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1 **Comparison of the Fajans and Mohr Techniques for** 2 **the Titration of Chloride Ions and Salinity** 3 **Determination**

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8 Key Words: Mohr, Fajans, dichlorofluorescein, chromate-dichromate, chlorinity, dextrin,
9 titration, seawater analysis, endpoint detection

10 ***Abstract***

11 The concentration of halides in solution, chloride being the most commonly analyzed,
12 can be determined by either a Mohr titration, using a chromate-dichromate (CHR-DC) indicator,
13 or a Fajans titration, using a dichlorofluorescein (DCF) indicator. When titrating sodium chloride
14 (NaCl) solutions with known chloride concentrations, the Fajans procedure yielded results within
15 0.1% of the indicated concentrations. When the Mohr and Fajans titrations were performed on
16 IAPSO Standard Seawater (IAPSO-SS), the results of the two techniques were practically
17 identical, within 0.003 units of each other. This strongly suggests that either of these techniques
18 can be used for the determination of chloride or other halides in a solution with certainty that one
19 will get comparable results. Due to better precision, ease of endpoint detection, and the less
20 hazardous nature of the reagents, the Fajans technique would be preferred over the Mohr
21 technique. The silver nitrate (AgNO₃) solutions used for these titrations were standardized
22 against solutions of NaCl. The two techniques yielded concentrations for the AgNO₃ that
23 differed by 0.29%, with the concentration determined using the Fajans indicator always higher.
24 For the Fajans standardizations, the average, standard deviation, and % RSD were $0.3519 \pm$
25 0.0006 mol/kg-soln, and 0.17%. For the Mohr titrations, the values were 0.3509 ± 0.0010 , and

26 0.28% respectively. The higher standard deviation for the Mohr titration data could be attributed
27 to a less defined endpoint. Based on this data and the degree of agreement between the two
28 techniques, it is important that the standardizations and sample titrations use the same indicator.

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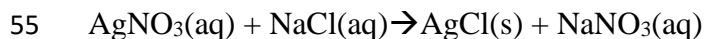
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52 Either the Mohr or Fajans titration methods can be used to determine the concentration of halides in a
53 solution, most commonly chloride, using AgNO_3 as the titrant. The reaction for chloride is:

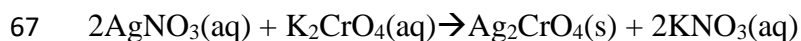
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57 Karl Friedrich Mohr, a German analytical chemist, born in 1806,
58 focused his studies on volumetric analyses (Scott 1950). He
59 developed his titration technique for chloride in solution in 1855.
60 He used a potassium chromate (K_2CrO_4) indicator for the titration of
61 halides. Now a $\text{K}_2\text{Cr}_2\text{O}_7$ indicator is suggested (Vogel 1989). In
62 the titration, Ag^+ and Cl^- react to form AgCl . Then excess silver
63 ions react with the indicator to form an insoluble red salt (Fig. 1).
64 The equation for the reaction with potassium chromate, the
65 dominant reagent of the indicator, is:

66



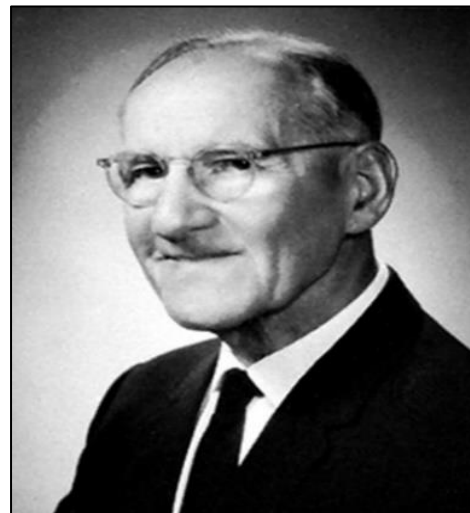
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69 The formation of the salt produces a gradual color change to a darker shade of reddish brown, which
70 signals the endpoint of the titration. A blank determination with the indicator alone in deionized water
71 must be determined.

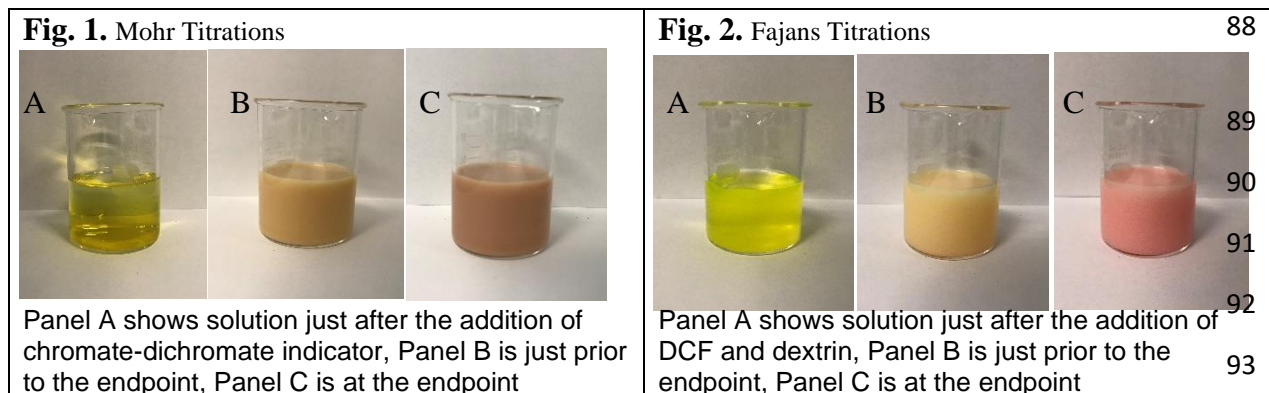
72 Kasimir Fajans, a Polish American physical chemist, born in 1887, primarily studied radioactivity
73 (Holmen 1989). In 1923, 68 years after the publication of the Mohr technique, Fajans published his
74 method of titration using an organic DCF adsorption indicator. In the titration, precipitated AgCl adsorbs



75 chloride ions to form a primary adsorption layer. After the
 76 equivalence point, excess silver ions are primarily adsorbed.
 77 Next, the negatively charged fluorescein component of the
 78 indicator forms a secondary adsorption layer on the surface of the
 79 precipitate, creating a pink-colored complex with the silver ions.
 80 The pink complex produces a distinct color change from greenish
 81 yellow to pink (Fig. 2). This analysis also requires a blank, which
 82 involves titrating various amounts of NaCl.



83 Though both techniques can be used to determine the concentration of chloride ions in a solution,
 84 little work has been performed to quantify the differences in the chloride ion concentration yields using
 85 the two different indicators. Using solutions of NaCl and IAPSO-SS, the accuracies and the precisions
 86 of the two techniques were investigated. Observations regarding the ease of endpoint detection and the
 87 blank determinations are detailed below.



94

95 **Materials**

96 Most chemical compounds required for the titrations were purchased from Acros Organics and Fisher
 97 Scientific. The ethanol and the IAPSO-SS were purchased from Koptec and OSIL, respectively. The
 98 balances were purchased from AND, Mettler Toledo, and Sartorius. The repeating pipettor and titrator

99 were purchased from Eppendorf and Metrohm. The digital density meter was purchased from Rudolph
100 Research Analytical. Full reagent and equipment information is provided in Appendix I.

101

102 ***Reagent Preparations***

103 NaCl solutions of approximately 0.6 mol/kg solution [hereafter mol/kg-soln] (Thompson et al. 2008)
104 were prepared by adding ~8 g of recrystallized NaCl to a total weight of ~250 g with DI water (DIW).
105 The NaCl was initially oven dried at 500 degrees Celsius.

106 The indicators were prepared as detailed in Vogel's *Textbook of Quantitative Chemical Analysis*
107 (Vogel et al. 1989).

108 The Fajans indicator was prepared by dissolving 0.1 g of DCF in a total volume of 100 mL using 70
109 mL of 95% ethanol and 30 mL of DIW. The indicator was stored in a dropper bottle

110 The Mohr indicator was prepared by adding ~4.2 g of potassium chromate and ~0.7 g of potassium
111 dichromate to 100 g of DIW.

112 The AgNO₃ solution was prepared to be ~0.35 mol/kg-soln by dissolving ~60 g of AgNO₃ in ~945 g
113 of DIW, the final weight of the solution being ~1000 g. The solution was stored in a brown bottle to
114 minimize photodegradation.

115

116 ***Titration Methods***

117 The titration scheme was based on an entry level analytical chemistry course to minimize sample and
118 reagent needs. Sample sizes were typically ~1 g and AgNO₃ titrants were ~1.6 g. The Fajans procedure
119 was in accordance with "Laboratory Protocol-Chlorinity by Fajans Method" from UC San Diego's
120 Analytical Chemistry Course, 100A (Vukovic 2017).

121 Before each day's work AgNO_3 solution was rinsed through the Dosimat system to purge previous
122 solution and eliminate bubbles from the delivery lines (Metrohm 2005).

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124 *AgNO_3 Standardization*

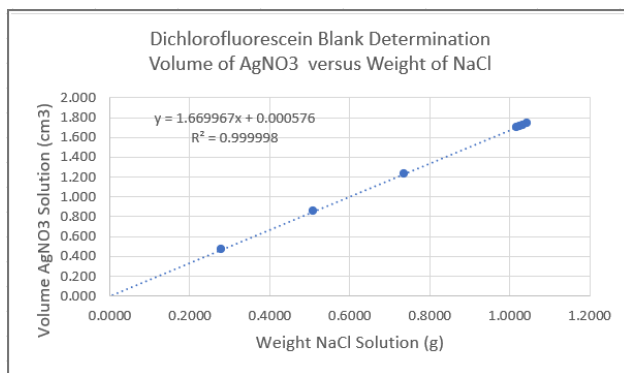
125 The AgNO_3 solution was standardized by titrating NaCl solutions multiple times with at least 3 trials per
126 technique. The concentration and standard deviation of the trials for each indicator were calculated and
127 compared.

128 *Sample Titrations*

129 For all Fajans analyses, ~1 g of either the NaCl or IAPSO-SS solution was weighed (in a capped
130 plastic pipette using a top loader balance for an approximation and an analytical balance for the final
131 weight) to 0.1 milligrams. The ~1 g of sample was added to the 100 mL titration beaker followed by
132 DIW to the 50 mL mark, ~0.05 g of Dextrin (Vogel et al. 1989) to prevent coagulation of the precipitate,
133 and 10 drops (~0.25 g) of DCF indicator. After adding a stir bar, the beaker was placed onto a magnetic
134 stir unit. The Dosimat dispensing tip, held in place using a ring stand and clamp, was then submerged in
135 the solution and the titration was started. Approximately 1.4 mL of the AgNO_3 solution was initially
136 dispensed, followed by decreasing increments from 0.05 mL to 0.001 mL. The endpoint was detected by
137 a distinct change from greenish yellow to a pink tint (Fig. 2). Since precipitate is needed for the
138 adsorption, the blank had to be determined with NaCl in the solution. The blank was determined by
139 titrating approximately 0.25, 0.50, 0.75, and 1 g of the NaCl solution with AgNO_3 . Using the titration
140 data, a calibration curve was generated (Fig. 3). The line of best fit was extrapolated to define the y-
141 intercept as the blank of ~0.0006 g.

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148 **Fig. 3.** Extrapolated line graph showing 7 different weights of NaCl solution (g) and the volume of AgNO₃ (cm³)
149 required to titrate them. The y-intercept of the equation (0.000576 g) is the blank for the Fajans titrations.

150

151 The Mohr procedure was the same as the Fajans, except no Dextrin was added and 1 mL of the CHR-
152 DC indicator was dispensed using an Eppendorf repeating pipettor. The endpoint was signaled by the
153 appearance of a very faint reddish-brown tint (Fig. 1). The blank for the Mohr titration was completed
154 by adding 1 mL of the indicator to DIW filled to the 50 mL mark of the beaker with ~0.5 grams of
155 CaCO₃ to better match the background of a titrated solution (Vogel et al. 1989). The blank was
156 consistently 0.006 cm³.

157 *NaCl Standardization-Fajans evaluation*

158 Two NaCl solutions of slightly different concentrations were prepared. One NaCl solution was used to
159 standardize the AgNO₃ solution using the Fajans method. Using the results of this standardization the
160 Fajans technique was then used to determine the concentration of the second NaCl solution. The second
161 NaCl solution concentration was calculated and compared to the known concentration. The percent
162 difference was calculated to estimate the accuracy of the Fajans technique. As part of the initial
163 evaluation of the Fajans technique, a sample of IAPSO-SS was analyzed. The salinity from the titrations
164 was compared with the given value.

165 ***Analysis of IAPSO Standard Seawater***

166 Using standardized AgNO_3 concentrations for the two techniques, IAPSO-SS was titrated using the
167 two methods. First the chlorinity (AMS, 2020), then the salinity of the seawater sample was calculated.
168 Two different salinity determinations were made, each with at least three trials to determine if the
169 differing standardization values of the two techniques affected the resulting calculated salinity values.
170 The averages and standard deviations for each technique were calculated. The calculated salinities for
171 each technique were compared to the known value.

172

173 ***Computations***

174 All analyses were performed by weight or volume, so appropriate densities were used to calculate
175 mass from the weight or blank corrected volumes.

176 The weights of dry NaCl were converted to mass using the density of the reagent. For the deionized
177 water, the density at 21 degrees Celsius was used. For the NaCl solutions, a density appropriate to the
178 concentration was calculated using data from the Handbook of Chemistry & Physics (Rumble 2022)
179 (Fig. 4). Because the temperature of the lab was constant within ± 2 degrees Celsius and the
180 concentration of the NaCl solutions were $\sim 0.6 \pm 0.5$ mol/kg-soln, values of density and molarity above
181 and below the target concentration were used. Using the provided densities, molarities were converted
182 to units of mol/kg and plotted against density. Because of the limited range of concentration these data
183 could be fit using a linear least squares fit, making subsequent computations straight forward. The same
184 procedure was used in determining the density necessary for the AgNO_3 solutions (Fig. 4). For the
185 IAPSO-SS samples, density was measured using a digital density meter.

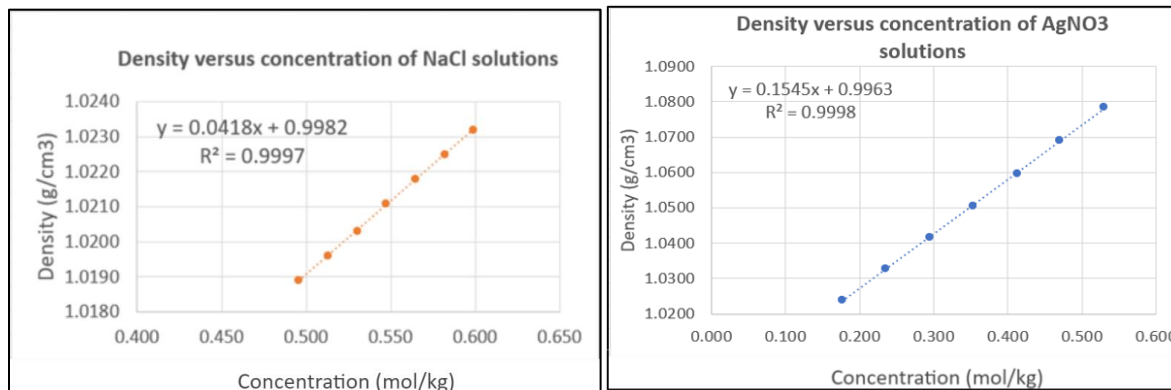
186 Blanks were determined for both techniques as described previously (for the Fajans titrations, see
187 (Fig. 3)

188

189

190

NaCl density

AgNO₃ density

191

192 **Fig. 4.** Densities of AgNO₃ and NaCl solutions (g/cm³) at 20 degrees Celsius plotted against the concentration of
 193 these solutions in units of mol/kg with a linear least squares fit.

194

195 The Dosimat titrator was calibrated (Fig. 5) using DIW; the temperature of the water for each
 196 dispensing was recorded. The weights were converted to mass and then volume. The differences
 197 between the calculated volumes and the nominal volumes were calculated. The data were plotted to
 198 determine the corrections needed to convert nominal volumes to corrected volumes. After determining
 199 the corrected volume and subtracting the blank, the volume of AgNO₃ solution was converted to mass
 200 using the appropriate density for the solution (Fig. 4).

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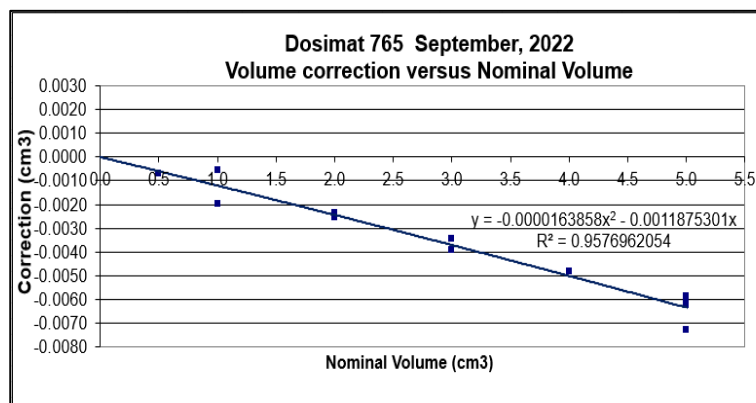
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210 **Fig. 5.** Dosimat reading for the nominal volume of DIW (cm³) versus the difference between the nominal volume
 211 and the calculated volume of DIW calculated from weight (cm³) for 11 volumes of DIW.

212

213 For the titrations of the IAPSO-SS, the weight was converted to a mass using the density determined
 214 using the digital density meter. Knowing the mass of the IAPSO-SS sample, the concentration of the
 215 AgNO_3 solution in units of mol/kg-soln, its density, and the equations found in American
 216 Meteorological Society (AMS) Glossary of Meteorology, the chlorinity of the IAPSO-SS was
 217 calculated. Using the equation relating the chlorinity of seawater to salinity, the salinity was calculated
 218 (Knudsen 1901; Riley et al. 1975).

219 $1.80655 * \text{the chlorinity} = \text{the salinity}$

220 *Assessment*

221

Table 1. AgNO_3 concentration yields (mol/kg-soln) for the Fajans and Mohr titrations of 6 NaCl solutions. The standard deviation for all standardization values and the percent difference between the two techniques for a given NaCl solution were recorded. Note that only the Fajans method was used to standardize AgNO_3 for the 4th NaCl solution.

Fajans Titrations (mol/kg-soln)	Mohr Titrations (mol/kg-soln)	% Difference Between the Values
0.3508 ± 0.0002	0.3494 ± 0.0004	0.42
0.3523 ± 0.0000	0.3516 ± 0.0003	0.21
0.3515 ± 0.0001	0.3501 ± 0.0006	0.41
0.3516 ± 0.0001	N/A	N/A
0.3527 ± 0.0002	0.3511 ± 0.0001	0.45
0.3522 ± 0.0002	0.3518 ± 0.0006	0.11

The

227 Fajans standardizations of AgNO_3 yielded average concentrations 0.29% higher than the Mohr
 228 standardizations (Table 1). The variable percent difference between the Fajans and Mohr standardization
 229 values can be attributed to the difficulty of reproducing the endpoint associated with the Mohr titration.

230 **Table 2.** NaCl solution 2 concentration (mol/kg-soln) calculated with the standardized concentration of AgNO_3
 231 from titrating NaCl solution 1 using the Fajans indicator. The standard deviations of the calculated concentrations
 232 and the percent difference between the determined NaCl solution 2 concentration and the known concentration of
 233 NaCl solution 2 were recorded.

AgNO_3 Concentration using NaCl Solution 1 (mol/kg-soln)	Concentration of NaCl Solution 2 (mol/kg-soln)	Known Concentration of NaCl Solution 2 (mol/kg-soln)	Percent Difference of Calculated and Known Solution 2 Values
0.3516 ± 0.0001	0.6500 ± 0.0001	0.6506	-0.09%

235

236 Using the Fajans titration technique to standardize the AgNO_3 concentration with one NaCl solution,
 237 then running the Fajans titration on a second NaCl solution of different concentration, the technique
 238 accurately determined the NaCl solution concentration. The percent difference between the known and
 239 determined NaCl solution two concentration was $<0.1\%$. This provided evidence that the titration
 240 technique yielded accurate results (Table 2). In a second test with the Fajans method the salinity of an
 241 IAPSO-SS was also accurately determined with the difference between the determined and accepted
 242 values of salinity being $<0.05\%$ (Table 3). This analysis was not performed with the Mohr technique
 243 since the technique has been used to determine seawater salinity for 150 years, so the accuracy was
 244 already known.

245 **Table 3.** Salinity value of IAPSO-SS determined by the Fajans analysis using standardized AgNO_3 . The
 246 standard deviation and percent difference between the Fajans and IAPSO-SS value were recorded.

Fajans Salinity Value	IAPSO Standard Adjusted Value	Percent Difference of the Fajans Value from the IAPSO-SS Value
34.983 ± 0.004	34.998	-0.043%

247

248 **Table 4.** IAPSO-SS salinity values determined by the Fajans and Mohr titrations using AgNO_3 standardization
 249 values on 2 different dates. The standard deviations and percent difference between the Fajans and Mohr salinity
 250 values were recorded.

Fajans	Mohr	IAPSO-SS Known Salinity or Adjusted Salinity	Percent Difference Fajans and Mohr
35.143 ± 0.005	35.143 ± 0.015	34.994	0.000
35.145 ± 0.014	35.143 ± 0.096	34.998	0.006

251

252 Despite the lower AgNO_3 standardization values for the Mohr titrations, both techniques yielded nearly
 253 the same salinity with an average 0.003% difference (Table 4). The fact that the differences between the
 254 determined salinity values and the accepted values are close to 0.42%, is likely the result of a NaCl
 255 solution of a concentration different than calculated, perhaps the result of preparation errors.

256

257 ***Discussion***

258 The initial analyses of NaCl solutions and a sample of IAPSO-SS provided proof that the Fajans
259 technique could give results in excellent agreement with the accepted values. Even though the AgNO₃
260 standardizations using the Fajans and Mohr techniques gave different results, the Fajans standardizations
261 being 0.29% greater than the Mohr, the two techniques gave almost identical results when running
262 samples of an IAPSO-SS. These tests indicate that the Fajans titration is an equally accurate and more
263 precise method of titration. The quantitative results, as described previously in this study [Assessment
264 section], and qualitative results, as described in this section [including differing endpoints and indicator
265 blanks], lead to the conclusion that the usage of the Fajans method for the determination of chloride ion
266 concentrations and salinity should be favored over the Mohr method.

267 The endpoints of the Fajans and Mohr titrations are vastly different. The Fajans technique results in a
268 clear shift from a greenish-yellow to a pink color at the endpoint (Fig. 2). However, the Mohr endpoint
269 has a much more gradual color change that is only a difference in shade of a reddish-brown color (Fig.
270 1). The less clear endpoint of the Mohr titration is the likely source of the decreased precision in the
271 AgNO₃ standardization results (Table 1). The lack of definition in the endpoint also leads to the Mohr
272 titration taking longer, as it required more examination between dispensing of the titrant and assessing if
273 the color change indicated the end point. If one's endpoint detection does not change from
274 standardization to the determination of an unknown, similar chloride ion concentrations and salinity will
275 be determined. Therefore, since each technique yields very similar results, the Fajans technique, with its
276 lower standard deviations and less time to complete should be favored.

277 The ~0.0006 mL blank of the Fajans titration is much smaller than the 0.006 mL blank of the Mohr
278 titration. Though smaller, the determination of the Fajans blank is much more time-consuming, requiring
279 a calibration curve with multiple weights of NaCl solution being titrated. Since the Fajans titration

280 blank is only ~ 0.0006 g, it has a small effect on the final calculated chloride ion concentrations; the error
281 in a titration of ~ 1.6 g would be approximately 0.05%, so in many cases it could be ignored.

282 When considering the hazardous nature of the reagents in the indicators, the Fajans indicator would be
283 preferred. The KCHR-KDC indicator is toxic. Chromate and dichromate are carcinogens and mutagens
284 that can cause lung and organ damage, genetic defects, and eye and skin irritation through inhalation and
285 absorption through the skin (Thermo Fisher Scientific 2018, 2021). The DCF indicator is non-hazardous
286 (Sigma-Aldrich 2019). In addition to the health hazards associated with the Mohr indicator reagents, the
287 toxicity also makes the disposal more difficult, as the two chromate reagents are harmful to sea life and
288 therefore require proper disposal through one's EH&S facility, not into the sewer.

289

290

291 *Comments and Recommendations*

292 These analyses provided insight into improved methodical proceedings in the Fajans titration. Dextrin
293 made the endpoint much easier to see by preventing coagulation of the AgCl precipitate which occurs
294 near the endpoint. Additionally, the Dosimat titrator increased the precision of the results compared to
295 the modified pipette capable of dispensing drops as small as 0.01 gram such as that used in the UCSD
296 undergraduate lab (Vukovic 2017).. Though there is a notable difference in the precision of the
297 Dosimat, using a modified pipette could still be suitable in a classroom setting where there would likely
298 be limited access to advanced pipetting instruments and less concern about a small error range.
299 Moreover, further trials using three NaCl solutions of slightly different concentrations, rather than one,
300 could reduce the possibility of preparation errors affecting results which might explain the 0.42%
301 difference between the IAPSO-SS and that calculated for both indicators.

302 Additionally, it must be noted that the AgNO_3 volumes that were recorded in the procedure account
 303 for a larger percent error than the NaCl weights. The NaCl and seawater solutions were weighed to four
 304 (4) decimal places. The Dosimat volume readings for the AgNO_3 solutions were only three (3) decimal
 305 places.

306

307

308 **References**

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- 345 by Fajans Method. UCSD

346

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353

354 *Appendix I:*

355 *Reagents*

356 Calcium Carbonate (CaCO₃), Fisher Scientific, Certified ACS, Lot 157095, 99.2% assay

357 Deionized water (DIW), 18.0 megohms

358 Dextrin, Acros Organics, Lot A0404822

359 2', 7'- Dichlorofluorescein, Acros Organics, Lot 40423367, pure

360 Ethanol, C₂H₅OH, Koptec, USP, Lot # A09042002A, 190 proof.

361 IAPSO Standard Seawater, OSIL, Batch P164, K15 = 0.99850, practical salinity = 34.994

362 In only one of the tests was a new bottle of P164 used. In the others, a previously opened bottle was used. The accepted

363 value was increased by 0.004 to compensate for the likely increase in the value, the result of evaporation and the re-

364 equilibration of the head space.

365 Potassium Chromate, CrK₂O₄, Acros Organics; Lot # A0307471, assay 99.5%

366 Potassium Dichromate ($\text{Cr}_2\text{K}_2\text{O}_7$), Acros Organics, Lot AO34082, purity 99.5%,
367 Silver Nitrate (AgNO_3), Fisher Scientific, USP, Lot 15843A
368 Sodium chloride, NaCl ; Fisher Scientific; Certified ACS crystalline, Lot # 217853, assay 99.0%
369 minimum

370

371 ***Equipment***

372 AND top loader balance, Fx-3000i, readability, 0.01 g
373 Eppendorf Repeater® plus repeating pipettor with 25 mL tip used to dispense 1 mL of the chromate-
374 dichromate indicator
375 Metrohm Dosimat, model 775, with handheld keyboard with speed and volume controls, readability,
376 0.001 mL
377 Mettler Toledo, model XP 205 analytical balance, readability, 0.01 mg
378 Mettler Toledo, model XP10002 S toploader balance, readability, 0.01 g
379 Rudolph Research Analytical, DDM 2911 Digital Density Meter, readability, 0.00001 g/cm^3
380 Sartorius analytical balance, ENTRIS2241-1SUS, readability 0.0001 g
381 Misc for titrations: beakers, kimwipes, stir bars, stir unit, etc.

382

383