the viscometer constant C.</p> <p>According to the OIML DI No 17 the uncertainty of 1st order SSp shall be between 0.2% and 1% and of 2nd order SSp between 0.3% and 2% depending on the viscosity. [According to OIML document D 17, [?], the ($k=2$) uncertainty of the 1st order VSS must not exceed 0.2% to 1% (depending on the viscosity) and that of the 2nd order VSS must not exceed 0.3% to 2% (depending on the viscosity).] These limiting values are met by the requirements described in clause 4 and include a period of 6 months for the validity of the calibration. It also includes an estimated maximum contribution of up to 20 % of the total uncertainty in order to cover undetected systematic deviations and [Undetected errors must either be estimated independently or not included in the analysis.] influences of minor importance like deviation from vertical alignment of the viscometer and uncertainty of the g-value [gravitational acceleration]. Table 1 shows the resulting measurement uncertainties provided [that] the requirement of clause 4 are met.</p>	
Clause 10	Poland	It should be "or" instead of "ore"	accepted
Appendix 4	Netherlands	We suppose that in formula 1, S_v^1 is to be replaced by S_v In the sentence starting with "Random..." (second last paragraph) remove the dot after the word "the"	accepted
	Poland	It should be "Random uncertainty of the " (delete dot)	accepted
	Japan	"95 % level of confidence" instead "of 15 % confidence level"	accepted
Appendix 5	Japan	"g/cm ³ " instead of "g/sm ³ "	accepted
	Netherlands	Last column in the Table "replace "g/cm ³ " by "g/sm ³ "	accepted

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References	USA	<p>Suggested additions to References</p> <p>EA-4/02 (1999) Expression of the uncertainty of measurement in calibration, European Co-operation for Accreditation of Laboratories. [This replaces WECC document 19.]</p> <p>NIST 1297 (1994) Guidelines for evaluating and expressing the uncertainty of NIST measurement results, National Institute of Standards and Technology (USA).</p> <p>NBS Monograph 55, NBS Viscometer Calibrating Liquids and Capillary Tube Viscometers</p> <p>Journal of Research of the National Bureau of Standards, Vol. 52, No. 3, March 1954, Research Paper 2479</p> <p>ASTM Method of Test D 2162, Basic Calibration of Master Viscometers and Viscosity Oil Standards</p>	accepted
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