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COMPARATIVE STUDY OF DPD REAGENTS FOR CHLORINE MEASUREMENT IN DRINKING WATER AND DEVELOPMENT OF A JAVASCRIPT INTERPOLATION TOOL

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ABSTRACT

Background: Determining chlorine in water ensures safety. Among other methods, the DPD colorimetric method is used. The DPD Method relies on colorimetric reactions to measure free and total chlorine concentration in water samples with pink compound formation**. Aims:** To perform a comparative chlorine analysis using DPD, assessing reagents from 3 makers and 2 Hach instruments to identify disparities and propose adjustments for more accurate measurements. **Methods**: Hach High-Range and Low-Range Free chlorine determination procedures were followed. DR300 and POCKET Colorimeter II spectrophotometers were used. Tests were conducted for each DPD manufacturer in low/high ranges and in two HACH devices to determine the chlorine concentrations. Hach was used as the reference; LaMotte and PoliControl compared against it. Statistical analyses were compiled using MS Excel. **Results:** The tests findings were gathered in Tables 1-5. JavaScript and HTML scripts were created to convert LaMotte and PoliControl outcomes into values equivalent to those of HACH through linear interpolation. **Discussion:** Various DPD reagents and equipment provided slightly different readings, prompting empirical evaluation of these differences. Adjusting the results to Hach's results was selected as both the reagent and spectrophotometer were from the same brand. Differences in spectrophotometer readings were more pronounced in high-range tests nearing the upper limit of the test. **Conclusions:** Equipment variations caused minor result differences; DPD reagents are not interchangeable without correlation. The Open-source code developed aided in reducing reading disparities.

Keywords: *DPD chlorine determination, comparative analyses, linear interpolation, water quality, water safety*.

1. INTRODUCTION:

Determining chlorine in water is crucial for ensuring water safety and quality. Various methods are employed for this purpose, including the DPD method, which relies on colorimetric reactions, iodometric titration for precise measurement, nephelometric method detecting turbidity due to chlorine reactions, chlorine ion-selective electrode for direct ion measurement, mercurimetric titration providing accurate quantification, and argentometric method utilizing silver nitrate reactions. These methods play a vital role in monitoring disinfection byproducts, ensuring compliance with regulatory standards, and safeguarding public health by accurately assessing chlorine levels in water sources, thereby contributing significantly to maintaining safe drinking water for communities worldwide.

DPD method (N,N-dietyl-p- phenylenedia_ mine): From the manufacturer Hach website (https://www.hach.com/p-dpd-free-chlorine-reagent-powder-pillows-10-ml-pk100/2105569), the sources "Chlorine, Free and Total, High Range" (2022), and "Chlorine, Free and Total, Low Range" (2022) were retrieved. The DPD method is widely used for measuring free residual chlorine in water. It relies on the reaction between free chlorine and the DPD reagent, forming a pink-colored compound proportional to the chlorine concentration. The color intensity is measured either spectrophotometrically or using colorimetric kits to quantify the chlorine level. The primary reagents involved are the DPD reagent and the sample containing free chlorine. (Moberg, L., and Karlberg, B., 2000; WILDE, E.1991)

The DPD (reagent) was added to the water sample and was oxidized by chlorine in the sample to two oxidation products, Würster dye and imine. Würster dye was relatively stable and would form a magenta color at neutral pH. At the same time, imine was relatively unstable and colorless, which would be formed at higher oxidant levels, i.e., higher chlorine concentration. The intensity of the magenta color was then measured photometrically, representing chlorine concentration in the samples. The DPD-chlorine reactions are illustrated in Figure 1. (Astuti, M. P., Xie, R., & Aziz, N. S. (2017).

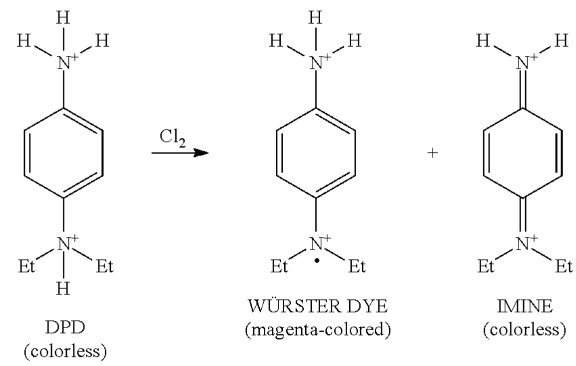

Figure 1. *Image source: US20150050195A1.*

Iodometric titration: According to ISO 7393-3:1990, and the method 4500-Cl B. iodometric method I. Iodometric titration is employed for determining chlorine levels by titrating a sample containing chlorine with a standardized iodine solution. Chlorine oxidizes iodide ions to form iodine, which is then titrated with a thiosulfate solution to calculate the chlorine concentration. Reagents include the sample with chlorine, iodine solution, sodium thiosulfate solution, starch indicator (to detect the endpoint), and sometimes potassium iodide as a catalyst.

Nephelometric method: The nephelometric method measures turbidity resulting from the reaction between residual chlorine and a specific reagent, causing solid particle formation. The turbidity produced correlates with the chlorine concentration (Lamb, Carleton, and Meldrum, 1920; Coll, 1957). The primary reagents used in this method are the sample, the specific reagent causing particle formation, and sometimes buffer solutions to maintain pH.

Chlorine ion-selective electrode: From ASTM D512-04, test method C, the chlorine ion-selective electrode directly measures chloride ions in a solution. It operates based on the selective response of the electrode to chloride ions, generating an electrical potential proportional to their concentration. This potential is measured and used to determine the chloride ion concentration. The main reagents involved in this method are the sample solution containing chloride ions and the chlorine ion-selective electrode. Unlike titration-based methods, this technique does not consume reagents but depends on the functioning of the electrode to measure ion concentrations.

Mercurimetric titration: ASTM D512-04, Test method A, informs that the Mercurimetric titration quantifies chloride ions in a solution by titrating it with a standard mercuric nitrate solution. The formation of a white precipitate of mercury(II) chloride indicates the endpoint. Reagents include the sample with chloride ions, a standardized mercuric nitrate solution, and occasionally an indicator to signal the endpoint.

Argentometric method: ASTM D512-04, test method B, informs that this method relies on the reaction between chloride ions and a standardized solution of silver nitrate ($AgNO_3$), forming a white precipitate of silver chloride (AgCl) that marks the endpoint of the titration. The sample containing chloride ions is titrated with a standardized solution of silver nitrate. Potassium chromate (K_2CrO_4) or potassium dichromate ($K_2Cr_2O_7$) is commonly used as an indicator to detect the endpoint, which is signaled by the formation of a reddish-brown color due to the reaction between the indicator and the excess silver ions after all chloride ions have reacted. Reagents include a standardized silver nitrate ($AgNO_3$) solution as the titrant, potassium chromate or potassium dichromate as an indicator to detect the endpoint, and the sample solution containing chloride ions to be quantified.

This research aims to conduct a comparative analysis of chlorine determination using the DPD method. This study involves evaluating the analytical results obtained from reagents of three different manufacturers and the use of two different analytical instruments from Hach. By carefully examining the results derived from these variations in reagents and instruments, this study aims to identify potential disparities or discrepancies in chlorine level measurements. Additionally, if necessary, it seeks to propose adjustments to reduce the identified variations, aiming to enhance the accuracy and consistency of chlorine measurements obtained with the DPD method.

2. MATERIALS AND METHODS:

2.1. Materials

- DPD Total Chlorine Reagent Powder Pillow (Hach);
- DPD Total Chlorine Reagent Powder Pillow (PoliControl);
- Chlorine DPD (6903A) (LaMotte);
- DR300 Pocket Colorimeter, Chlorine (Equipment);
- POCKET Colorimeter II Colorimeter for Chlorine analysis (Equipment);
- Chlorinated Water samples;
- Computer;
- Stopwatch;

2.2. Methods

2.2.1 DPD Chemical Methods

Hach procedures for the determination of chlorine were followed from “Chlorine, Free and Total, High Range" (2022), and "Chlorine, Free and Total, Low Range" (2022).

Two categories of samples were gathered for thorough analysis. The low-range free chlorine concentration of the samples was obtained from a chlorinated water sample prepared in the laboratory, which measured approximately 0.68 mg/L of chlorine, while the high-range free chlorine concentration of the samples was obtained from a sample prepared in the laboratory and its concentration measured around 5.80 mg/L of chlorine.

A blank sample was prepared for each device, and for each sample range by rinsing a sample cell and its cap three times with the respective sample. Each cell was placed into its respective reading device and the ZERO button (blue in both equipment) was pressed, and the display showed “0.00”.

Subsequently, the cell was filled with the sample up to the 10-milliliter mark. This meticulous procedure was consistently followed for both pieces of equipment utilized, namely the DR300 Pocket Colorimeter and POCKET Colorimeter II Colorimeter for Chlorine analysis, as presented in Figure 2.

Figure 2. DR300 Pocket Colorimeter (left) and POCKET Colorimeter II Colorimeter for Chlorine analysis.

Following this, a series of tests were conducted for each DPD manufacturer (Hach, PoliControl, LaMotte), as in Figure 3. Five tests were made for each DPD manufacturer in the low range (L.R.) in each equipment, and five tests were made for each DPD manufacturer in the high range (H.R.) in each equipment, as summarized in Figure 4.

For each test, a water sample was acquired and placed in the cell, and a single DPD pillow (or equivalent) was employed for measurement. The chlorine content registered in the equipment corresponded proportionally to the chlorine content present in the sample.

The HACH DPD manufacturer was considered the reference because HACH was the manufacturer of the reading devices. The other products from LaMotte and PoliControl were compared against it.

Figure 3. 1- *LaMotte DPD, 2- Hach DPD, and 3- PoliControl DPD*.

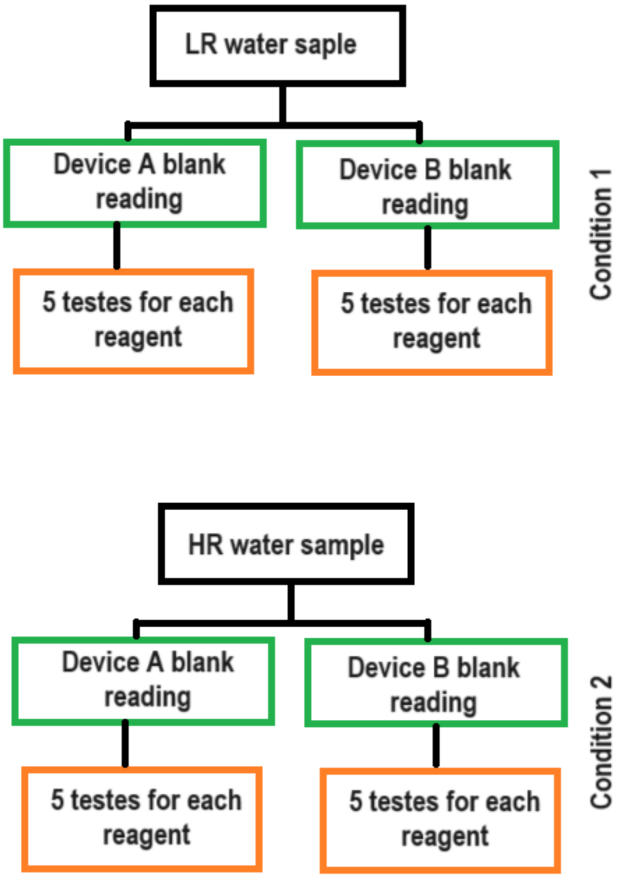

Figure 4. Test model.

2.2.2 Statistical methods

The test analysis data were collected and compiled into Tables 1 to 4 for subsequent MS-Excel statistical analyses. Later, the statistical analyses were used to write a linear interpolation script to present the analytical results of the DPD reagents from PoliControl and LaMotte in an equivalent form of the Hach DPD reagent. Table 5 represents the differences among the different models of spectrophotometers.

The equations used to calculate the Average and standard deviation came from MS-Excel functions: MED(XX:X) and DESVPAD.A(XX:X) (the names of the functions are in Portuguese).

The Percentage Difference was calculated using Equation 1. It was written in M.S. Excel as “=ABS((B1 - A1) / A1) * 100”. The ABS function in Excel stands for "Absolute Value.", and It was used to return the absolute (positive) value of the result.

$$\text{Percentage Difference} = \left|\frac{\text{New Value} - \text{Old Value}}{\text{Old Value}}\right| \times 100$$

(Eq. 1)

3. RESULTS AND DISCUSSION

3.1. Results

3.1.1. Analytical Results

Tables 1 to 4 sumarize the statistical results from the tests done using the different DPD reagents.

Table 5 note the sligt variations from the use of different spectrophotometers.

Table 5. Variation of the readings in the different equipment.

High range reading	Hach	Policontrol	LaMotte
pocket colorimeter II Average	0,67	0,67	0,52
DR300 Average	0,67	0,66	0,52
Equipment difference (%)	0	1,49	0
High range reading	**Hach**	**Policontrol**	**LaMotte**
pocket colorimeter II Average	5,8	6	4,7
DR300 Average	6,2	6,4	5
Equipment difference (%)	6,90	6,67	6,38

3.1.2.1. *HTML / Javascript code for linear interpolating the results from the* Policontrol reagent using the Hach reagent as “reference.”

```
<!DOCTYPE html>
<!--
This code is licensed under CC BY 4.0
(https://creativecommons.org/licenses/by
/4.0/)
You are free to share and adapt the
code, but you must provide appropriate
credit to the original author.
DOI:
10.48141/SBJCHEM.v31.n36.2023_TREIN_pgs_
33_45.pdf
-->
<html lang="en">
  <head>
    <meta charset="utf8_general_ci">
    <title>Linear Interpolation</title>
```

```
    <style>
      body {
      font-family: Arial, sans-serif;
      display: flex;
      justify-content: center;
      align-items: center;
      height: 100vh;
      margin: 0;
      }
      h1 {
      text-align: center;
      }
      label,
      input,
      button {
      display: block;
      margin: 10px auto;
      text-align: center;
      }
      input,
      button {
      padding: 8px;
      border-radius: 5px;
      border: 1px solid #ccc;
      width: 200px;
      }
      button {
      cursor: pointer;

      background-color: #007bff;
      color: white;
      transition: background-color 0.3s
ease;
      }
      button:hover {
      background-color: #0056b3;
      }
      p {
      text-align: center;
      margin-top: 20px;
      }
      #resultado {
      font-weight: bold;
      }
    </style>
  </head>
  <body>
    <div style="text-align: center;">
      <h1>Linear Interpolation</h1>
      <label for="valorB">

      Please insert the value of the
reagent "Policontrol":<br>
      </label>
      <input type="number"
id="valorB"><br><br>
      <button
onclick="calculateResult()">
      Calculate
      </button>
      <p>
The adjusted result of the value, to be
equivalent to the Hach product, is:
<span id="resultado"></span>
        </p>
      </div>
      <script>
        function calculateResult() {
// Reference values
const x = [0.67, 5.80];
const secondLineValues = [0.67, 6.0];
// Get the value entered by the user for
B
const valueB =
parseFloat(document.getElementById('valo
rB').value);

// Perform linear interpolation
const result =
linearInterpolation(secondLineValues, x,
valueB);

// Display the result on the page
document.getElementById('resultado').tex
tContent = result.toFixed(2);
        }

        function linearInterpolation(x, y,
value) {
const [x0, x1] = x;
const [y0, y1] = y;

// Linear interpolation formula
const result = y0 + ((value - x0) * (y1
- y0)) / (x1 - x0);

return result;
        }
      </script>
  </body>
</html>
</html>
```

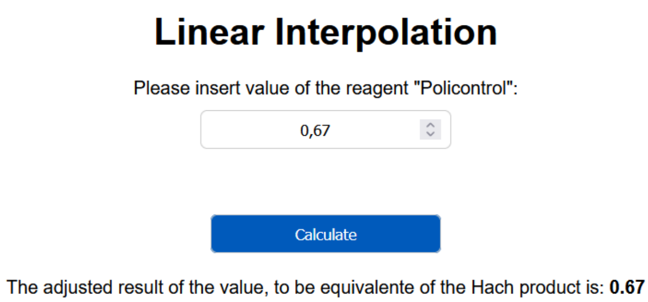

Figure 5. *The expected interface of the code from item 3.1.2.1. HTML / Javascript code for the linear interpolation of the results from the Policontrol.*

To run the code, please visit https://acaria.org/codes/code5.htm.

3.1.2.2. HTML / Javascript code for linear interpolating the results from the LaMotte reagent using the Hach reagent as "reference."

```
<!DOCTYPE html>
<!--
This code is licensed under CC BY 4.0
(https://creativecommons.org/licenses/by/4.0/)
You are free to share and adapt the
code, but you must provide appropriate
credit to the original author.
DOI:
10.48141/SBJCHEM.v31.n36.2023_TREIN_pgs_33_45.pdf
-->
<html lang="en">
  <head>
    <meta charset="utf8_general_ci">
    <title>Linear Interpolation</title>
    <style>
      body {
      font-family: Arial, sans-serif;
      display: flex;
      justify-content: center;
      align-items: center;
      height: 100vh;
      margin: 0;
      }
      h1 {
      text-align: center;
      }
      label,
      input,
      button {
      display: block;
      margin: 10px auto;
      text-align: center;
      }
      input,
      button {
      padding: 8px;
      border-radius: 5px;
      border: 1px solid #ccc;
      width: 200px;
      }
      button {
      cursor: pointer;

      background-color: #007bff;
      color: white;
      transition: background-color 0.3s
ease;
      }
      button:hover {
      background-color: #0056b3;
      }
      p {
      text-align: center;
      margin-top: 20px;
      }
      #resultado {
      font-weight: bold;
      }
    </style>
  </head>
  <body>
    <div style="text-align: center;">
      <h1>Linear Interpolation</h1>
      <label for="valorB">

      Please insert the value of the
reagent "LaMotte":<br>

      </label>
      <input type="number"
id="valorB"><br><br>
      <button
onclick="calculateResult()">
      Calculate
      </button>
      <p>
The adjusted result of the value, to be
equivalent to the Hach product, is:
<span id="resultado"></span>
      </p>
    </div>
    <script>
      function calculateResult() {
// Reference values
const x = [0.67, 5.8]; // Values from
Hach
const secondLineValues = [0.52, 4.7];//
Values from LaMotte

// Get the value entered by the user for
B
const valueB =
parseFloat(document.getElementById('valorB').value);

// Perform linear interpolation
const result =
linearInterpolation(secondLineValues, x,
valueB);

// Display the result on the page
document.getElementById('resultado').textContent = result.toFixed(2);
      }

      function linearInterpolation(x, y,
value) {
const [x0, x1] = x;
const [y0, y1] = y;

// Linear interpolation formula
const result = y0 + ((value - x0) * (y1
- y0)) / (x1 - x0);

return result;
      }
    </script>
  </body>
</html>
```

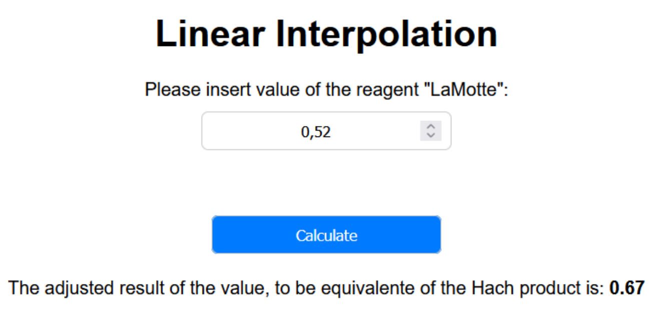

Figure 6. *The expected interface of the code from item 3.1.2.2. HTML / Javascript code for the linear interpolation of the results from the LaMotte*.

To run the code, please visit https://acaria.org/codes/code6.htm.

3.2. Discussions

3.2.1. Different results for the same sample

Unsurprisingly, the DPD reagents from different suppliers or the different types of equipment provided slightly different readings. However, it was relevant to infer values, or magnitudes, to the differences empirically observed.

As it is commonly known, one of the best methods to determine the chlorine concentration is the Argentometric method (ASTM D512-04, Test method B). However, the method has its challenges and limitations that make it unattractive when it is necessary to perform the analyses of hundreds of samples daily. So, it is worth pointing out that the goal of the research was not to find the best method to determine the chlorine concentration in water samples but to observe if there were variations in the same method with different manufacturers of the DPD reagent and types of equipment.

Additionally, it is relevant to observe that if the experimental conditions were different, other results would be found, such as if the spectrophotometer were from a different brand; the results that were considered the most accurate could be others. Therefore, the authors are not saying that the quality of reagent "A" or "B" is better or worse than reagent "C", but that in any serious company, the acquisition department can not buy the DPD reagents as if they were Interchangeable.

3.2.2. Adjusting the different results to be expressed in terms of the Hach result.

The authors chose to express the analytical results of the study in terms of the Hach DPD reagent because it was available for the study both the reagent and the spectrophotometer from the same brand.

To do this representation a simple linear interpolation was performed, and the mathematical aspect was represented in a friendly format using HTML and JavaScript code.

3.2.3. Different results in different spectrophotometers from the same brand

The differences among the two spectrophotometer models in the low-range sample reading were less representative than in the high-range. This increased variation in the high-range tests was also expected since was approaching the upper limit of the test method.

3.2.4. Practical implications of the different results

Real-world applications may face some challenges, such as in the low-range readings; depending on the test conditions, the test results with low values may induce a water treatment plant operator to unnecessarily increase the addition of chlorine, increasing the cost of operation. A simple solution to this possible issue would be the production of a linear correlation chart between the Argentometric method and the conditions of the DPD test method being used in this hypothetical situation.

3.2.5. Practical implications of the different results

Table 6. *Representation of the* 95% confidence intervals of different DPD reagents and equipment.

Confidence intervals LR pocket colorimeter II					
Hach		Policontrol		LaMotte	
From	To	From	To	From	To
0,66	0,70	0,66	0,68	0,51	0,53
Confidence intervals LR DR300					
0,66	0,70	0,65	0,67	0,51	0,52
Confidence intervals HR pocket colorimeter II					
5,71	5,89	5,87	6,09	4,65	4,79
Confidence intervals HR DR300					
6,11	6,33	6,31	6,45	4,95	5,09

Table 6 outlines the 95% confidence intervals of sample readings for different DPD reagents and equipment combinations. With a 95% confidence level, the true mean of the population readings is estimated to be within these intervals.

4. CONCLUSIONS:

Reading the same chlorinated water samples using different equipment resulted in slight variations in test outcomes. DPD reagents from various manufacturers are not entirely interchangeable and must not be utilized without proper correlation against a reference standard. The open-source codes developed for linear interpolation of obtained data yielded satisfactory values and reduced the disparity in the results.

5. DECLARATIONS

5.1. Study Limitations

The study is limited to the sample size, materials (DPD from different manufacturers), and experimental conditions.

5.2. Acknowledgements

Chat GPT and Google Bard generated part of this manuscript's text and codes.

5.3. Funding source

The authors funded this research.

5.4. Competing Interests

The authors have no financial, personal, or professional connections that might bias or influence the research, analysis, or conclusions presented in this work. Therefore we declare no conflict of interest associated with this publication.

5.5. Open Access

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4. Chlorine, Free and Total, High Range. (2022) USEPA DPD Method 1 Method DPD 0.1 to 8.0 mg/L Cl 2 Powder Pillows. (n.d.). Retrieved December 9, 2023, from https://cdn.bfldr.com/7FYZVWYB/at/xtn3qr6g6gzrvfgr3c7kg43/DOC3165301449_7ed.pdf

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Table 1. Low-Range test results in the pocket colorimeter II.

		Instantaneous reading	
Low range reading	**Hach**	**Policontrol**	**LaMotte**
Test 1	0,69	0,65	0,51
Test 2	0,67	0,66	0,53
Test 3	0,72	0,68	0,51
Test 4	0,67	0,67	0,52
Test 5	0,67	0,67	0,52
Statistics	-	-	-
Mean	0,684	0,666	0,518
Standard error	0,009798	0,00509902	0,0037417
Median	0,67	0,67	0,52
Mode	0,67	0,67	0,51
Standard deviation	0,0219089	0,011401754	0,0083666
Sample variance	0,00048	0,00013	7E-05
Kurtosis	1,7447917	-0,177514793	-0,6122449
Skewness coefficient	1,5309606	-0,404796009	0,5122408
Range	0,05	0,03	0,02
Minimum	0,67	0,65	0,51
Maximum	0,72	0,68	0,53
Sum	3,42	3,33	2,59
Count	5	5	5
Confidence level (95.0%)	0,0272035	0,014157148	0,0103885
% of the difference of the mean from the HACH		2,63%	24,27%

Table 2. Low-Range test results in the DR300 Pocket Colorimeter.

		Instantaneous reading	
Low range reading	**Hach**	**Policontrol**	**LaMotte**
Test 1	0,68	0,64	0,52
Test 2	0,71	0,66	0,52
Test 3	0,67	0,67	0,51
Test 4	0,67	0,67	0,52
Test 5	0,67	0,66	0,5
Statistics	-	-	-
Mean	0,68	0,66	0,514
Standard error	0,007746	0,005477226	0,004
Median	0,67	0,66	0,52
Mode	0,67	0,66	0,52
Standard deviation	0,0173205	0,012247449	0,0089443
Sample variance	0,0003	0,00015	8E-05
Kurtosis	3,6666667	2	0,3125
Skewness coefficient	1,9245009	-1,360827635	-1,2577882
Range	0,04	0,03	0,02
Minimum	0,67	0,64	0,5
Maximum	0,71	0,67	0,52
Sum	3,4	3,3	2,57
Count	5	5	5
Confidence level (95.0%)	0,0215063	0,015207216	0,0111058
% of the difference of the mean from the HACH		2,94%	24,41%

Table 3. The high-range test results from the pocket colorimeter II.

		Instantaneous reading	
High range reading	**Hach**	**Policontrol**	**LaMotte**
Test 1	5,7	6	4,8
Test 2	5,8	5,9	4,7
Test 3	5,7	6,1	4,7
Test 4	5,9	5,8	4,8
Test 5	5,9	6,1	4,6
Statistics	-	-	-
Mean	5,8	5,98	4,72
Standard error	0,0447214	0,058309519	0,0374166
Median	5,8	6	4,7
Mode	5,7	6,1	4,8
Standard deviation	0,1	0,130384048	0,083666
Sample variance	0,01	0,017	0,007
Kurtosis	-3	-1,487889273	-0,6122449
Skewness coefficient	2,22E-14	-0,541387051	-0,5122408
Range	0,2	0,3	0,2
Minimum	5,7	5,8	4,6
Maximum	5,9	6,1	4,8
Sum	29	29,9	23,6
Count	5	5	5
Confidence level (95.0%)	0,1241664	0,161893178	0,1038851
% of the difference of the mean from the HACH		3,10%	18,62%

Table 4. High-range test results in the DR300 Pocket Colorimeter.

		Instantaneous reading	
High range reading	**Hach**	**Policontrol**	**LaMotte**
Test 1	6,1	6,4	5,1
Test 2	6,2	6,3	4,9
Test 3	6,1	6,4	5
Test 4	6,4	6,3	5,1
Test 5	6,3	6,5	5
Statistics	**-**	**-**	**-**
Mean	6,22	6,38	5,02
Standard error	0,0583095	0,037416574	0,0374166
Median	6,2	6,4	5
Mode	6,1	6,4	5,1
Standard deviation	0,130384	0,083666003	0,083666
Sample variance	0,017	0,007	0,007
Kurtosis	-1,4878893	-0,612244898	-0,6122449
Skewness coefficient	0,5413871	0,512240833	-0,5122408
Range	0,3	0,2	0,2
Minimum	6,1	6,3	4,9
Maximum	6,4	6,5	5,1
Sum	31,1	31,9	25,1
Count	5	5	5
Confidence level (95.0%)	0,1618932	0,103885063	0,1038851
% of the difference of the mean from the HACH		2,57%	19,29%