

# Template for comments and convener's observations

Date:2020-03-04

Document: TC17/SC3/P1

Project: Revision of R 54: pH scale for aqueous solutions

Country Code <sup>1</sup>	Part	Clause/ Subclause	Paragraph/ Figure/Table	Type of comment <sup>2</sup>	Comments	Proposed change	Convener's responses
0001 DE				ge	English is not adequate.	Text must be revised throughout the Recommendation by a native English-speaking person.	Agreed. Corrected.
0002 US				ge	<p>Technically, very little has changed since the document was last issued. It is not clear that there is an advantage to this revision. Further, the use of the IUPAC recommendations from 2002 as a reference is preferred.</p> <p>The IUPAC recommendations are appropriate for aqueous and dilute (0.1 molal) ionic strength solutions, according to the Bates-Guggenheim method for primary standards. There is no mention of ionic strength here, nor what implications it might have for the methods to be used.</p>	Recommend against revision and phasing out of this recommendation.	<p>Noted the comments and revised the Project, but had some explanation. The revision was proposed due to some discrepancies in the data of the existing scale with the real data obtained as results of key comparisons within the framework of the BIPM. Of course, the Recommendation takes into account the IUPAC article. However, please note that IUPAC 2002 is just an article. While this Recommendation has the status of OIML, which is signed by countries and can be used in the field of legal metrology. In any case, we revised the WD of the Project and taking into account all the comments.</p> <p>See paragraph 1.2.</p>
0003 US				ge	The document is incomplete as a stand-alone reference for pH. It would benefit from a full protocol for the means of verifying pH and doing		Noted. Corrected. Although, the Title contents the matrix (aqueous solution). For other

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					the calibration. There is no discussion, for example of matrix matching of buffers, which is an issue for SI traceability.  It is unclear what the purpose of the document is. Is it meant for calibration of a Harned Cell; for production of secondary solutions as reference materials? There appear to be some inconsistencies with tables in the IUPAC document.		matrixes it is possible to develop another Recommendation.  Noted.
0004 IR1				Ge	The English language should be improve		Agreed. Corrected.
0005 IR2				Ge	The scope in standard is missing		Noted. See paragraph 1.1.
0006 IR3				Ge	Clause should have number continuously		Agreed.
0007 IR9				ge	In the text unit of temperature is shown with both °C and K one of them should be use in all the text and preferably °C.		Agreed. The temperatures were corrected in K, as the K is the SI unit.
0008 DK					No commets		Noted
0009 JP			Foreword <sup>2nd</sup> para.	Edit.	The term “pH-metry” is not used generally.	Replace “pH-metry” with “pH measurements” for example.	Agreed. Corrected.
0010 US			Table 1 Note 3	Ed	Introduce term “expanded” to reflect the range of uncertainties.	Change to read, “Note 3. Recommended target expanded uncertainties of pH measurement are in the order of $U(pH) \leq 0.003$ ( $k = 2$ ) at 298.15 K. In the temperature range 278.15 – 323.15 K (except 298.15 K) the target expanded uncertainty of pH measurement is 0.005.”	Agreed. Corrected.
0011 DE			Whole document	Ed	Term “dehydrate” inadequate	use “dihydrate” instead	Agreed. Corrected.
0012 DE		01	Introduction	Ge	The background and objective of the Recommendation is kept much too short	Background and objective should be outlined in more detail	Noted

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0013 DE		01	Introduction	ge	Differential measurement principle (using the so called Bauke-cell) is not mentioned, even though it is a commonly used method.	Mention secondary standards derived from primary standards as mentioned in ref [1]/8.2	Noted
0014 US		01.02		Ed	Clarify use of PT electrode. Is this a secondary calibration?	“The primary method of measuring pH is applied in the electrochemical Harned cell, using a platinum/palladium coated platinum foil electrode, hydrogen gas and silver-silver chloride electrodes, in the pH range 3.542 – 10.245 in the temperature range 278.15 – 323.15 K.”	Agreed. Corrected.
0015 JP		01.02		Gen./Tech.	<i>The primary method of measuring pH is applied in the electrochemical <u>Harned cell</u>, using hydrogen gas and silver-silver chloride electrodes, in pH range 3.542 - 10.245 in the temperature range 278.15 - 323.15 K.</i>  In the latest IUPAC Recommendations 2002, both primary and secondary standard solutions in a range of approximately pH 1 - 13 were provided using the Harned cell method. The secondary standard solutions in R 54 (1WD) in the pH range of 1.47 - 13.16 were, however, provided using the glass electrode method based on the old versions (1985) of IUPAC Recommendations.	The present statements should be harmonized with those of IUPAC Recommendations 2002. Also, it shall be examined if the descriptions on the solutions provided using the glass electrode method are necessary.	Agreed. Corrected.
0016 DE		01.02	Introduction	ge	The number of digits of the stated pH range is unreasonably accurate for the statement of a range, that depends on temperature and pH values that show batch to batch variations.	... roughly ranging from pH 3.5 to 10.3	Agreed. Corrected.
0017 US		01.03		Ed	Clarify temperature range of measurements. Also note the IUPAC guidelines are for pH 3-10 at 25°C	“The working method of pH in the pH range 1.47 – 13.16 and the temperature range 278.15 – 323.15 K is by measurement with a pH-meter and an electrode system, consisting of a reference electrode and a glass electrode.	Agreed. Corrected.
0018		01.03		Gen./Tech.	The second and third paragraphs read:	The present statements should be harmonized	Agreed. Corrected.

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JP					<p><i>The working measurement method of pH is applied in measuring system, using <u>glass electrode</u>, in pH range 1.47 - 13.16 (pH-meter with the electrode system, consisting of a reference electrode and glass electrode) in the temperature range 278.15 - 323.15 K.</i></p> <p>In the latest IUPAC Recommendations 2002, both primary and secondary standard solutions in a range of approximately pH 1 - 13 were provided using the Harned cell method. The secondary standard solutions in R 54 (1WD) in the pH range of 1.47 - 13.16 were, however, provided using the glass electrode method based on the old versions (1985) of IUPAC Recommendations.</p>	with those of IUPAC Recommendations 2002. Also, it shall be examined if the descriptions on the solutions provided using the glass electrode method are necessary.	
0019 JP		1.3		Edit.	The term “pH-metr” is not generally used.	Replace “pH-metr” with “pH meter”.	Agreed. Corrected.
0020 IR4		1.3		te	, in pH range 3.542 - 10.245 in , is not correct	, in pH range <b>3.564</b> - 10.245 in the temperature	Agreed. Corrected.
0021 IR5		1.3		Ge	pH-metr with the electrode system	Modify to: <b>pH-meter</b> with the electrode system and as well as in all of the text	Agreed. Corrected.
0022 US		1.4		Ed	Edits for clarity	In practice, the buffer solutions, with pH values assigned by the primary method, are produced by metrological organizations. The pH values obtained by the working procedure are used in analytical and scientific laboratories.	Noted.
0023 DE		1.4	Introduction	ge	(accredited) calibration laboratories are a third important user group	Mention calibration laboratories	Agreed. Corrected.
0024 US		2.1		Ed	Minor language edits.	This Recommendation is used for calibration and verification of pH-meters that consists of electrodes and measuring transducer intended for pH determination for aqueous solutions.	Agreed. Corrected.

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						<p>The Recommendation is harmonized with the International Recommendation IUPAC “Measurement of pH. Definition, standards, and procedures, IUPAC Recommendations 2002” [1], and takes into consideration Recommendations of some international standards [2-5].</p> <p>This Recommendation relates to analytical laboratory determinations of pH of dilute aqueous solutions with ionic strength not greater than 0.1 mol kg<sup>-1</sup>.</p>	
0025 JP		2.2	Entire draft and Field of application	Gen.	<p>The second paragraph of “Field of application” reads:</p> <p><i>The Recommendation is harmonized with the International Recommendation IUPAC “Measurement of pH. Definition, standards, and procedures, IUPAC Recommendations 2002” [1], and takes into consideration Recommendations of some international standards [2-5].</i></p> <p>Although this paragraph declares a harmonization with the latest IUPAC Recommendations 2002, which are considered as a de facto standard in pH measurement, many lacks in harmonization remain particularly in “Introduction” and Tables 1 to 4. Some parts still seem to maintain a harmonization with the old versions (1985) of IUPAC Recommendations.</p>	<p>This is a general comment to the entire draft. For concrete proposed changes, please refer our other comments.</p>	Agreed. Corrected.
0026 IR6		3		Ge	<p><b>It should better this clause “Notional definition of pH”</b> change to terms and definitions and pH define in the subclause.</p>	Change to: <b>terms and definitions</b>	Agreed. Corrected.

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0027 JP		3.1	1 <sup>st</sup> para.	Edit.	“ <i>pH</i> ” and “ <i>H</i> <sup>+</sup> ” should not be italicized.	Correct “ <i>pH</i> ” and “ <i>H</i> <sup>+</sup> ” to be unitalicized.	Agreed. Corrected.
0028 DE		4.1		te	Description of the Harned cell is not complete	“pH value can be determined from electrochemical data from the cell without transference using the hydrogen gas electrode <b>combined with a silver-silver chloride electrode</b> , known as the Harned cell.	Agreed. Corrected.
0029 US		4.1	Table 1	ge	This table appears to be a replication of the values from Table 2 of the 2002 IUPAC recommendations.		Agreed.
0030 US		4.1	Table 1	te	Solution P No. 1 value at 293.15 is stated as 3.564. Where does this value come from? It is not in the IUPAC reference. There is only a single issue of the NIST SRM corresponding to this, certified only from 25°C to 95°C		Agreed.
0031 US		4.1	Table 1	te	Solution P No. 2 at 313.15 K is 4.027 and 323.15 K is 4.050. This agrees with Table 2 of IUPAC recommendations, but is out of the ± 0.003 pH unit tolerance of Table 4-6 of Bates. When compared to the average pH(S) value of SRM 185d (issued 1967) through SRM 185g (issued 1991), they disagree by 0.005 – 0.008 pH units.		Noted.
0032 US		4.1	Table 1 Note 1	te	NIST carefully inspects materials before using them in the renewal of a standard reference material (SRM). We would reject any buffer material with a pH value outside of a ± 0.003 tolerance. So, if the table here is based on data from NIST SRMs, then there will be no batch-to-batch differences greater than 0.003 pH units. In this case the language can be changed to that suggested.	Change to read, “Note 1. Since there can be significant variations in the purity of samples of the same nominal chemical composition, batch-to-batch variations of 0.003 pH units are typical.”	Agreed.
0033 JP		4.1	Table 1	Gen./Tech.	In the pH values of Table 1, the value of “Solution P No 1” at 293.15 °C (3.564) does not exist in IUPAC Recommendations 2002. An appropriate	Please confirm these values. Also, please provide the evidences for the values appropriately.	Agreed.

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					evidence for this value should be provided. In addition, the value of “Solution P No 2” at 293.15 °C (4.001) is different from that of IUPAC Recommendations 2002 (4.000). Reason(s) of the difference should be explained with an appropriate evidence.		
0034 US		4.1	Table 2	te	It is not clear where the data for this table comes from – perhaps a pH meter and glass electrode or reference electrode with transference. Is it intended for secondary purchased buffers? How is this different from Table 3, where a glass electrode system is used to define pH values? Further explanation about the process would be helpful.		Corrected. Table 2 was deleted.
0035 US		4.1	Table 2	te	The use of a glass electrode is not a primary method. Nothing in the document indicates a process for calibration for an electrode. Without calibration, it is not possible to have three decimal places of accuracy.		Corrected. Table 2 was deleted.
0036 JP		4.1	Table 2	Gen./Tech.	Table 2 contradicts the guidelines proposed by IUPAC Recommendations 2002. According to this recommendation, pH standard solutions produced using the glass electrode are not regarded as the primary buffer solutions. Moreover, there are no evidences for the values in this table. Purposes and practical users of this table are not clear.	In order to harmonize with IUPAC Recommendations 2002, it shall be examined if pH standard solutions provided using the glass electrode method are necessary. If this table is maintained, necessary evidences shall be given.	Corrected. Table 2 was deleted.
0037 US		4.1.1		Ed	Minor language edits	Begin, “The pH value can be determined...”	Agreed.
0038 IR8		4.1.1		te	using the hydrogen gas electrode, known as the Harned cell	Modify to:	Agreed.

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					this sentence should be complete as	, using the hydrogen gas electrode and silver-silver chloride electrodes, known as the electrochemical Harned cell	
0039 IR10		4.1.1		ed	«Measurement of pH. Definition, standards, and procedures, IUPAC Recommendations 2002» [2].	Modify to: «Measurement of pH. Definition, standards, and procedures, IUPAC Recommendations 2002» [1].	Agreed.
0040 US		4.1.2		Ed	Edit for clarity	These six primary buffer solutions are recommended:	Agreed.
0041 IR7		4.1.2		ge	It should better define primary buffer solutions and working buffer solutions in the terms and definitions		Noted.
0042 IR11		4.1.2		ge	molality values are not rounded the same. Number of significant figures should be the same preferably with three decimal rigid. 0.025 + 0.025 mol·kg <sup>-1</sup> (solution P № 3); 0.008695 + 0.03043 mol·kg <sup>-1</sup> (solution P № 4);		Agreed.
0043 JP		4.1.2	3th and 4th bullets of the list	Edit. /Tech.	The terms “potassium dihydrogen orthophosphate” and “disodium hydrogen orthophosphate” are based on the old versions of IUPAC Recommendations.	Replace “potassium dihydrogen <u>orthophosphate</u> ” with “potassium dihydrogen <u>phosphate</u> ”. Also, replace “ <u>disodium</u> hydrogen <u>orthophosphate</u> ” with “ <u>sodium</u> hydrogen <u>phosphate</u> ”.	Agreed.
0044 DE		4.1.5	Table 1	ed	Solution 2 at 293.15 K deviates from ref [1] table 2,	Should be 4.000, to be consistent	Agreed.
0045 DE		4.1.5	Table 1	ed	Ref [1] doesn't state the pH value for solution 2 at 293.15	Add reference	Agreed.
0046 DE		4.1.5	Table 1	ge	For practical reasons temperatures should be given in °C rather than K	Use of unit °C	Noted.

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0047 DE		4.1.5	Table 1	ge	Note 3: uncertainty does not comply with ref [1], Annex A1	Typical expanded (k=2) uncertainty is U(pH)=0.003 - 0.004	Agreed.
0048 DE		4.1.5	Table 1	Ed	Reference for values of table 1 is not stated explicitly, I guess it is ref [1]?	Make a clear reference	Agreed.
0049 DE		4.1.5	Table 2	Ed	Reference for values of table 1 is not clear	Make a clear reference	Agreed.
0050 DE		4.1.5	Table 2	ge	The typical standard uncertainty of a glass electrode according to ref [1], Annex A5 and table A5c is 0.005, U(pH(X))=0.01 respectively. 3 digits in table 2 are therefore not adequate. The necessity of table 2 is questionable anyway, since it does not provide additional information compared to table 1. Moreover, the actually relevant values for glass electrode measurements are given in table 3.	Omit table 2. If not, the values of table 2 should state only 2 digits to comply with the uncertainty of a glass electrode measurement.	Corrected. Table 2 was deleted.
0051 US		4.1.6		Ed	For clarity	“The difference in pH values given in Tables 1 and 2 is due to the presence of a diffusion potential between the glass and reference electrodes.”	Corrected. Table 2 was deleted.
0052 DE		4.2	Table 3	Ed	Reference for values of table 3 is not clear.	State reference for table 3 explicitly.	Corrected. Table 3 was deleted.
0053 US		4.2	Table 3	te	The source of values for this table is unclear, nor is it clear what cell/measurement method is being used to arrive at the values. This appears to combine some secondary reference materials and some primary reference materials from tables 1 and 2 respectively, and then the values are rounded. Three new secondary materials are introduced into this table, and it is not clear what the reasoning behind this is. Finally, how are uncertainties being evaluated? For IUPAC, target error is 0.01 to 0.03 pH units.		Corrected. Table 3 was deleted.

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0054 JP		4.2	Table 3	Edit. / Tech.	Table 3 of this IWD does not conform with Table 3 of "IUPAC Recommendations 2002".	Table 3 of this IWD should be harmonized with that of IUPAC Recommendations 2002. It is necessary to show the evidence of the data.	Corrected. Table 3 was deleted.
0055 US		4.2	Tables 1-3	Ge/ed	The document would benefit from a consistency in naming the solutions similarly throughout. As written, this introduces confusion. (e.g. Solution S No. 7 in Table 3 is Solution P No. 5 in Table 1)		Corrected
0056 US		4.2.1		Te		Change "voltage" to "impedance".	Corrected
0057 US		4.2.1		Ed	It is not clear what is meant by, "To measure pH the predetermined graduation of the metrical index for two or more primary buffer solutions (Solution P № 1...6, Table 2) with exact determined pH values is demanded."	"At least two buffer solutions, with assigned pH values traceable to the primary cell, are required for calibration of the pH meter and electrode pair."	Corrected
0058 IR12		4.2.1		te	It is the <b>voltmeter</b> with high input voltage ( $\geq 10$ T $\Omega$ ) It is better to use <b>potentiometer instead of voltmeter</b>	It is the <b>potentiometer</b> with high input voltage ( $\geq 10$ T $\Omega$ )	Corrected
0059 IR13		4.2.1		te	It is the voltmeter with high input voltage ( $\geq 10$ T $\Omega$ ) high input voltage is not correct and it should be high input impedance.	It is the voltmeter with high input <b>impedance</b> ( $\geq 10$ T $\Omega$ )	Corrected
0060 JP		4.2.1	1 <sup>st</sup> para.	Edit. / Tech.	The present expression "the voltmeter with high input <u>voltage</u> " may not be appropriate because it is understood as "a voltmeter for measuring a high voltage".	Another expression "the voltmeter with high input <u>resistance</u> " seems more appropriate. It is recommended to replace "voltage" with "resistance".	Corrected
0061 JP		4.2.1	1 <sup>st</sup> para.	Tech.	Input resistance in the expression "high input voltage ( $\geq 10$ T $\Omega$ )" may be too large.	For reference, JIS (Japan Industrial Standards) Z 8805 specifies a value of input voltage more than $1 \times 10^{11} \Omega$ (= 0.1 T $\Omega$ ).	Corrected

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0062 JP		4.2.2		Edit.	The term “pH-metry” is not used generally.	Replace “pH-metry” with “pH measurements” for example.	Corrected
0063 IR14		4.2.2		te	Solution of potassium tetraoxalate dehydrate $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ Dehydrate should be change to dihydrate	Solution of potassium tetraoxalate <b>dihydrate</b> $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$	Corrected
0064 IR15		4.2.2			Solution of potassium tetraoxalate <b>dehydrate</b> $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O} - 0.05 \text{ mol} \cdot \text{kg}^{-1}$ (solution S № 2); Dehydrate should be change to dihydrate	Solution of potassium tetraoxalate <b>dihydrate</b> $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O} - 0.05 \text{ mol} \cdot \text{kg}^{-1}$ (solution S № 2);	Corrected
0065 JP		4.2.2	5 <sup>th</sup> and 6 <sup>th</sup> bullets of the list (p. 8)	Edit. /Tech.	The terms “potassium dihydrogen orthophosphate” and “disodium hydrogen orthophosphate” are based on the old versions of IUPAC Recommendations.	Replace “potassium dihydrogen <u>orthophosphate</u> ” with “potassium dihydrogen <u>phosphate</u> ”.  Also, replace “ <u>disodium</u> hydrogen <u>orthophosphate</u> ” with “ <u>sodium</u> hydrogen <u>phosphate</u> ”.	Corrected
0066 US		5	Appendix A	te	For the heading of the 4 <sup>th</sup> column, need to specify a temperature if the solution is prepared on a volumetric basis. All other cases seem to point to a weight (molal or mol kg <sup>-1</sup> basis).		Noted
0067 US		5	Appendix A	Ed	Use “molality” rather than “concentration” for heading of 5 <sup>th</sup> column.		Agreed.
0068 JP		5	Appendix A Table 4	Edit. / Tech.	Table 4 of this 1WD does not conform with Table 2 of “IUPAC Recommendations 2002”.	Table 4 of this 1WD should be harmonized with IUPAC Recommendations 2002. It is necessary to show the evidence of the data.	Text is partially revised
0069 DE		5	Table 4	Ed	Heading of column 5: mol/kg is the unit for “Molality”	Replace “concentration” by “molality”	Agreed.
0070 DE		5	Table 4	Ge	Meaning of column 4 is not clear	Explain or omit	Agreed.
0071 DE		5	Table 4	Ge	Columns 6 and 7 are redundant, information is already given in tables 1 and 2	omit columns	Text is partially revised

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0072 DE		5	Table 4	Ge	The information given in tables 4 is rather sparse compared to the corresponding information given in table 1 of ref [1]	Add relevant information or omit the table and refer to ref [1]	Noted.
0073 IR19		5	Table 4 Column 4		Column head should be replaced with	Mass needed to make 1l solution g	Agreed.
0074 IR20		5	Table 4 Column 4	te	It should be prepared at the temperature in which the density of the solution is equal to one kg per l	The applicable temperature should be added to column head	Agreed.
0075 US		6	Appendix B	te	For the heading of the 4 <sup>th</sup> column, again, need to specify a temperature if the solution is prepared on a volumetric basis. All other cases seem to point to a weight (molal or mol kg <sup>-1</sup> basis).		Agreed.
0076 US		6	Appendix B	Ed	Use “molality” rather than “concentration” for heading of 5 <sup>th</sup> column.		Agreed.
0077 IR21		6	Table 4 Column 4	te	It should be prepared at the temperature in which the density of the solution is equal to one kg per l	The applicable temperature should be added to column head	Agreed.
0078 DE		6	Table 5	Ed	Reference not clear	State reference	Corrected. Table 5 was deleted.
0079 DE		6	Table 5	ed	Heading of column 5: mol/kg is “Molality”	Replace “concentration” by “molality”	Corrected. Table 5 was deleted.
0080 DE		6	Table 5	Ge	Meaning of column 4 is not clear	Explain or omit	Corrected. Table 5 was deleted.
0081 DE		6	Table 5	Ge	Columns 6 is redundant, information is already given in tables 3	omit columns	Corrected. Table 5 was deleted.
0082 DE		6	Table 5	Ge	The information given in table 5 is sparse.	Add information in analogy to ref [1] table 1 or give a reference	Corrected. Table 5 was deleted.
0083 IR16		6	Table5 Column 2 Second row	te	Potassium tetraoxalate <b>dehydrate</b>	Potassium tetraoxalate <b>dihydrate</b>	Corrected. Table 5 was deleted.
0084 IR18		6	Table5 Column 2 Third row	te	Potassium tetraoxalate <b>dehydrate</b>	Potassium tetraoxalate <b>dihydrate</b>	Corrected. Table 5 was deleted.

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<sup>1</sup> Country code (enter the ISO 3166 two-letter country code, e.g. CN for China)

<sup>2</sup> Type of comment: ge = general te = technical ed = editorial

## Template for comments and convener's observations

Date:2020-03-04	Document: TC17/SC3/P1	Project: Revision of R 54: pH scale for aqueous solutions
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Country Code <sup>1</sup>	Part	Clause/Subclause	Paragraph/Figure/Table	Type of comment <sup>2</sup>	Comments	Proposed change	Convener's responses
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Z:\Website\_procedures\Comments collation tools\R 54\TC17\_SC3\_P1\_N002-comments-Japan-171201.docx: Collation successful

Z:\Website\_procedures\Comments collation tools\R 54\TC17\_SC3\_P1\_N002-comments\_template.docx: Collation successful

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FAILED TEST (number of files): 0

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<sup>1</sup> Country code (enter the ISO 3166 two-letter country code, e.g. CN for China)

<sup>2</sup> Type of comment: ge = general te = technical ed = editorial