

Supplement 2: *Reagent Chemicals, 10th Edition*

Updated October 13, 2010

REAGENT	PAGE	SUPPLEMENT												
Aluminum Potassium Sulfate Dodecahydrate	140	Assay. In line 10 of the test delete “zinc chloride” and replace with “EDTA.”												
Aluminum Nitrate nonohydrate	139	Assay – last line, one milliliter of 0.1M Zinc Chloride should be 0.1M EDTA.												
Aluminum Sulfate, Hydrated	142	Assay. In line 10 of the test delete “zinc chloride” and replace with “EDTA.”												
Ammonium Iodide	162	<p>Barium. Delete previous method, change as follows Barium. (By flame AAS, page 63).” “Sample Stock Solution. Dissolve 50.0 g of sample in about 70 mL of water. Transfer to a 100 mL volumetric flask, at room temperature, dilute to the mark with water, and mix (1 mL = 0.50 g).”</p> <p>Add to the table:</p> <table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th></th> <th style="text-align: center;">Wavelength (nm)</th> <th style="text-align: center;">Sample wt (g)</th> <th style="text-align: center;">Standard Added (mg)</th> <th style="text-align: center;">Flame Type</th> <th style="text-align: center;">Background</th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">Ba</td> <td style="text-align: center;">553.6</td> <td style="text-align: center;">5.0</td> <td style="text-align: center;">.10; .20</td> <td style="text-align: center;">N/A</td> <td style="text-align: center;">No</td> </tr> </tbody> </table> <p>Also, delete the word “Barium” from the p162, line reading “For the determination of Phosphate, Sulfate, Barium, Heavy Metals...”</p>		Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type	Background	Ba	553.6	5.0	.10; .20	N/A	No
	Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type	Background									
Ba	553.6	5.0	.10; .20	N/A	No									
Ammonium Nitrate	165	Add to Assay specification “≥ 95.0 %.”												
Ammonium Phosphate, Dibasic	170	Insoluble Matter Test. Delete the second sentence which begins with “Save the filtrate....”												
Ammonium Phosphate, Monobasic	172	Insoluble Matter Test. Delete the second sentence which begins with :Save the filtrate....”												
1,3-Benzenediol	199	ACS and USP Melting point harmonization. Change the lower limit from “110.0” to “109.0.”												
Cadmium Chloride, Anhydrous	221	Sulfate. In the sulfate test change the sample size from “1.0 g” to “0.5 g.”												
Cadmium Chloride, Crystals	223	Sulfate. In the sulfate test change the sample size from “2.0 g” to “1.0 g.”												

Calcium Chloride Dihydrate	238	Ammonium. Replace the entire paragraph for this test to correct several errors as cited below: “Ammonium. Dilute 10 mL (2-g sample) of sample solution A to 45 mL, and add 15 mL of 10% sodium hydroxide reagent solution. Filter through a filtering crucible previously washed with 10% sodium hydroxide reagent solution. Dilute 6 mL of the filtrate to 50 mL and add 2 mL of Nessler reagent. Any color should not exceed that produced by 0.01 mg of ammonium ion (NH ₄) in an equal volume of solution containing 1.5 mL of 10% sodium hydroxide reagent solution and 2 mL of Nessler reagent.”
Citric Acid, Anhydrous, and Citric Acid, Monohydrate	262	Sulfate Spec and Test.- rename to “Sulfur Compounds (as SO ₄).” Two places to change.
Chromotropic Acid, Disodium Salt	260	<i>Typical appearance</i> under General Description. Replace “white to off-white” by “tan to brown.”
Chromium Trioxide	258	Sodium Specification is 0.2% rather than 0.02%.
Colorimetry and Turbidimetry	39	In the equation for %NO ₃ replace equal sign in “=% maximum allowable” with times sign to read “x % maximum allowable.”
2',7'-Dichlorofluorescein	287	Adsorption instead of absorption Two places to change.
(Ethylenedinitrilo)tetraacetic	318	Specifications. In the chemical formula beside the cited Nitritotriacetic acid specification change the second subscript from “2” to “3.”
Ferric Nitrate Nonahydrate	325	Chloride Specification. In front of the Maximum Allowable specification delete the “?” sign, leaving “5 ppm.”
Formaldehyde Solution	330	Replace the current version of Note with the following version: <i>Note: Typically, this reagent contains 9-15 % v/v of methanol as a stabilizer.</i> Assay. In line 4 delete “50 mL of 1 N sodium hydroxide and”. In lines 5 and 6 delete “sodium hydroxide” and replace with “sulfuric acid”. Rewrite the calculation as cited below: $\% \text{ HCHO} = \frac{[(\text{sample titer} - \text{blank titer})] \times N \text{ H}_2\text{SO}_4 \times 3.003}{\text{sample wt (g)}}$

		<p>[WES Editing Note: The above equation has been typed using the equation writing program not supplied with my standard version of Microsoft Office. This program permits the alignment of the dividing line between the numerator and denominator to be centered with the equal sign. However, the program does not adapt well to the use of numerical subscripts of good size that are lowered below the accompanying letters as in chemical formulas. Clearly the printing program for <i>Reagent Chemicals</i> allows this.]</p>
Formic Acid, 96%	333	Iron Test. In line 1 change “12.0 g” to “6.0 g.”
Heavy Metals General Test	35-37	<p>The test has been revised to use a dilution buffer. The text cited below replaces text beginning with the heading “Heavy Metals (as Lead)” on page 35, continues through page 36, and ends on page 37 with the text under the heading “Standard/Control Solution.”</p> <p>Heavy Metals (as Lead)</p> <p>The heavy metals test is designed to limit the common metallic impurities (Ag, As, Bi, Cd, Cu, Hg, Mo, Pb, Sb, and Sn) that produce colors when sulfide ion is added to slightly acid solutions that contain them. In this test, such heavy metals are expressed “as lead” by comparing the color developed in test solution with that developed in lead standard/control solution. The optimum condition for proper performance of the test are: pH of solution between 3 and 4, total volume of 50 mL, use of pH 3.5 ammonium acetate dilution buffer solution, use of freshly prepared hydrogen sulfide water, and use of standard/control containing 0.02 mg of lead ion (Pb). These conditions are rarely modified in the individual procedures, and it will be only after a method validation that the method modification would be suitable for the individual reagent. The color comparisons should be made, using matched 50-mL color-comparison tubes, by viewing vertically over a white background. In some instances, an appropriate amount of the sample is added to the standard, called thereafter the control (see page 107), because the color developed in the test may be affected by the sample being tested. In other instances, no such effort occurs and the standard may be prepared without any of the sample. In general, the individual standard procedure provides only for the preparation of the sample solution. The remainder of the test directions, being the same for all reagents, is provided in this chapter.</p> <p>Two general test procedures are provided. Method 1 is</p>

	<p>to be used unless otherwise specified in the individual standard. This method is used, generally, for those substances that yield clear, colorless solution under specified test conditions. Method 2 is used generally, only for certain organic compounds.</p> <p>Unless otherwise directed in the individual specification, the test shall be carried out as cited below.</p> <p>Procedure. Test in a well-ventilated fume hood.</p> <p><i>pH 3.5 Ammonium Acetate Dilution Buffer:</i> Dissolve 5.0 g of ammonium acetate in about 450 mL of water in a 500 mL beaker. Using a pH meter, while stirring, adjust the pH to 3.5 with slow addition of glacial acetic acid or with ammonium hydroxide (10% NH₃) as needed. Dilute to 500 mL with water and store in a plastic bottle.</p> <p>Method 1</p> <p>Test Solution. Using the solution prepared as directed in the individual standard, adjust the pH to between 3 and 4 (by using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH₃). Dilute with pH 3.5 ammonium acetate dilution buffer solution to 40 mL, and mix.</p> <p>Standard/Control Solution. To 20 mL of water, or to the portion of that solution specified in the individual standard, add 0.02 mg of lead (Pb).* Dilute with water to 25 mL and mix. Adjust the pH (within 0.1 unit) to the value established for the test solution (using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH₃), dilute with pH 3.5 ammonium acetate dilution buffer solution to 40 mL, and mix.</p> <p>To each of the tubes containing the test solution and the standard/control solution, add 5.0 mL of freshly prepared hydrogen sulfide water, and dilute with pH 3.5 ammonium acetate dilution buffer to 50 mL and mix. Any brown color produced within 5 min in the test solution should not be darker than that produced in the standard/control solution.</p> <p>*When a control solution is specified in the individual specification and a direction is given therein to add a weight of lead ion (Pb), no further lead shall be added. Proceed to the dilution of pH adjustment, as necessary.</p>
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		<p>Method 2</p> <p>Test Solution. Transfer the quantity of substance specified in the individual specification to a suitable crucible, add sufficient sulfuric acid to wet the sample, and carefully heat at a low temperature until thoroughly charred. (The crucible may be loosely covered with a suitable lid during the charring.) Add to the carbonized mass 2 mL of nitric acid and 0.25 mL of sulfuric acid, and heat cautiously until white fumes of sulfur trioxide are no longer evolved. Ignite, preferably in a muffle furnace, at 500 to 600°C until the carbon is burned off completely. Cool, add 4 mL of 6 N hydrochloric acid, and cover. Digest on a hot plate (=100°C) for 15 min, uncover, and slowly evaporate on the hot plate to dryness. Moisten the residue with 0.05 mL of hydrochloric acid, add 10 mL of hot water, and digest for 2 min. Add ammonium hydroxide (10%) dropwise, until the solution is just alkaline, and dilute with water to 25 mL. Adjust the pH of the solution to between 3 and 4 (by using a pH meter) with 1 N acetic acid or with ammonium hydroxide (10%) as needed, dilute with pH 3.5 ammonium acetate dilution buffer solution to 40 mL, and mix.</p> <p>Standard/Control Solution. To 20 mL of water, add 0.02 mg of lead ion (Pb), and dilute with water to 25mL. Adjust the pH (within 0.1 unit) to the value established for the test solution (by using a pH meter) with 1N acetic acid or ammonium hydroxide (10% NH₃) as needed, dilute with pH 3.5 ammonium acetate dilution buffer solution to 40mL, and mix (as described in Method 1).</p> <p>To each of the tubes containing the test solution and the standard/control solution, add 5.0 mL of freshly prepared hydrogen sulfide water, and dilute with pH 3.5 ammonium acetate dilution buffer to 50 mL and mix. Any brown color produced within 5 min in the test solution should not be darker than that produced in the standard/control solution.</p> <p>[WES Editing Note: Red highlight print has been retained from minutes of Kishor Desai's revision of text.]</p>
Hydrochloric Acid	351-352	Bromide test: Revert to the test as written in the 9th edition.
Hydrofluoric Acid	357	Sulfate and Sulfite Test. In line 1 of the test delete the word "Teflon". Arsenic Test. In line 1 of the test delete the word "platinum".

Lead Monoxide	390	Under Ca, Cu, Fe, K, Ag, Na Sample Stock Solution: Change “30 ml nitric acid” to “20 ml of acetic acid”.												
Lead Subacetate	393	Nitrate. Note below the preparation of Blank Solution C that the paragraph contains superfluous directions beginning in line 4 with “Set a spectrophotometer...” Delete this and other material through end of paragraph. Text is in the general instructions on pages 38-39.												
Lithium Chloride	397	Nitrate. Drop specification and test. No lot has ever failed.												
Reagents, Buffers, and Indicators	101	Ferric Chloride, 5%. Replace “50 g” in line 1 with “25 g”.												
Lithium Perchlorate	401-403	Anhydrous and Trihydrate Assay. On page 402 in the Assay spec, second line, delete “· 3H ₂ O” from the chemical equation. In the last line of the Assay paragraph, end the final sentence at “LiClO ₄ .” In addition omit the second calculation equation entirely.												
Manganese Chloride	419	Assay: correct “few milligrams” to “50 milligrams” ascorbic acid.												
Manganese Chloride Tetrahydrate	419	Sulfate. Note that a factor of “0.412” should be inserted in the numerator of the equation for the percent sulfate calculation. As printed, the result yields the percent of Barium Sulfate.												
Phosphoric Acid	490	Sulfate. Delete the test as written. Use only the following: “sulfate (page 40, method 1). Use 1.7 g (1.0 mL).”												
Potassium Iodide	539	Barium. Delete individual barium test, and add to the AA text as follows: “Calcium, Magnesium, Sodium and Barium. (By flame AAS, page 63).” Delete existing sample stock solution, and substitute as follows: “Sample Stock Solution. Dissolve 50.0 g of sample in about 70 mL of water. Transfer to a 100 mL volumetric flask, at room temperature, dilute to the mark with water, and mix (1 mL = 0.50 g).” Add to the table: <table border="1" data-bbox="657 1696 1429 1774"> <thead> <tr> <th></th> <th>Wavelength (nm)</th> <th>Sample wt (g)</th> <th>Standard Added (mg)</th> <th>Flame Type</th> <th>Background</th> </tr> </thead> <tbody> <tr> <td>Ba</td> <td>553.6</td> <td>5.0</td> <td>.10; .20</td> <td>N/A</td> <td>No</td> </tr> </tbody> </table>		Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type	Background	Ba	553.6	5.0	.10; .20	N/A	No
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Potassium Permanganate	548	Assay. In the calculation at the top of the page rewrite the factor “23.58” to be “23585”.												

Procedure for pH of a 5% Solution at 25.0 °C	49	Add the following sentence cited below to the end of the paragraph: “Where no other tolerances are included in the test, allow measurement at 25.0 ± 2.0 °C.”												
Salicylic Acid	570	Assay. In line 10 following “ <i>Column:</i> ” change “Decyl” to “Octyl”.												
Sodium Acetate Trihydrate	581	Phosphate (page 40, method 2).												
Sodium Bromide	593	Heavy Metals. Delete the entire text. Replace with reference to the front of the book followed by two sentences, as cited below: “(Page 36, Method 1). Dissolve 6.0 g in about 20 mL of water, and dilute with water to 50 mL. Use 25 mL to prepare the sample solution, and use the remaining 5.0 mL to prepare the control solution,”												
Sodium Carbonate	598	<u>Assay:</u> In 4 th line change “0.0621 g” to 0.062 g” And in the equation change “6.21” to 6.20.”												
Sodium Dodecyl Sulfate	610, 612	Sodium chloride and sodium sulfate specification and tests. Drop foregoing specification and tests. Retain the assay specification as printed.												
Sodium Iodide	622	Barium. Delete individual barium test, and add to AA text as follows: “ Calcium, Magnesium, Potassium, and Barium. (By flame AAS, page 63).” Delete existing sample stock solution, and substitute as follows: “ Sample Stock Solution. Dissolve 50.0 g of sample in about 70 mL of water. Transfer to a 100 mL volumetric flask, at room temperature, dilute to the mark with water, and mix (1 mL = 0.50 g).” Add to the table: <table border="1" data-bbox="657 1549 1432 1627"> <thead> <tr> <th></th> <th>Wavelength (nm)</th> <th>Sample wt (g)</th> <th>Standard Added (mg)</th> <th>Flame Type</th> <th>Background</th> </tr> </thead> <tbody> <tr> <td>Ba</td> <td>553.6</td> <td>5.0</td> <td>.10; .20</td> <td>N/A</td> <td>No</td> </tr> </tbody> </table>		Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type	Background	Ba	553.6	5.0	.10; .20	N/A	No
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Sodium Perchlorate	633	Under assay, specification, delete H ₂ O. Under assay last line: delete “or 0.01405 g of NaClO ₄ ·H ₂ O” Also delete the second equation.												

Specific Rotation	46	In the line below the equation at the bottom of page 46, in the phrase: “A is corrected specific rotation”, change the word “specific” to “optical”. Two places to change.												
Succinic Acid	663	Phosphate test Change sample weight from “1.0 g” to “2.0 g”.												
Volumetric Solutions	116-118	Three Molar solutions mistakenly have calculations in Normality: Cupric sulfate, 0.1M (Page 116); EDTA, 0.1M (Page 117); Lead nitrate, 0.1M (Page 118). Change the “N” to “M” and include in supplement as an errata.												
Water, Reagent	718	Chloride, Nitrate, Phosphate, and Sulfate. At the end of line 4 insert the following parenthetical expression: “(resistivity 18 megaohms-cm)”												
Zinc Acetate Dihydrate	724	Lead test. Incorporate lead with the four other metals already tested by AA as cited below. “Calcium, Magnesium, Potassium, Sodium, and Lead (by flame AAS, Page 63”. Add to the table: <table border="1" data-bbox="657 926 1432 1003"> <thead> <tr> <th></th> <th>Wavelength (nm)</th> <th>Sample wt (g)</th> <th>Standard Added (mg)</th> <th>Flame Type*</th> <th>Background Correction</th> </tr> </thead> <tbody> <tr> <td>Pb</td> <td>217.0</td> <td>2.0</td> <td>.04; .08</td> <td>A/A</td> <td>Yes</td> </tr> </tbody> </table> <p>*A/A = Air/Acetylene</p>		Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type*	Background Correction	Pb	217.0	2.0	.04; .08	A/A	Yes
	Wavelength (nm)	Sample wt (g)	Standard Added (mg)	Flame Type*	Background Correction									
Pb	217.0	2.0	.04; .08	A/A	Yes									
Zinc Chloride, 0.1 M	122	Change as follows: Weigh accurately 6.54 g of Zinc (13.63 g of ZnCl ₂) and dissolve in 80 mL of 10% hydrochloric acid. Warm if necessary to complete dissolution, cool, dilute with water to volume in a 1-L Volumetric flask, and mix thoroughly. Standardize as follows: pipet and transfer 40.0 ml of Zinc chloride solution to 400 ml beaker, and sufficient hexamethylene tetramine to produce purple-red color. Titrate with freshly standardized 0.1 M EDTA to a color change of purple-red to lemon yellow. $M = \frac{\text{mL} \times M \text{ EDTA}}{\text{mL ZnCl}_2}$												
Zinc Sulfate, 0.1M	122	Weigh accurately 28.76 g of Zinc Sulfate heptahydrate and dissolve in 500 ml of water in a 1-L volumetric flask. Dilute to the mark with water and mix thoroughly. Standardize as follows: pipet and transfer 40.0 ml of Zinc sulfate solution to 400 ml beaker, and add about 100 ml of water, 50 mg of Xylenol Orange indicator mixture, and sufficient hexamethyl tetramine to produce purple-red color. Titrate												

		with freshly standardized 0.1 M EDTA to a color change of purple-red to lemon yellow. $M = \frac{\text{mL} \times M \text{ EDTA}}{\text{mL ZnSO}_4}$
Organic Errata:		Page 333, Acetic Acid, Column: 1.8 m x 64mm Should say Acetic Acid, Column: 1.8 m x 6.4mm Page 743, Assay by Liquid Chromatography Detector: UV, 254 nm should say 220 nm And Sample Size: 1 uL of a 100 ug/mL solution should say 10 uL of a 5000 ug/mL solution Page 751 and 803, CAS number for Phenthoate, 2597-13-7 Should say CAS number for Phenthoate, 2597-03-7, Page 803 change is 1/3 of the way down the right side of the page
Created 6/23/08		

Additions to Supplements on 4-24-10

REAGENT	PAGE	SUPPLEMENT	Comment
Calmagite Indicator Solution	p 99	Dissolve 0.10g in 100 ml of water. The solution should be complete and free of turbidity.	New: Insert in p99, after Cadmium Nitrate, 3%.
Hydroxylamine_Sulfate	p 365 Under Assay: Line 6	Change equivalence factor used in assay procedure from 0.00410 g to 0.004104 g.	
Sodium Borohydride	p 591 Under Assay: Line 9	Change equivalence factor used in assay procedure from 0.00473 g to 0.0149 g.	
5_Sulfosalicylic_Acid Dihydrate	page_669	Replace benzene with toluene in the extraction step in the first sentence of this test.	Elimination of the use of benzene in Salicylic Acid analysis
Procedure for Flame AAS	page 64	Text of example to be written as follows: <i>Example</i> , Figure 2-6 shows a	Example procedure is set up for Calcium, Manganese,

		<p>typical procedure, as it appears in this book, that could be used for trace or ultratrace elemental analysis of a reagent chemical, using the method of standard additions, which is illustrated here.</p> <p>To a first set of three 25-ml volumetric flasks, add 2.0ml (0.20g) of sample stock solution. To two of the flasks, add the specified amounts of sodium shown in the table in figure 2-6. Add 2ml of 5% potassium chloride to all three flasks and dilute to the mark with deionized water</p> <p>To a second set of three 25-ml volumetric flasks, add 10.0 ml (1.0 g) of sample stock solution. To two of the flasks, add the specified amounts of potassium and strontium shown in the table in figure 2-6. Dilute the contents of the three flasks to the mark with deionized water.</p> <p>Analyze the solutions by means of a suitable atomic absorption spectrophotometer, using the conditions outlined in the table in Figure 2-6. Calculate the trace metal content of the sample by the method of standard additions.</p>	<p>Potassium, Sodium and Strontium analysis but table with supporting data for procedure (Fig 2-6) only has information on Potassium, Sodium and Strontium. Since this was only an example of procedure, committee decided to eliminate Calcium and Manganese from written procedure</p>
Bromphenol Blue	p 213 under GENERAL DESCRIPTION	<p><i>Typical Appearance: add (sultone):</i> pale orange solid Also add the following <i>Typical Appearance (sodium salt):</i> blue black solid</p>	
Sodium Chloride	p 602	Iodide Test: Remove the following from the second line	

		in the paragraph, “and 1.0 mg of bromide ion (Br) “. In addition, remove the entire following sentence from the 5 th and 6 th lines in the paragraph, “Reserve the water solution for the test for bromide”	
Potassium oxalate Monohydrate	p 543 Under General Description	Aqueous solubility: 36 g in 100 mL at 20 °C (add “g”)	
Isopropyl ether	p379 line 6	Add Subscript 2 for Na in the equation for peroxide. Correct it to Na ₂ S ₂ O ₃	
Lithium Sulfate Monohydrate	p 403 Last line of Equation and Assay procedure	% Li ₂ SO ₄ · H ₂ O equation : Delete H ₂ O from equation and 63.98 should be changed to 5.497 Also last line of Assay paragraph: One milliliter of 0.1N sodium hydroxide corresponds to 0.05497 g of Li ₂ SO ₄	