Below are collecting methodologies for EPA Methods 525.2 and 625.1, which are both from the method, section 8 from EPA Method 525.1 and section 9 from EPA Method 625.1.

EPA Method 525.2 section 8:

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection -- When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about two minutes). Adjust the flow to about 500 mL/min. and collect samples from the flowing stream. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample. Automatic samplers that composite samples over time should use refrigerated glass sample containers if possible.

8.2 Sample Dechlorination and Preservation -- All samples should be iced or refrigerated at 4° C and kept in the dark from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of 40-50 mg of sodium sulfite (this may be added as a solid with stirring or shaking until dissolved) to each water sample. It is very important that the sample be dechlorinated prior to adding acid to lower the pH of the sample. Adding sodium sulfite and HCl to the sample bottles prior to shipping to the sampling site is not permitted. Hydrochloric acid should be used at the sampling

site to retard the microbiological degradation of some analytes in water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of acidic compounds like pentachlorophenol.

8.2.1 If cyanizine is to be determined, a separate sample must be collected. Cyanazine degrades in the sample when it is stored under acidic conditions or when sodium sulfite is present in the stored sample. Samples collected for cyanazine determination MUST NOT be dechlorinated or acidified when collected. They should be iced or refrigerated as described above and analyzed within 14 days. However, these samples MUST be dechlorinated and acidified immediately prior to fortification with internal standards and surrogates, and extraction using the same quantities of acid and sodium sulfite described above.

8.2.2 Atraton and prometon are not efficiently extracted from water at pH 2 due to what appears to be their ionization in solution under acidic conditions. In order to determine these analytes accurately, a separate sample must be collected and dechlorinated with sodium sulfite, but no acid should be added. At neutral pH, these two compounds are recovered from water with efficiencies greater than 90%. The data in Tables 3, 4, 5, 6, and 8 are from samples extracted at pH 2.

8.3 Holding Time -- Results of the time/storage study of all method analytes showed that all but six compounds are stable for 14 days in water samples when the samples are dechlorinated, preserved, and stored as described in Section 8.2. Therefore, samples must be extracted within 14 days. If the following analytes are to be determined, the samples cannot be held for 14 days but must be

extracted immediately after collection and preservation: carboxin, diazinon, disulfoton, disulfoton sulfoxide, fenamiphos, and terbufos. Sample extracts may be stored at 4° C for up to 30 days after sample extraction.

8.4 Field Blanks

8.4.1 Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sampling site along with the empty sample containers. Return the FRB to the laboratory with the filled sample bottles.

8.4.2 When sodium sulfite and hydrochloric acid are added to samples, use the same procedure to add the same amounts to the FRB.

EPA Method 625.1 section 9:

9. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9.1 Collect samples as grab samples in amber or clear glass bottles, or in refrigerated bottles using automatic sampling equipment. If clear glass is used, protect samples from light. Collect 1-L of ambient waters, effluents, and other aqueous samples. If the sensitivity of the analytical system is sufficient, a smaller volume (e.g., 250 mL), but no less than 100 mL, may be used. Conventional sampling practices (Reference 15) should be followed, except that the bottle must not be pre-rinsed with sample before collection. Automatic sampling equipment must be

as free as possible of polyvinyl chloride or other tubing or other potential sources of contamination. If needed, collect additional sample(s) for the MS/MSD (Section 8.3).

9.2 Ice or refrigerate samples at ≤6 °C from the time of collection until extraction, but do not freeze. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. Any method suitable for field use may be employed to test for residual chlorine (Reference 16). Add more sodium sulfate if 80 mg/L is insufficient but do not add excess sodium thiosulfate. If sodium thiosulfate interferes in the determination of the analytes, an alternate preservative (e.g., ascorbic acid or sodium sulfite) may be used. If preservative has been added, shake the sample vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be extracted within 7 days of collection and sample extracts must be analyzed within 40 days of extraction.