3.6 Appendices

Appendix 1: Exercise - Practice with Samples

Training Samples

Training samples may vary, and depend on what is located in the laboratory. The training samples, using USP products whenever possible, also demonstrate the trainee's ability and proficiency in performing regulatory tests. The following were used for the above training program and are examples of products that have been used successfully for training. However, other products may be used that demonstrate the same level of training. *Grayed parts of the following products were not used in the exercises but may be added to complete a full product analysis.* Full worksheets are to be generated for each product analyzed. *Italicized items 1, 4 & 7 are also proficiency samples.* (* also identified as a proficiency test in Appendix 2).

USP < > refers to the General Chapters – General Tests and Assays , General Requirements for Tests and Assays found in the back of the USP/NF

I. Training Samples

1. ***Acetaminophen (API)*** – USP, current edition, {Section 3.4.2.1B; 3.4.2.2 IB; 3.4.2.2 IIB; 3.4.2.3 IIB; 3.4.2.3 IIIB}
    - 1.1 Identification
        - Test A: Infrared <197K> *
        - Test B: UV <197U>
        - Test C: TLC <201> *
    - 1.2 Melting Range <741>
    - 1.3 Water Method 1 <921> KF Titration *
    - 1.4 Residue on Ignition <281>
    - 1.5 Chloride <221> - limit test
    - 1.6 Sulfate <221> - limit test
    - 1.7 Sulfide – limit test
    - 1.8 Heavy Metals, Method II <231>
    - 1.9 Free p-aminophenol - colorimetric
    - 1.10 Limit of p-chloroacetanilide - TLC
    - 1.11 Readily carbonizable substances <271>
    - 1.12 Organic volatile impurities Method V <467> GLC – capillary column.
    - 1.13 Assay – UV *

2. **Ascorbic Acid Tablets** – USP, current edition, { Section 3.4.2.1B, 3.4.2.4 IB}
    - 2.1 Assay – titration
    - 2.2 Identification – Test B

Test A – spot test
Test B – color test
Test C – color test

2.3 Dissolution, Procedure for Pooled Sample <711>
2.4 Uniformity of Dosage (Weight Variation)

3. **Aspirin Tablets** – USP, current edition { Section 3.4.2.3 IVB, 3.4.2.4 IB}
   3.1 Identification
   Test A – color test
   Test B – IR <197K>
   3.2 Dissolution <711> Apparatus 1 – UV Det
   3.3 Uniformity of Dosage Units <905>
   3.4 Limit of Free Salicylic Acid – HPLC
   3.5 Assay – HPLC with ion-pair reagent

4. ***Acetaminophen and Caffeine Tablets*** – USP, current edition, {Section 3.4.2.3 IVB, 3.4.2.4 IB}
   4.1 Identification - HPLC
   4.2 Dissolution <711> Apparatus 2 - HPLC *
   4.3 Uniformity of Dosage Units <905> HPLC
   4.4 Assay – HPLC two component with IS *

5. **Acetaminophen Oral Solution** – USP, current edition,{Section 3.4.2.3 IB, 3.4.2.3 IIIB, 3.4.2.3 IVB}
   5.1 Identification – FTIR <197K>
   A: HPLC
   B: TLC <201>
   5.2 pH <791>
   5.3 Alcohol Content (if present) Method II <611>
   (if alcohol not present spike at 5% ethanol)
   5.4 Assay – HPLC <621>

6. Caffeine API – USP, current edition {Section 3.4.2.3 IIIB.2}
   6.1 Identification
   A. Infrared Absorption <197M>
   B. HPLC
   6.2 Melting Range <741>
   6.3 Water, Method III <921>
   6.4 Residue on Ignition <281>
   6.5 Heavy Metals, Method II <231>
   6.6 Chromatographic Purity
   6.7 Assay – HPLC <621>

7. *Saccharin* – NF, current edition, {3.4.2.1 B4)
    - 7.1 Identification – FTIR
    - 7.2 Melting Range <741>
    - 7.3 Loss on Drying <731>
    - 7.4 Readily carbonizable substances <271>
    - 7.5 Residue on Ignition <281>
    - 7.6 Toluenesulfonamides – Column and gas chromatography (FID)
    - 7.7 Selenium <291>
    - 7.8 Heavy Metals, Method II <231>
    - 7.9 Benzoic and salicylic acids – chemical test
    - 7.10 Assay – Titration *

8. Dextrose – USP, current edition, {Section 3.4.2.1B, 3.4.2.2 IVB}
    - 8.1 Identification – color precipitate
    - 8.2 Color of Solution – limits test
    - 8.3 Specific Rotation <781S>
    - 8.4 Acidity – titration
    - 8.5 Water Method III <921> (loss on drying)
    - 8.6 Residue on Ignition <281>
    - 8.7 Chloride <221> - limit test
    - 8.8 Sulfate <221> - limit test
    - 8.9 Arsenic Method I <211> - limit test
    - 8.10 Heavy Metals <231> limit test
    - 8.11 Dextrin – limit test
    - 8.12 Soluble starch, sulfites – color
    - 8.13 Assay - Optical Rotation (Use current USP procedure for Dextrose Injection).

9. Naltrexone HydrochlorideTablets – USP, current edition, { Section 3.4.2.3 IVB}
    - 9.1 Identification - HPLC
    - 9.2 Dissolution <711> Apparatus 2 – HPLC Determination
    - 9.3 Uniformity of Dosage Units – HPLC gradient
    - 9.4 Assay – HPLC gradient

10. Glycerin – USP, current edition
    - 10.1 Identification –
        - A. FTIR
        - B. Limit of Diethylene Glycol and Ethylene Glycol (GC-FID)
    - 10.2 Assay – Titration
    - 10.3 Impurities
        - Inorganic Impurities
        - Organic Impurities –
            - Procedure 1 Related Compounds (GC-FID)
            - Procedure 2 – Limit of Chlorinated Compounds

Procedure 3 – Fatty Acids and Esters
10.4 Specific Gravity <841>
10.5 Water Determination <921>

II. Additional Training Samples as needed:

Additional training samples may be added or substituted to demonstrate and reinforce the analytical procedures listed in the training program.