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Hydraulic fluid power — Sample calculations for ISO 11171

Transmissions hydrauliques — Calculs des échantillons pour l'ISO 11171

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Foreword

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This document was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

ISO 11171:2022, like its predecessors, retains traceability to the internationally accepted definition of a metre and reports particle size in units of µm(c). The methods for determining data acceptance criteria, coincidence error limit, working flow rate and resolution remain unchanged, but mathematical calculations and tools were first introduced in ISO 11171:2020 to ensure consistency in terms of how automatic particle counter (APC) calibration curves are created and used. For example, mathematical techniques have been introduced to determine the APC threshold settings used to obtain calibration data and a tool provided to generate calibration curves. Other mathematical equations to estimate the standard error of the calibration, to calculate normalized concentrations for diluted samples, and to calibrate at particle sizes larger than 30 µm(c) were first introduced in 2020. This document uses example calculations that fully conform to ISO 11171.

Hydraulic fluid power — Sample calculations for ISO 11171

1 Scope

This document shows how to use the normative mathematical formulae and tools of ISO 11171. Examples are used to demonstrate their use for calibrating automatic particle counters (APCs).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11171, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11171 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Example 1: Selection of threshold voltage settings

The method of selecting threshold voltages for particle sizing calibration is specified in ISO 11171:2022, 6.3, which requires that:

— a minimum of 12 different threshold settings be used to construct a calibration curve;

— the first (lowest) threshold setting, J, be 1,5 times the threshold noise level of the APC;

— the highest threshold setting, H, corresponds to a particle size of approximately 30 µm(c) or smaller for primary calibrations and corresponds to a size that does not exceed the largest reported particle size that is in conformance with ISO 11171:2022, Annex F, for secondary calibrations;

— intermediate threshold settings be logarithmically spaced such that the value of each channel is K times greater than its preceding channel, where K is a constant defined by Formula (1):

$$K = 10^{(\log H - \log J)/(G-1)} \quad (1)$$

where G is the number of threshold settings used to construct the calibration curve and is greater than or equal to 12.

This example considers an APC with eight threshold settings that can be adjusted in 1 mV increments. The threshold noise level of the APC was determined to be 5 mV and its manufacturer indicated that 30 µm(c) is expected to correspond to a threshold voltage setting of about 2 600 mV.

The calibration curve will be determined using 12 threshold voltage settings, the minimum number permitted by ISO 11171. Thus, the value of H is 2 600 mV and the value of G is 12. The value of J is determined by Formula (2):

$$J = 1,5 \times 5 = 7,5 \qquad (2)$$

Since this APC can only be adjusted in 1 mV increments, the value of J is rounded up to 8 mV for calibration. Using the values of G, H and J, the value of K can be calculated by Formula (3):

$$K = 10^{(\log H - \log J)/(G-1)} = 10^{(\log(2\,600) - \log(8))/(12-1)} = 1,692 \qquad (3)$$

The threshold settings for the 10 intermediate channels are set at values corresponding to 1,692 times the value of each preceding channel as shown in Table 1.

Table 1 — Threshold voltage settings for APC in Example 1

Threshold setting number	Calculation	Threshold voltage setting[a]
1	1,5 × 5 mV =	8 mV
2	1,692 × 8 mV =	14 mV
3	1,692 × 14 mV =	23 mV
4	1,692 × 23 mV =	39 mV
5	1,692 × 39 mV =	66 mV
6	1,692 × 66 mV =	111 mV
7	1,692 × 111 mV =	188 mV
8	1,692 × 188 mV =	317 mV
9	1,692 × 317 mV =	537 mV
10	1,692 × 537 mV =	908 mV
11	1,692 × 908 mV =	1 537 mV
12	1,692 × 1 537 mV =	2 600 mV

[a] Threshold voltage settings rounded off to the nearest mV based upon the capabilities of the APC in the example.

This APC only has the minimum number of channels required in ISO 11171:2022, 6.4 (eight channels), but ISO 11171:2022, 6.3, requires data from twelve or more threshold settings to construct a calibration curve. ISO 11171:2022, 6.8, requires that data from at least two different samples be obtained for each of the threshold voltage settings (refer to ISO 11171:2022, 6.8) and that the channels used for a particular sample be distributed over the entire range to the extent possible. To meet these requirements in this example, eight different threshold settings chosen from the list of twelve can be used for the first sample and different combinations of eight threshold settings used for each of the other two samples.

An example of how to allocate threshold settings among the eight channels is shown in Table 2, where the first column lists the twelve required threshold voltage settings and columns 2, 3 and 4 show the channels used to collect data at these settings for the indicated sample. The last column in Table 2 shows the number of samples for which data are obtained for each threshold setting, confirming these requirements have been met.

Table 2 — Allocation of threshold voltage settings among the eight channels of the Example 1 APC

Threshold voltage setting mV[a]	Channel number corresponding to indicated threshold voltage setting and sample			Number of samples at indicated threshold voltage setting
	Sample A	Sample B	Sample C	
8	1		1	2
14		1	2	2
23	2		3	2
39	3	2		2
66		3	4	2
111	4		5	2
188	5	4		2
317		5	6	2
537	6		7	2
908	7	6		2
1 537		7	8	2
2 600	8	8		2

[a] Threshold voltage settings determined in Table 1.

5 Example 2: Evaluating data quality

ISO 11171:2022, 6.6, specifies how to verify the acceptability of particle count data for APC calibration purposes. In brief, the process involves:

— calculation of the total number of particles, N, counted for a given APC channel and sample;

— calculation of the data quality factor, D_Q;

— identification of potential outliers among the data if the D_Q is unacceptable.

This process is used throughout ISO 11171 to ensure the integrity of data used for APC calibration. This example uses a calibration suspension sample analysed as described in ISO 11171:2022, 6.5 and 6.6, using a sample volume, V, of 10 mL. Unless otherwise noted, the term "particle concentration" refers to cumulative particle concentration throughout this document. Particle concentrations of 26 068 particles/mL, 25 757 particles/mL, 25 802 particles/mL, 31 771 particles/mL and 25 834 particles/mL were obtained. The mean particle concentration, $\bar{X}$, for these five counts is 27 046. The mean observed number of particles counted for the five particle counts, X, is given by Formula (4):

$$X = \bar{X}V = 27\,046 \times 10 = 270\,460 \tag{4}$$

The total number of particles, N, counted for the sample is calculated using Formula (5):

$$N = 5X = 270\,460 \times 5 = 1\,352\,300 \tag{5}$$

This value is greater than 1 000, as required by ISO 11171:2022, 6.6, hence is sufficiently high for calibration purposes.

Using ISO 11171:2022, Table C.2, and the value of X previously calculated, the maximum allowable D_Q for the data can be determined. Referring to the first two columns of the table, a value of 270 460 for X corresponds to the first row of the table, i.e. X greater than or equal to 10 000. The maximum allowable D_Q can be found in the third column, which is used for ISO 11171:2022, 6.6, 6.13, B.5, C.9, D.4, D.9, E.6

and F.5. Thus, the maximum allowable D_Q is 11,0 for this example. The value of $D_{Q,}$ expressed as a percentage, for the data in this example is calculated using Formula (6):

$$D_Q = \frac{X_{max} - X_{min}}{\overline{X}} \times 100 = \frac{31\,771 - 25\,757}{27\,046} \times 100 = 22{,}24 \qquad (6)$$

where

X_{max} is the maximum number of counts observed among the five particle counts or 31 771;

X_{min} is the minimum number of counts observed among the five particle counts or 25 757.

Since the value of D_Q is greater than the maximum allowable D_Q, the data are unacceptable for calibration purposes and can be examined for possible outliers.

The outlier test parameter, D_0, for the data in this example is calculated using Formula (7):

$$D_0 = \frac{X_{max} - X_{min}}{|X_0 - X_N|} = \frac{31\,771 - 25\,757}{|31\,771 - 26\,068|} = 1{,}05 \qquad (7)$$

where

X_0 is the observed particle concentration of suspected data outlier (either X_{max} or X_{min}), 31 771 particles/mL;

X_N is the observed particle concentration closest in value to the suspected outlier, 26 068 particles/mL.

If the value of D_0 is less than 1,44, as in this example, X_0 can be discarded as a statistical outlier. In Example 2, D_0 was found to be 1,05, well below 1,44, hence the suspect data point, 31 771, can be discarded as an outlier. The remaining four data points are used to recalculate $\overline{X}$, giving a value of 25 865 particles/mL, which will be used in constructing the calibration curve.

In another example, data from a different channel setting is considered for the same calibration suspension sample analysed in the previous example. For this channel setting, particle concentrations of 810 particles/mL, 802 particles/mL, 800 particles/mL, 805 particles/mL and 803 particles/mL were obtained.

The mean particle concentration, $\overline{X}$, for these five counts is 804. The mean observed number of particles counted for the five particle counts, X, is given by Formula (8):

$$X = \overline{X}V = 804 \times 10 = 8\,040 \qquad (8)$$

The total number of particles, N, counted for the sample is calculated using Formula (9):

$$N = 5X = 8\,040 \times 5 = 40\,200 \qquad (9)$$

This value is greater than 1 000, as required by ISO 11171:2022, 6.6, hence is sufficiently high for calibration purposes.

Using ISO 11171:2022, Table C.2, and the value of X previously calculated, the maximum allowable D_Q for the data can be determined. Referring to the first two columns of the table, a value of 8 040 for X corresponds to the second row of the table, i.e. X greater than or equal to 5 000 but less than 10 000. The maximum allowable D_Q can be found in the third column, which is used for ISO 11171:2022, 6.6, 6.13, B.5, C.9, D.4, D.9, E.6 and F.5. Thus, the maximum allowable D_Q is 11,3 for this example. The value of D_Q, expressed as a percentage, for the data in this example is calculated using Formula (10):

$$D_Q = \frac{X_{max} - X_{min}}{\overline{X}} \times 100 = \frac{810 - 800}{804} \times 100 = 1{,}20 \qquad (10)$$

where

X_{max} is the maximum number of counts observed among the five particle counts or 810;

X_{min} is the minimum number of counts observed among the five particle counts or 800.

Since the value of D_Q is less than the maximum allowable D_Q, the data are acceptable for calibration purposes and can be used in constructing the calibration curve.

6 Example 3: Dilution of samples

To facilitate calibration at small particle sizes, ISO 11171:2022, Annex G, provides a standardized procedure for diluting calibration suspensions and ISO 11171:2022, 6.7, specifies a method for normalizing the resultant particle count data. To use this procedure, it is necessary to know the coincidence error limit of the APC and the approximate size of the smallest particles that it can count. In this example, the APC is capable of counting particles as small as 2 µm(c) and has a coincidence error limit, X_A, of 12 713 particles/mL. The certified particle size distribution of the calibration samples is shown in Table 3.

Table 3 — Certified particle size distribution of calibration sample for Example 3

Certified particle size µm(c)	**Certified particle concentration** particles/mL
2	33 066
3	17 714
4	10 865
5	6 637,0
6	4 210,0
7	2 886,4
8	2 007,2
9	1 478,7
10	1 114,9
11	847,55
12	649,63
13	502,37
14	389,25
15	299,27
16	230,39
17	180,38
18	142,77
19	114,53
20	93,176
21	77,445
22	65,134
23	55,040
24	46,831
25	40,194
26	34,678
27	29,990

Table 3 *(continued)*

Certified particle size µm(c)	**Certified particle concentration** particles/mL
28	26,006
29	22,665
30	19,697

The expected number concentration of particles in the diluted calibration suspension samples at the smallest particle size, X_D, is calculated as shown in Formula (11):

$$X_D = \frac{X_A}{1,3} = \frac{12\,713}{1,3} = 9\,779 \tag{11}$$

The smallest size that the APC can count is 2 µm(c). Referring to Table 3, the certified particle concentration at this size, X_C, is 33 066 particles/mL. The minimum dilution ratio, D_{RR}, that is required to achieve X_D can be estimated using Formula (12):

$$D_{RR} = \frac{X_C}{X_D} = \frac{33\,066}{9\,779} = 3,38 \tag{12}$$

ISO 11171:2022, Annex G, permits either volumetric or mass dilution. In this example, it can be assumed that diluted calibration suspension samples were prepared by mixing 100,0 mL of calibration suspension weighing 86,0 g with 300,0 mL of dilution fluid weighing 258,0 g. Both sample and fluid have a density of 0,86 g/mL. If volumetric dilution is used, the actual dilution ratio, D_R, is given by Formula (13):

$$D_R = \frac{v_0 + v_S}{v_S} = \frac{300,0 + 100,0}{100,0} = 4,00 \tag{13}$$

where

v_0 is the volume of dilution fluid = 300,0 mL;

v_S is the volume of sample fluid = 100,0 mL.

If mass dilution is used for the same sample, the actual dilution ratio, D_R, is given by Formula (14):

$$D_R = \frac{\dfrac{M_t - M_s}{\rho_d} + \dfrac{M_s}{\rho_s}}{\dfrac{M_s}{\rho_s}} = \frac{\dfrac{344,0 - 86,0}{0,86} + \dfrac{86,0}{0,86}}{\dfrac{86,0}{0,86}} = 4,00 \tag{14}$$

where

M_t is the total mass of diluted sample = 344,0 g;

M_s is the mass of sample = 86,0 g;

ρ_d is the density of dilution fluid = 0,86 g/mL;

ρ_s is the density of sample = 0,86 g/mL.

Since D_R is 4,00, greater than D_{RR}, the actual diluted samples will have particle concentrations below the coincidence error limit of the APC and can be used for calibration.

Continuing the example, the diluted sample was analysed and a mean particle concentration, $\overline{X}$, of 7 041,6 particles/mL obtained. This is below the coincidence error limit of 12 713 particles/mL hence the data are not in coincidence error. Formula (15) can be used to calculate the particle concentration in the undiluted sample, $\bar{X}_N$:

$$\bar{X}_N = \bar{X} D_{\mathrm{R}} = 7\,041{,}6 \times 4{,}00 = 28\,166 \tag{15}$$

where D_{R} is 4,00 as calculated in Formula (14). Thus, the value of $\bar{X}_N$, 28 166, is used for this threshold setting when constructing the calibration curve for this APC.

7 Example 4: Relating particle size to threshold voltage setting for particles 30 µm(c) and smaller

Once acceptable particle count data are obtained, particle size data can be related to threshold voltage setting. ISO 11171:2022, 6.9, provides a link to an Excel[1)] spreadsheet tool that uses the constrained cubic spline interpolation method for this purpose.

This example uses the same APC and calibration suspensions used in the previous examples. Table 2 lists the twelve threshold settings used and how they were allocated among the eight channels of the APC for each of the three samples. Table 3 is the certified particle size distribution for the secondary calibration suspensions. Table 4 shows the threshold settings and corresponding particle count data for the three calibration suspension samples in the first four columns of the completed worksheet from ISO 11171:2022, 6.9, for this example. The values for all twelve of the threshold settings are entered consecutively in the first column in order of decreasing value (highest to lowest). No empty cells are permitted in the first column between the maximum and minimum threshold settings. Mean normalized particle concentrations, $\bar{X}_N$, for the eight channel settings used for sample 1 are entered in the second column adjacent to their corresponding threshold settings. Similarly, $\bar{X}_N$ for the eight channels of sample 2 are entered in the third column and for sample 3 in the fourth column. Each row contains data from at least two different calibration samples. If a threshold setting was not used for a particular sample, the corresponding normalized particle concentration data cell in the spreadsheet is left empty. The mean $\bar{X}_N$ for the samples for each threshold setting is automatically calculated and displayed in yellow column 5. A value of #DIV/0! is displayed in column 5 for any threshold setting lacking particle count data. A numerical value for the mean $\bar{X}_N$ is displayed for each threshold setting before proceeding.

All particle size and corresponding concentration data obtained from the certificate of analysis for the calibration samples, given in Table 3 for this example, are entered in blue columns 6 and 7. The corresponding interpolated threshold voltage setting for each size is automatically displayed in yellow column 8. A value of 0 is displayed for any particle size whose concentration lies outside the range of the particle concentrations shown in column 5, or if no particle concentration is entered. The particle size values shown in blue column 6 and corresponding threshold settings in yellow column 8 relate particle size to threshold setting, e.g. a particle size of 2 µm(c) corresponds to 10 mV in the example. These values will later be used to construct the calibration curve.

1) Excel is the trademark of a product supplied by Microsoft. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Table 4 — Output from the worksheet from ISO 11171:2022, 6.9, for Example 4

Enter all of the threshold settings determined in 6.3 in column A in order of decreasing value of threshold setting. For each calibration suspension sample, enter the Values of $\overline{X}_N$ obtained for each threshold setting actually used for a sample in the appropriate cells of columns B, C or D. If a threshold setting was not used for a particular sample, leave the cell for that sample blank	The particle size and concentration data from the particle size calibration summary portion of Table 4 are entered in columns F and G. The corresponding interpolated threshold voltage setting displayed in column H of this worksheet is entered in Table 4.

Threshold voltage setting	$\overline{X}_N$ for first sample	$\overline{X}_N$ for second sample	$\overline{X}_N$ for third sample	Mean $\overline{X}_N$	Particle size	Particle concentration	Interpolated threshold voltage setting
mV	particles per millilitre	particles per millilitre	particles per millilitre	particles per millilitre	µm(c)	particles per millilitre	mV
2 600	19, 63	19,72		19,675	2	33 066	10
1 537		386,06	308,99	347,52	3	17 714	28
908	1 041,32	1 024,9		1 033,1	4	10 865	83
537	2 261,0		2 341,4	2 301,2	5	6 637,0	190
317		3 932,2	4 128,8	4 030,5	6	4 210,0	305
188	6 397,2	6 980,3		6 688,9	7	2 886,4	442
111	9 043,8		9 707,1	9 375,4	8	2 007,2	595
66		11 980	12 272	12 126	9	1 478,7	738
39	15 553	15 442		15 498	10	1 114,9	873
23	19 741		19 219	19 480	12	649,63	1 176
14		25 369	28 675	27 022	14	389,25	1 479
8	35 784		36 508	36 146	16	230,39	1 805
					18	142,77	2 105
					21	77,445	2 362
					25	40,194	2 515
					30	19,697	2 600
							0

ISO 11171:2022, 6.9, defines how the standard uncertainty in $\bar{X}_N$ is calculated for each threshold setting. By way of illustration, this example uses cumulative particle count data at 14 mV and the previous dilution ratio, D_R, of 4,0 as in Example 1. In an actual calibration, these data would be taken from ISO 11171:2022, Table 2.

To calculate the standard uncertainty, the total number of acceptable particle counts, N_C, and the standard deviation of the particle concentrations for all the acceptable counts for all the samples is calculated. Referring to Table 5, the total number of acceptable particle counts is ten, i.e. five counts obtained for each of two different samples, in the example. The standard deviation, s, is calculated using the individual particle concentration data, X_i, for the ten counts shown in Table 5. In this example, the standard deviation, s, for the ten counts is 466,6 particle/mL. The standard uncertainty, s_N, for the 14 mV threshold setting is given by Formula (16):

$$s_N = \frac{sD_R}{\sqrt{N_C}} = \frac{466,6 \times 4,00}{\sqrt{10}} = 590,3 \tag{16}$$

Table 5 — Particle concentration data for secondary calibration samples

Count	Sample 1 X_i particles/mL	Sample 2 X_i particles/mL
1	6 376	7 312
2	6 220	7 186
3	6 507	6 866
4	6 288	7 444
5	6 321	7 036

8 Example 5: Relating particle size to threshold voltage setting for primary calibration of sizes larger than 30 µm(c)

Latex spheres are used for primary calibrations at particle sizes larger than 30 µm(c). Selection criteria for the sizes of latex to be used are specified in ISO 11171:2022, 6.11. The settings for the first four channels of the APC are defined in ISO 11171:2022, 6.12, while ISO 11171:2022, 6.14, specifies how to use the particle count data from these channels to determine the threshold setting corresponding to the particle size of the latex.

This example uses the same hypothetical APC used in previous examples and assumes the intention to calibrate to 50 µm(c). ISO 11171:2022, 6.11, states that the smallest polystyrene latex particle size "shall be between 35 µm and 45 µm". In this example, commercially available 39,33 µm latex particles, with a standard deviation of 0,5 µm, are used to meet this requirement. The next size of latex, D, "shall be approximately equal to the size of the [previous] smaller latex sphere [L] times a constant with a value between 1,1 and 1,5". Thus, its value of D is within the range given by Formula (17):

$$1,1 \times L \leq D \leq 1,5 \times L \tag{17}$$

In this example, L is 39,33 µm, hence D for the second size of latex will be between 43,3 µm and 59,0 µm. A commercially available latex with particle size of 50,2 µm meets this condition. Since its size is greater than the required largest particle size for this calibration, 50 µm(c), no additional latex particle sizes are needed. If calibration to larger sizes had been required, Formula (17) would have been used to determine the acceptable particle size range of the next largest latex size with L equal to 50,2 µm in this example and the process for selecting latex sizes repeated until the entire particle size range was covered.

The first four channels of the APC are used to determine the threshold settings corresponding to the particle sizes of the latex particles. Referring to ISO 11171:2022, 6.12, the first channel is set to 1,5 times the threshold noise level of the APC or 8 mV as determined in Example 1. The third channel is set to a threshold voltage setting that is expected to correspond to the size of the polystyrene latex spheres or 39,33 µm in this example. Since this is beyond the range of the data obtained in Example 4, the initial threshold setting for this channel is obtained by extrapolation to this size. In this case, it is estimated that 39,33 µm will correspond to a setting of approximately 3 400 mV. The second channel is set to a threshold voltage setting corresponding to 0,56 times the threshold voltage setting of the third channel or 3 400 times 0,56. The fourth channel is set to the threshold voltage setting corresponding to 1,56 times the threshold voltage setting of the third channel or 3 400 times 1,56. Thus, the first four channels are set at 8 mV, 1 904 mV, 3 400 mV and 5 304 mV. Using these settings and the APC in the differential mode, the results shown in Table 6 were obtained for the 39,33 µm latex.

Table 6 — Differential particle counts for initial analysis of 39,33 µm latex spheres

Channel	Threshold setting mV	Differential particle counts
1	8	2
2	1 904	17
3	3 400	6 310
4	5 304	not applicable

Referring to ISO 11171:2022, 6.14, the difference, *D*, in particle counts between channels 2 and 3 is given by Formula (18):

$$D=\left(1-\frac{N_2}{N_3}\right)\times 100=\left(1-\frac{17}{6\,310}\right)\times 100=99{,}7 \tag{18}$$

The absolute value of *D* is greater than 3 %, thus the threshold setting estimated for channel 3 does not correspond to the size of the latex spheres and the channels need to be readjusted. Since the value of *D* is positive, the setting for channel 3 is too low and the process repeated using a new estimated threshold setting. A value of 3 529 mV will be the new estimated threshold setting. The second, third and fourth channels are readjusted such that:

— the second channel setting is set at 3 529 × 0,56 or 1 976 mV;

— the third channel setting is set to correspond to the estimated size of the latex particles or 3 529 mV;

— the fourth channel setting is set at 3 529 × 1,56 or 5 505 mV.

Using these settings and the APC in the differential mode, the results are shown in Table 7 were obtained for the 39,33 µm latex.

Table 7 — Differential particle counts for analysis of 39,33 µm latex spheres

Channel	Threshold setting mV	Differential particle counts
1	8	3
2	1 976	3 148
3	3 529	3 172
4	5 505	NA

D can now be recalculated using Formula (19):

$$D=\left(1-\frac{N_2}{N_3}\right)\times 100=\left(1-\frac{3\,148}{3\,172}\right)\times 100=0{,}8 \tag{19}$$

The absolute value of *D* is 0,8 %, which is less than 3 %, indicating that the threshold setting of channel 3 corresponds to the size of the latex particles, i.e. a setting of 3 529 mV corresponds to 39,33 µm. This size and threshold setting is used to construct the calibration curve.

The process was repeated for 50,2 μm latex particles and it was found that a setting of 4 238 mV corresponds to this size.

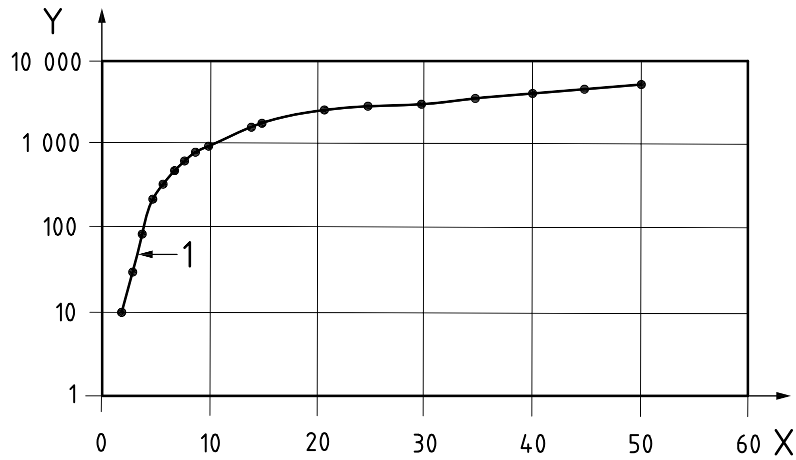
9 Example 6: Construction of calibration curve

Once particle sizes and corresponding threshold settings over the entire size range of interest have been determined, as in Examples 4 and 5, the calibration curve for the APC is constructed as detailed in ISO 11171:2022, 6.15. This clause provides a link to a Excel[1)] spreadsheet tool used to construct a calibration curve by constrained cubic spline interpolation.

This example continues to use the same APC, calibration suspensions and data as in previous examples. The particle size and threshold setting data from Examples 4 and 5 are required to be entered in order of increasing particle size into columns 1 and 2 of the worksheet from ISO 11171:2022, 6.15, as shown in Table 8. Particle sizes of interest selected from within the size range of the data are entered in column 3. The corresponding threshold voltage settings for each size are automatically displayed in column 4, and the calibration curve graphically shown in Figure 1.

Table 8 — Output from the worksheet from ISO 11171:2022, 6.15, for Example 6

Data from Table 4		Threshold settings corresponding to sizes of interest	
Enter the particle sizes and corresponding threshold settings from Table 4 (also columns F and H of the Clause 6.9 worksheet) in order of increasing value of particle size.		Particle sizes of interest (that lie within the range of the data in column A) are entered in order of increasing particle size. The corresponding interpolated threshold voltage settings are displayed in column D.	
Particle size	**Threshold setting**	**Particle size**	**Interpolated threshold voltage vetting**
μm(c)	mV	μm(c)	mV
2,00	10	2,00	10
3,00	28	3,00	28
4,00	83	4,00	83
5,00	190	5,00	190
6,00	305	6,00	305
7,00	442	7,00	442
8,00	595	8,00	595
9,00	738	9,00	738
10,00	873	10,00	873
12,00	1 176	14,0	1 479
14,00	1 479	15,0	1 642
16,00	1 805	21,0	2 362
18,00	2 105	25,0	2 515
21,00	2 362	30,0	2 600
25,00	2 515	35,0	3 048
30,00	2 600	40,0	3 581
39,33	3 529	45,0	3 926
50,20	4 238	50,0	4 226
			0
			0
			0
			0



Key

x particle size, µm(c)

y threshold voltage setting, mV

1 APC calibration curve

Figure 1 — ISO 11171:2022, 6.15, calibration curve for Example 6

10 Example 7: Calculation of coefficient variation for volume measurement using ISO 11171:2022, Clause A.8

To meet ISO 11171 APC performance specifications, the coefficient of variation for volume measurement, $C_{V,\mathrm{vol}}$, for the APC is required to be 3 % or less. ISO 11171:2022, Clause A.8, describes how this is done.

In this example, five consecutive counts for the same sample were obtained at 1,5 times the threshold noise level of the APC. Counts of 26 068 particle/mL, 26 757 particle/mL, 25 802 particle/mL, 25 834 particle/mL and 27 464 particle/mL were obtained. $C_{V,\mathrm{vol}}$ is obtained using Formulae (20), (21) and (22):

$$C_{V,\mathrm{vol}} = \frac{100}{\bar{X}} \sqrt{\frac{n\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2}{n(n-1)}} \qquad (20)$$

$$C_{V,\mathrm{vol}} = \frac{100}{\bar{X}} \sqrt{\frac{5\left(26\,068^2 + 26\,757^2 + 25\,802^2 + 25\,834^2 + 27\,464^2\right) - \left(26\,068 + 26\,757 + 25\,802 + 25\,834 + 27\,464\right)^2}{5(5-1)}} \qquad (21)$$

$$C_{V,\mathrm{vol}} = \frac{100}{26\,385} \sqrt{\frac{(5 \times 3\,482\,887\,729) - (131\,925)^2}{20}} = 2{,}71 \qquad (22)$$

where

X_i is particle concentration in particles/mL for acceptable particle count i;

$\bar{X}$ is mean particle concentration, in particles per millilitre, for the five counts;

n is number of acceptable counts obtained at indicated threshold voltage setting = 5.

In this example, the $C_{V,\mathrm{vol}}$ is 2,71 %, less than the required 3 %, so the APC meets this APC performance specification.

11 Example 8: Determination of coincidence error limit

The coincidence error limit of an APC is the highest concentration of United States National Institute of Standards and Technology (NIST) standard reference material RM 8632x that can be counted with an error of less than 5 % resulting from the presence of more than one particle in the sensing volume of the sensor at a time. The procedure for determining the coincidence error limit of an APC is found in ISO 11171:2022, Annex B.

In this example, an APC with a manufacturer estimated coincidence error limit, X_M, of 14 000 particles/mL and a NIST RM 8632a concentrate with a mass concentration, γ, of 100 mg/l is used.

Using ISO 11171:2022, Table A.1, the expected particle concentration, X_A, of the concentrate at 2 µm(c) is 2 021 000 particles/mL. A sample volume, V_S, of 400 mL is prepared for each gravimetric concentration to be analysed. The volume of concentrate required to make 400 mL of each gravimetric level required by ISO 11171 in this example is given by Formula (23):

$$V_1 = \frac{X_M V_S L}{100 \times X_A} = \frac{14\,000 \times 400 \times L}{100 \times 2\,021\,000} \tag{23}$$

where L is the percentage of X_M required to be analysed for a specific sample.

Values of L ranging from 10 % to 150 % in 10 % increments are required. Table 9 shows the value of V_1 for each value of L and the corresponding values for γ and the observed mean particle number concentration, $\bar{X}$, obtained for each sample in this example.

Table 9 — Data for determination of coincidence error limit

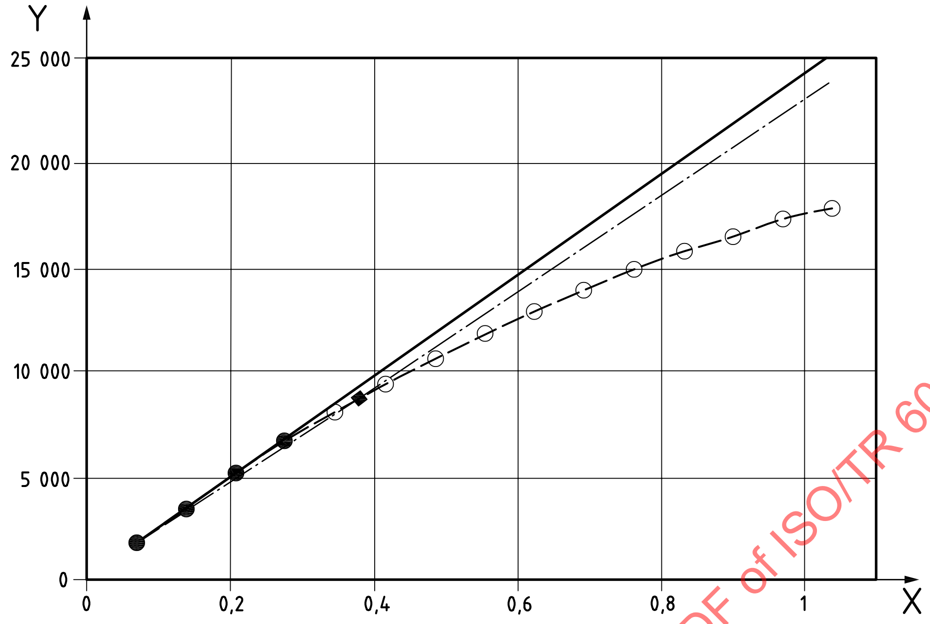
L	γ	V_1	$\bar{X}$	X_t	E
%	mg/l	mL	particles per mL	particles per mL	%
10	0,07	0,28	1 744	1 679	3,89
20	0,14	0,55	3 423	3 358	1,91
30	0,21	0,83	5 112	5 037	1,49
40	0,28	1,11	6 612	6 716	−1,56
50	0,35	1,39	8 015	8 395	−4,54
60	0,42	1,66	9 364	10 075	−7,05
70	0,48	1,94	10 593	11 754	−9,88
80	0,55	2,22	11 768	13 433	−12,40
90	0,62	2,49	12 853	15 112	−14,95
100	0,69	2,77	13 872	16 791	−17,38
110	0,76	3,05	14 815	18 470	−19,79
120	0,83	3,33	15 688	20 149	−22,14
130	0,90	3,60	16 458	21 828	−24,60
140	0,97	3,88	17 245	23 507	−26,64
150	1,04	4,16	17 777	25 186	−29,42

The theoretical relationship between mass concentration and particle concentration is a straight line whose slope is determined by linear regression using only the values corresponding to L equals 10 %, 20 %, 30 % and 40 %. The resultant linear regression equation is of the form shown in Formula (24):

$$\bar{X} = a\gamma = 24\,239\gamma \tag{24}$$

where a is the slope of the regression equation with the Y-intercept set to 0.

The value of a in this example is 24 239. The individual values for γ and $\bar{X}$ for all the samples as well as the theoretical relationship between γ and $\bar{X}$ are plotted as shown in Figure 2.



Key

X γ, mg/l

Y $\bar{X}$, particle/mL > indicated size

● data point used to define theoretical relationship

——— theoretical relationship between γ and $\bar{X}$

—·— 95 % slope line

○ data point

— — — actual relationship between γ and $\bar{X}$

◆ intersection of actual relationship and 95 % slope line

Figure 2 — Coincidence error plot for Example 8

Using Formula (24), the theoretical concentration X_t for each value of γ can be calculated. These results are shown in column 5 of Table 9 and plotted in Figure 2. The difference between X_t and $\bar{X}$ expressed as a percentage E can be calculated using Formula (25):

$$E = \frac{\bar{X} - X_t}{X_t} \times 100 \tag{25}$$

These results shown in column 6 of Table 9 are used to determine the coincidence error limit of the APC. A line with a slope of 0,95a, 23 027 in this example, is plotted in Figure 2. The value of $\bar{X}$ where the 95 % line and the curve representing the actual relationship between γ and $\bar{X}$ intersect defines the coincidence error limit of the APC in particles/mL. In this example the coincidence error limit is shown as point A in Figure 2 and is 8 650 particles/mL.

12 Example 9: Determination of flow rate limits using ISO 11171:2022, Annex C

ISO 11171:2022, Annex C, is used to determine the flow rate limits of the APC and to verify that it meets the flow rate performance specifications of the standard. There are two general types of APC: those whose flow rate can be adjusted and those that provide a single constant flow rate. To generate valid

