METHOD 0011

SAMPLING FOR SELECTED ALDEHYDE AND KETONE EMISSIONS FROM STATIONARY SOURCES

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of the analytes listed in the following table:

Analyte	CAS No.[a]
Formaldehyde	50-00-0
Acetaldehye	75-07-0
Acetophenone	98-86-2
Isophorone	78-59-1
Propionaldehyde	123-38-6

[a] Chemical Abstract Service Registry Number

This method has been applied specifically to the above analytes. Many laboratories have extended method application to other aldehydes and ketones. This method is possibly applicable to other aldehydes and ketones from stationary sources as specified in the regulations. However, this method is not applicable to quinone (CAS No. 106-51-4), acrolein (CAS No. 107-02-08), methyl ethyl ketone (CAS No. 78-93-3), and methyl isobutyl ketone (CAS No. 108-10-1).

1.2 The detection limit for a 30 ft^3 (849 L) sample over a 1 hour sampling period may be as low as 10 ppbv for acetophenone and isophorone, 60 ppbv for propionaldehyde, 40 ppbv for acetaldehyde, and 90 ppbv for formaldehyde. Because the derivatization reaction is based on the formation of an equilibrium state between reactants and products, for some compounds quantitative recoveries may not be achieved until the concentration exceeds 200 ppbv.

1.3 This method is restricted to use by, or under the close supervision of, analysts experienced in sampling organic compounds in air. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

Gaseous and particulate pollutants are withdrawn isokinetically from an emission source and are collected in aqueous acidic 2,4-dinitrophenylhydrazine. Formaldehyde present in the emissions reacts with the 2,4-dinitrophenylhydrazine to form the formaldehyde dinitrophenylhydrazone derivative. The dinitrophenylhydrazone derivative is extracted, solvent-exchanged, concentrated, and then analyzed by high performance liquid chromatography (HPLC) according to Method 8315 or other appropriate technique.

3.0 INTERFERENCES

3.1 A decomposition product of 2,4-dinitrophenylhydrazine, 2,4-dinitroaniline, can be an analytical interferant if concentrations are high. The 2,4-dinitroaniline can coelute with the 2,4-dinitrophenylhydrazone of formaldehyde under the high performance liquid chromatography conditions used for the analysis. High concentrations of highly oxygenated compounds, especially acetone, that have the same retention time or nearly the same retention time as the dinitrophenylhydrazone of formaldehyde, and that also absorb at 360 nm, will interfere with the analysis.

3.2 Formaldehyde, acetone, and 2,4-dinitroaniline contamination of the aqueous acidic 2,4-dinitrophenylhydrazine (DNPH) reagent is frequently encountered. The reagent must be prepared within five days of use in the field and must be stored in an uncontaminated environment both before and after sampling, in order to minimize blank problems. Some concentration of acetone contamination is unavoidable, because acetone is ubiquitous in laboratory and field operations. However, the acetone contamination must be minimized.

3.3 Dimethylolurea creates a slight positive interference; and hexamethylenetetramine and paraformaldehyde significantly interfere with the determination of formaldehyde. These compounds can decompose in the acidic reagent used to collect the sample to form formaldehyde;

3.4 Tolualdehyde interferes with the determination of acetophenone because they coelute chromatographically;

3.5 High levels of nitrogen dioxide can interfere by consuming all of the reagent.

4.0 APPARATUS AND MATERIALS

4.1 This sampling train configuration is adapted from Method 5 (see Ref. 1) procedures. The sampling train consists of the following components: Probe nozzle, pitot tube, differential pressure gauge, metering system, barometer, and gas density determination equipment. A schematic of the sampling train is shown in Figure 1.

4.1.1 Probe Nozzle - The probe nozzle shall be quartz or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant inner diameter. The nozzle shall be buttonhook or elbow design. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g., 0.32 to 1.27 cm (1/8 to ½ in.), or larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Sec. 8.1.

4.1.2 Probe Liner - Borosilicate glass or quartz shall be used for the probe liner. The tester should not allow the temperature in the probe to exceed 120 ±14°C (248 ±25°F).

4.1.3 Pitot Tube - The pitot tube shall be Type S or any other appropriate device. The Type S pitot tube shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter be between 0.48 and 0.95 cm. There shall be an equal distance from the base of each leg to its face-opening plane; it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned but slight misalignments of the openings are permissible. The

Type S pitot tube assembly shall have a known coefficient, determined as outlined in Method 2 (see Ref. 1). The pitot tube shall be attached to the probe to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2) during sampling.

4.1.4 Differential Pressure Gauge - The differential pressure gauge shall be an inclined manometer or equivalent device as described in Method 2. One manometer shall be used for velocity-head readings and the other for orifice differential pressure readings.

4.1.5 Impingers - The sampling train requires a minimum of five impingers, connected as shown in Figure 1, with ground glass (or equivalent) vacuum-tight fittings. For the first, third, fourth, and fifth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.27 cm (½ in) inside diameter glass tube extending to 1.27 cm (½ in.) from the bottom of the flask. For the second impinger, use a Greenburg-Smith impinger with the standard tip. Place a thermometer capable of measuring temperature to within 1°C (2°F) at the outlet of the fifth impinger for monitoring purposes.

4.1.6 Metering System - The necessary components of the metering system are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment as shown in Figure 1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems may be used which are capable of maintaining sample volumes to within 2%. The metering system may be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates.

4.1.7 Barometer - The barometer may be mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service Station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase (vice versa for elevation decrease).

4.1.8 Gas Density Determination Equipment - The gas density determination equipment includes a temperature sensor and pressure gauge (as described in Method 2) and gas analyzer, if necessary (an Orsat of Fyrite type combustion gas analyzer, or equivalent. For analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

4.2 Sample Recovery

4.2.1 Probe Liner - Probe nozzle and brushes; Teflon® bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel,

Teflon®, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the impingers.

4.2.2 Wash Bottles - Three wash bottles are required. Teflon® or glass wash bottles are recommended. Polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to the organic solvents used for sample recovery.

4.2.3 Graduated Cylinder and/or Balance - A graduated cylinder or balance is required to measure condensed water to the nearest 1 mL or 1 g. Graduated cylinders shall have divisions not greater than 2 mL. Laboratory balances capable of weighing to ±0.5 g are required.

4.2.4 Amber Glass Storage Containers - One-liter wide-mouth amber flint glass bottles with Teflon®-lined caps are required to store impinger water samples. The bottles must be sealed with Teflon® tape.

4.2.5 Rubber Policeman and Funnel - A rubber policeman and funnel are required to aid in the transfer of materials into and out of containers in the field.

4.3 Reagent Preparation

4.3.1 Bottles/Caps - Amber 1 - 4 L bottles with Teflon®-lined caps are required for storing cleaned DNPH solution. Additional 4-L bottles are required to collect waste organic solvents.

4.3.2 Large Glass Container - At least one large glass (8 to 16 L) is required for mixing the aqueous acidic DNPH solution.

4.3.3 Stir Plate/Large Stir Bars/Stir Bar Retriever - A magnetic stir plate and large stir bar are required for the mixing of the aqueous acidic DNPH solution. A stir bar retriever is needed for removing the stir bar from the large container holding the DNPH solution.

4.3.4 Buchner Filter/Filter Flask/Filter Paper - A large filter flask (2-4 L) with a buchner filter, appropriate rubber stopper, filter paper, and connecting tubing are required for filtering the aqueous acidic DNPH solution prior to cleaning.

4.3.5 Separatory Funnel - At least one large separatory funnel (2 L) is required for cleaning the DNPH prior to use.

4.3.6 Beakers - Beakers (150 mL, 250 mL, and 400 mL) are useful for holding/measuring organic liquids when cleaning the aqueous acidic DNPH solution and for weighing DNPH crystals.

4.3.7 Funnels - At least one large funnel is needed for pouring the aqueous acidic DNPH into the separatory funnel.

4.3.8 Graduated Cylinders - At least one large graduated cylinder (1 to 2 L) is required for measuring organic-free reagent water and acid when preparing the DNPH solution.

4.3.9 Top-loading Balance - A one-place top loading balance is needed for weighing out the DNPH crystals used to prepare the aqueous acidic DNPH solution.

4.3.10 Spatulas - Spatulas are needed for weighing out DNPH when preparing the aqueous DNPH solution.

4.4 Crushed Ice - Quantities of crushed ice ranging from 10-50 lb may be necessary during a sampling run, depending upon ambient temperature. Samples which have been taken must be stored and shipped cold; sufficient ice for this purpose must be allowed.

5.0 REAGENTS

5.1 Reagent Grade Chemicals - Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free Reagent Water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Silica Gel - Silica gel shall be indicating type, 6-16 mesh. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

5.4 2,4-Dinitrophenylhydrazine (DNPH), $[2,4\text{-}(0_2N)_2C_6H_3]NHNH_2$ - The quantity of water may vary from 10 to 30%.

5.4.1 The 2,4-dinitrophenylhydrazine reagent must be prepared in the laboratory within five days of sampling use in the field. Preparation of DNPH can also be done in the field, with consideration of appropriate procedures required for safe handling of solvent in the field. When a container of prepared DNPH reagent is opened in the field, the contents of the opened container should be used within 48 hours. All laboratory glassware must be washed with detergent and water and rinsed with water, methanol, and methylene chloride prior to use.

NOTE: DNPH crystals and DNPH solution are potential carcinogens and should be handled with plastic gloves at all times, with prompt and extensive use of running water in case of skin exposure.

5.4.2 Preparation of Aqueous Acidic DNPH Derivatizing Reagent - Each batch of DNPH reagent should be prepared and purified within five days of sampling, according to the procedure described below.

NOTE: Reagent bottles for storage of cleaned DNPH derivatizing solution must be rinsed with acetonitrile and dried before use. Baked glassware is not essential for preparation of DNPH reagent. The glassware must not be rinsed with acetone or an unacceptable concentration of acetone contamination will be introduced. If field preparation of DNPH is performed, caution must be exercised in avoiding acetone contamination.

5.4.2.1 Place an 8 L container under a fume hood on a magnetic stirrer. Add a large stir bar and fill the container half full of organic-free reagent water. Save the

empty bottle from the organic-free reagent water. Start the stirring bar and adjust the stir rate to be as fast as possible. Using a graduated cylinder, measure 1.4 L of concentrated hydrochloric acid. Slowly pour the acid into the stirring water. Fumes may be generated and the water may become warm. Weigh the DNPH crystals to ±0.1 g (see Table 1 for approximate amounts) and add to the stirring acid solution. Fill the 8 L container to the 8 L mark with organic-free reagent water and stir overnight. If all of the DNPH crystals have dissolved overnight, add additional DNPH and stir for two more hours. Continue the process of adding DNPH with additional stirring until a saturated solution has been formed, as evidenced by the presence of visible crystals after continued stirring. Filter the DNPH solution using vacuum filtration. Gravity filtration may be used, but a much longer time is required. Store the filtered solution in an amber bottle at room temperature.

5.4.2.2 Within five days of proposed use, place about 1.6 L of the DNPH reagent in a 2 L separatory funnel. Add approximately 200 mL of methylene chloride and stopper the funnel. Wrap the stopper of the funnel with paper towels to absorb any leakage. Invert and vent the funnel. Then shake vigorously for 3 minutes. Initially, the funnel should be vented frequently (every 10 - 15 seconds). After the layers have separated, discard the lower (organic) layer.

5.4.2.3 Extract the DNPH a second time with methylene chloride and finally with cyclohexane. When the cyclohexane layer has separated from the DNPH reagent, the cyclohexane layer will be the top layer in the separatory funnel. Drain the lower layer (the cleaned extracted DNPH reagent solution) into an amber bottle that has been rinsed with acetonitrile and allowed to dry.

5.4.3 DNPH Reagent Check - Take two aliquots of the extracted DNPH reagent. The size of the aliquots is dependent upon the exact sampling procedure used, but 100 mL is reasonably representative. Analyze one aliquot of the reagent according to Sec. 7 of Method 8315 as a Quality Control check to ensure that the background in the reagent is acceptable for field use. Save the other aliquot of aqueous acidic DNPH for use as a method blank when the analysis is performed. The reagent is acceptable for use if the background meets the AIC (Acceptable Impurity Concentration) as specified in Sec. 5.4.5.

5.4.4 Shipment to the Field - Tightly cap the bottle containing extracted DNPH reagent using a Teflon®-lined cap. Seal the bottle with Teflon® tape. After the bottle is labeled, the bottle may be placed in a friction-top can (paint can or equivalent) containing a 1-2 inch layer of granulated charcoal and stored at ambient temperature until use.

5.4.4.1 If the DNPH reagent has passed the Quality Control criteria, the reagent may be packaged to meet necessary shipping requirements and sent to the sampling area. If the Quality Control criteria are not met, the reagent solution may be re-extracted or the solution may be re-prepared and the extraction sequence repeated.

5.4.4.2 If the DNPH reagent is not used in the field within five days of extraction, an aliquot may be taken and analyzed as described in Sec. 7 of Method 8315. If the reagent meets the Quality Control requirements, the reagent may be used. If the reagent does not meet the Quality Control requirements, the reagent must be discarded and new reagent must be prepared and tested.

5.4.5 Calculation of Acceptable Concentrations of Impurities in DNPH Reagent - The acceptable impurity concentration (AIC, µg/mL) is calculated from the expected analyte concentration in the sampled gas (EAC, ppbv), the volume of air that will be sampled at standard conditions (SVOL, L), the formula weight of the analyte (FW, g/mol), and the volume of DNPH reagent that will be used in the impingers (RVOL, mL):

$$AIC = 0.1 \times [EAC \times SVOL \times FW/24.4 \times (FW + 180)/FW](RVOL/1{,}000)$$

where:

0.1 is the acceptable contaminant concentration,
24.4 is a factor relating ppbv to g/L,
180 is a factor relating underivatized to derivatized analyte, and
1,000 is a unit conversion factor.

5.4.6 Disposal of Excess DNPH Reagent - Excess DNPH reagent may be returned to the laboratory and recycled or treated as aqueous waste for disposal purposes. 2,4-Dinitrophenylhydrazine is a flammable solid when dry, so water should not be evaporated from the solution of the reagent.

5.5 Field Spike Standard Preparation - To prepare a formaldehyde field spiking standard at 4010 mg/L, use a 500 µL syringe to transfer 0.5 mL of 37% by weight of formaldehyde (401 g/L) to a 50 mL volumetric flask containing approximately 40 mL of methanol. Dilute to 50 mL with methanol.

5.6 Hydrochloric Acid, HCl - Reagent grade hydrochloric acid (approximately 12N) is required for acidifying the aqueous DNPH solution.

5.7 Methylene Chloride, CH_2Cl_2 - Methylene chloride (suitable for residue and pesticide analysis, GC/MS, HPLC, GC, Spectrophotometry or equivalent) is required for cleaning the aqueous acidic DNPH solution, rinsing glassware, and recovery of sample trains.

5.8 Cyclohexane, C_6H_{12} - Cyclohexane (HPLC grade) is required for cleaning the aqueous acidic DNPH solution.

NOTE: Do not use spectroanalyzed grades of cyclohexane if this sampling methodology is extended to aldehydes and ketones with four or more carbon atoms.

5.9 Methanol, CH_3OH - Methanol (HPLC grade or equivalent) is necessary for rinsing glassware.

5.10 Acetonitrile, CH_3CN - Acetonitrile (HPLC grade or equivalent) is required for rinsing glassware.

5.11 Formaldehyde, HCHO - Formaldehyde (analytical reagent grade, or equivalent) is required for preparation of standards. If other aldehydes or ketones are used, analytical reagent grade, or equivalent, is required.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Because of the complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

6.2 Laboratory Preparation

6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576 (Air Pollution Technical Document, see references), unless otherwise specified.

6.2.2 Weigh several 200 to 300 g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

6.3 Preliminary Field Determinations

6.3.1 Select the sampling site and the minimum number of sampling points according to Method 1 (see Ref. 1) or other relevant criteria. Determine the stack pressure, temperature, and range of velocity heads using Method 2. A leak-check of the pitot lines according to Method 2 must be performed. Determine the stack gas moisture content using Approximation Method 4 (see Ref. 1) or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack gas dry molecular weight, as described in Method 2. If integrated Method 3 (see Ref. 1) sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

6.3.2 Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 L/min (1.0 cfm). During the run, do not change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Sec. 2 of Method 2).

6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

6.3.4 A minimum of 45 ft^3 of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of formaldehyde from incineration systems (45 ft^3 is equivalent to one hour of sampling at 0.75 dscf). Additional sample volume shall be collected as necessitated by the capacity of the DNPH reagent and analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Sec. 10.0.

6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus 0.5 min.

6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-volume samples. In these cases, careful documentation must be maintained in order to allow accurate calculation of concentrations.

6.4 Preparation of Collection Train

6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon® film or aluminum foil until just prior to assembly or until sampling is about to begin.

NOTE: Appendix A at the end of this procedure contains guidance on the addition of a filter as a check on the survival of particulate material through the impinger system. This filter can be added to the impinger train either after the second impinger or after the third impinger.

6.4.2 Place 200 mL of purified DNPH reagent in the first impinger and 100 mL of reagent in the second and third impingers, and leave the fourth impinger empty. Transfer approximately 200 to 300 g of preweighed silica gel from its container to the fifth impinger. Care should be taken to ensure that the silica gel is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

6.4.3 With a glass or quartz liner, install the selected nozzle using a Viton-A O-ring when stack temperatures are less than 260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connecting systems utilizing either 316 stainless steel or Teflon® ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

6.4.4 Assemble the train as shown in Figure 1. During assembly, do not use any silicone grease on ground-glass joints upstream of the impingers. Use Teflon® tape, if required. A very light coating of silicone grease may be used on ground-glass joints downstream of the impingers, but the silicone grease should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone grease contamination. If necessary, Teflon® tape may be used to seal leaks. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperature.

6.4.5 Place crushed ice all around the impingers.

6.4.6 Turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize.

6.5 Leak-Check Procedures

6.5.1 Pre-test Leak Check

6.5.1.1 After the sampling train has been assembled, turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to

stabilize. If a Viton-A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 381 mm Hg (15 in. Hg) vacuum.

NOTE: A lower vacuum may be used, provided that the lower vacuum is not exceeded during the test.

6.5.1.2 If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first attaching a carbon-filled leak check impinger to the inlet and then plugging the inlet and pulling a 381 mm Hg (15 in. Hg) vacuum. (A lower vacuum may be used if this lower vacuum is not exceeded during the test.) Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum. Alternatively, leak-check the probe with the rest of the sampling train in one step at 381 mm Hg (15 in. Hg) vacuum. Leakage rates no greater than 4% of the average sampling rate or less than or equal to 0.00057 m^3/min (0.02 cfm), whichever is less, are acceptable.

6.5.1.3 The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with the fine-adjust valve fully open and coarse-adjust valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do not reverse direction of the fine-adjust valve, as liquid will back up into the train. If the desired vacuum is exceeded, either perform the leak check at this higher vacuum or end the leak check, as shown below, and start over.

6.5.1.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe. When the vacuum drops to 127 mm (5 in.) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed to prevent the liquid in the impingers from being forced backward into the sampling line and silica gel from being entrained backward into the third impinger.

6.5.2 Sampling Run Leak Check

6.5.2.1 If, during the sampling run, a component change (i.e., impinger) becomes necessary, a leak check shall be conducted immediately after the interruption of sampling and before the change is made. The leak check shall be done according to the procedure described in Sec. 6.5.1, except that it shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable. If a higher leakage rate is obtained, the tester must void the sampling run.

NOTE: Any correction of the sample volume by calculation reduces the integrity of the pollutant concentration data generated and must be avoided.

6.5.2.2 Immediately after a component change and before sampling is reinitiated, a leak check similar to a pre-test leak check must also be conducted.

6.5.3 Post-test Leak Check - A leak check is mandatory at the conclusion of each sampling run. The leak check shall be done with the same procedures as the pre-test leak check, except that the post-test leak check shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable. If, however, a higher leakage rate is obtained, the tester shall record the leakage rate and void the sampling run.

6.6 Sampling Train Operation

6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, below 28 L/min (1.0 cfm). Maintain a temperature around the probe of 120°C (248° ±25°F).

6.6.2 For each run, record the data on a data sheet such as the one shown in Figure 2. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 2 at least once at each sample point during each time increment and additional readings when significant adjustments (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

6.6.3 Clean the stack access ports prior to the test run to eliminate the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot tube coefficient is 0.84 ±0.02 and the stack gas equivalent density (dry molecular weight) is equal to 29 ±4. APTD-0576 details the procedure for using the nomographs. If the stack gas molecular weight and the pitot tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps are taken to compensate for the deviations.

6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack in order to prevent liquid from backing up through the train. If necessary, the pump may be turned on with the coarse-adjust valve closed.

6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent nonrepresentative dilution of the gas stream.

6.6.6 Traverse the stack cross section, as required by Method 1, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

6.6.7 During the test run, make periodic adjustments to keep the temperature around the probe at the proper levels. Add more ice and, if necessary, salt, to maintain a temperature of less than 20°C (68°F) at the silica gel outlet. Also, periodically check the level and zero of the manometer.

6.6.8 A single train shall be used for the entire sampling run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. An additional train or additional trains may also be used for sampling when the capacity of a single train is exceeded.

6.6.9 When two or more trains are used, separate analyses of components from each train shall be performed. If multiple trains have been used because the capacity of a single train would be exceeded, first impingers from each train may be combined, and second impingers from each train may be combined.

6.6.10 At the end of the sampling run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak check. Also, leak check the pitot lines as described in Method 2. The lines must pass this leak check in order to validate the velocity-head data.

6.6.11 Calculate percent isokinetic variation (see Method 5) to determine whether the run was valid or another test should be made.

7.0 SAMPLE RECOVERY AND PREPARATION FOR ANALYSIS

7.1 Preparation

7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be handled safely, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling because a vacuum will be created, drawing liquid from the impingers back through the sampling train.

7.1.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet, being careful not to lose any condensate that might be present. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used, let any condensed water or liquid drain into the impingers. Cap off any open impinger inlets and outlets. Ground glass stoppers, Teflon® caps, or caps of other inert materials may be used to seal all openings.

7.1.3 Transfer the probe and impinger assembly to an area that is clean and protected from wind so that the chances of contaminating or losing the sample are minimized.

7.1.4 Inspect the train before and during disassembly, and note any abnormal conditions.

7.1.5 Save a portion of all washing solution (methylene chloride, water) used for cleanup as a blank. Transfer 200 mL of each solution directly from the wash bottle being used and place each in a separate, pre-labeled sample container.

7.2 Sample Containers

7.2.1 Container 1 - Probe and Impinger Catches. Using a graduated cylinder, measure to the nearest mL, and record the volume of the solution in the first three impingers. Alternatively, the solution may be weighed to the nearest 0.5 g. Transfer the impinger solution from the graduated cylinder into the amber flint glass bottle. Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all surfaces to which the sample is exposed (including the probe nozzle, probe fitting, probe liner, first impinger, and impinger connector) with methylene chloride. Use less than 500 mL for the entire wash (250 mL would be better, if possible). Add the washings to the sample container.

7.2.1.1 Carefully remove the probe nozzle and rinse the inside surface with methylene chloride from a wash bottle. Brush with a Teflon® bristle brush, and rinse until the rinse shows no visible particles or yellow color, after which make a final rinse of the inside surface. Brush and rinse the inside parts of the Swagelok fitting with methylene chloride in a similar way.

7.2.1.2 Rinse the probe liner with methylene chloride. While squirting the methylene chloride into the upper end of the probe, tilt and rotate the probe so that all inside surfaces will be wetted with methylene chloride. Let the methylene chloride drain from the lower end into the sample container. The tester may use a funnel (glass or polyethylene) to aid in transferring the liquid washes to the container. Following the rinse with a Teflon® brush. Hold the probe in an inclined position, and squirt methylene chloride into the upper end as the probe brush is being pushed with a twisting action through the probe. Hold the sample container underneath the lower end of the probe, and catch any methylene chloride, water, and particulate matter that is brushed from the probe. Run the brush through the probe three times or more. Rinse the brush with methylene chloride or water, and quantitatively collect these washings in the sample container. After the brushings, make a final rinse of the probe as described above.

NOTE: Between sampling runs, brushes must be kept clean and free from contamination.

7.2.1.3 Rinse the inside surface of each of the first three impingers (and connecting tubing) three separate times. Use a small portion of methylene chloride for each rinse. Water will be required for the recovery of the impingers in addition to the specified quantity of methylene chloride. There will be at least two phases in the impingers. This two-phase mixture does not pour well, and a significant amount of the impinger catch will be left on the walls. The use of water as a rinse makes the recovery quantitative. Make a final rinse of each surface, using both methylene chloride and water.

7.2.1.4 After all methylene chloride and water washings and particulate matter have been collected in the sample container, tighten the lid so that solvent, water, and DNPH reagent will not leak out when the container is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Seal the container with Teflon® tape. Label the container clearly to identify its contents.

7.2.1.5 If the first two impingers are to be analyzed separately to check for breakthrough, separate the contents and rinses of the two impingers into individual containers. Care must be taken to avoid physical carryover from the first impinger to the second. The formaldehyde hydrazone is a solid which floats and froths on top of the impinger solution. Any physical carryover of collected moisture into the second impinger will invalidate a breakthrough assessment.

7.2.2 Container 2 - Sample Blank. Prepare a sample blank by using an amber flint glass container and adding a volume of DNPH reagent and methylene chloride equal to the total volume in Container 1. Process the blank in the same manner as Container 1.

7.2.3 Container 3 - Silica Gel. Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. The impinger containing the silica gel may be used as a sample transport container with both ends sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon® caps may be used. The silica gel impinger should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If the silica gel is removed from the impinger, the tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use water or other liquids to transfer the silica gel. If a balance is available in the field, the spent silica gel (or silica gel plus impinger) may be weighed to the nearest 0.5 g.

7.2.4 Sample containers should be placed in a cooler, cooled by although not in contact with ice. Sample containers must be placed vertically and, since they are glass, protected from breakage during shipment. Samples should be cooled during shipment so they will be received cold at the laboratory.

7.3 The dinitrophenylhydrazone derivative is then analyzed by high performance liquid chromatography (HPLC) (Method 8315) or other appropriate technique.

8.0 CALIBRATION

8.1 Probe Nozzle - Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When the nozzles become nicked or corroded, they shall be replaced and calibrated before use. Each nozzle must be permanently and uniquely identified.

8.2 Pitot Tube - The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked or corroded and if it meets design and intercomponent spacing specifications.

8.3 Metering System

8.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to

correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak check be conducted. For metering system having diaphragm pumps, the normal leak check procedure will not detect leakages within the pump. For these cases, the following leak check procedure will apply: make a ten-minute calibration run at 0.00057 m^3/min (0.02 cfm). At the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

8.3.2 After each field use, check the calibration of the metering system by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). Set the vacuum at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

8.3.3 Leak Check of Metering System - The portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. Use the following procedure: Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 - 18 cm (5 - 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by greater than 5%, either the test series must be voided or calculations for test series must be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

8.4 Probe Heater - The probe heating system must be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.5 Temperature Gauges - Each thermocouple must be permanently and uniquely marked on the casting. All mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F (American Society for Testing and Materials) specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at the ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change greater than 1.5%.

8.5.1 Impinger and Dry-gas Meter Thermocouples - For the thermocouples used to measure the temperature of the gas leaving the impinger train, a three-point calibration at ice water, room air, and boiling water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2°C (3.6°F) with those of the absolute value of the reference thermometer.

8.5.2 Probe and Stack Thermocouple - For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice water, boiling water, and hot oil

bath temperatures must be performed. Use of a point at room air temperature is recommended. The thermometer and thermocouple must agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

8.6 Barometer - Adjust the barometer initially and before each test series to agree to within ±2.5 mm Hg (0.1 in. Hg) of the mercury barometer or the corrected barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

8.7 Triple-beam or Electronic Balance - Calibrate the balance before each test series, using Class S standard weights. The weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

9.0 CALCULATIONS

Perform calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

9.1 Total Formaldehyde - Determine the total formaldehyde in mg, using the following equation:

$$\text{Total mg formaldehyde} = C_d \times V \times DF \times \left[\frac{\text{g/mole aldehyde}}{\text{g/mole DNPH derivative}}\right] \times 10^{-3} \text{ mg/µg}$$

where:

C_d = measured concentration of DNPH-formaldehyde derivative, µg/mL
V = organic extract volume, mL
DF = dilution factor

9.2 Formaldehyde Concentration In Stack Gas - Determine the formaldehyde concentration in the stack gas using the following equation:

$$C_f = K \text{ [total formaldehyde, mg]} / V_{m(std)}$$

where:

K = 35.31 ft^3/m^3 if $V_{m(std)}$ is expressed in English units
= 1.00 m^3/m^3 if $V_{m(std)}$ is expressed in metric units
$V_{m(std)}$ = volume of gas sample as measured by dry gas meter, corrected to standard conditions, dscm (dscf)

9.3 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop are obtained from the data sheet.

9.4 Dry Gas Volume - Calculate $V_{m(std)}$ and adjust for leakage, if necessary, using the equation in Sec. 6 of Method 5.

9.5 Volume of Water Vapor and Moisture Content - Calculate the volume of water vapor and moisture content from Equations 5-2 and 5-3 of Method 5.

10.0 DETERMINATION OF VOLUME TO BE SAMPLED

To determine the minimum sample volume to be collected, use the following sequence of equations.

10.1 From prior analysis of the waste feed, the concentration of formaldehyde (FORM) introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the maximum amount of FORM allowed to be present in the effluent. This amount may be expressed as:

$$\text{Max FORM}_i \text{ Mass} = [(WF)(\text{FORM}_i \text{ conc})(100 - \%DRE)] / 100$$

where:

WF = mass flow rate of waste feed per h, g/h (lb/h)
FORM_i = concentration of FORM (wt %) introduced into the combustion process
DRE = percent Destruction and Removal Efficiency required
Max FORM = mass flow rate (g/h [lb/h]) of FORM emitted from the combustion sources

10.2 The average discharge concentration of the FORM in the effluent gas is determined by comparing the Max FORM with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary Method 1 - 4 determinations:

$$\text{Max FORM}_i \text{ conc} = [\text{Max FORM}_i \text{ Mass}] / DV_{eff(std)}$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf)
FORM_i conc = anticipated concentration of the FORM in the exhaust gas stream, g/dscm (lb/dscf)

10.3 In making this calculation, it is recommended that a safety margin of at least ten be included.

$$[LDL_{FORM} \times 10] / [\text{FORM}_i \text{ conc}] = V_{tbc}$$

where:

LDL_{FORM} = detectable amount of FORM in entire sampling train
V_{tbc} = minimum dry standard volume to be collected at dry-gas meter

10.4 The following analytical detection limits and DNPH Reagent Capacity (based on a total volume of 200 mL in two impingers) must also be considered in determining a volume to be sampled.

11.0 QUALITY CONTROL

11.1 Sampling - See EPA Manual 600/4-77-027b for Method 5 quality control.

11.2 Analysis - The quality assurance program required for this method includes the analysis of field and method blanks, procedure validations, analysis of field spikes, and analysis of reagent checks. The assessment of combustion data and positive identification and quantitation of formaldehyde are dependent on the integrity of the samples received and the precision and accuracy of the analytical methodology. Quality Assurance procedures for this method are designed to monitor the performance of the analytical methodology and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

11.2.1 Field Blanks - Field blanks may be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, methylene chloride and water, and unused DNPH reagent. In the case of results exceeding regulatory limits, field blank data may be useful for convincing the regulatory official that contamination was the cause. This may result in retesting rather than a violation charge. Collection of the field blank is optional but recommended.

11.2.2 Method Blanks - A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

11.2.3 Field Spikes - A field spike is performed by introducing 200 µL of the Field Spike Standard into an impinger containing 200 mL of DNPH solution. Standard impinger recovery procedures are followed and the field spike sample is returned to the laboratory for analysis. The field spike is used as a check on field handling and recovery procedures. An aliquot of the field spike standard is retained in the laboratory for derivatization and comparative analysis.

11.2.4 Matrix Spike Sample - In addition to those stack samples necessary for basic data needs, one complete sample (of the same time duration) must be collected for use as a matrix spike sample as described in Sec. 8.0 of Method 8315. This sample must be recovered and shipped in exactly the same manner as the other stack samples. Every effort should be made to ensure that this sample represents the average stack matrix of the sample batch. For example, the matrix spike sample should be taken the same day as the other samples in the group, if at all possible. If it is known or suspected that the stack gas matrix is varying widely during the overall sampling run, it is advisable to take more than one matrix spike sample and composite them.

11.2.5 DNPH Reagent Checks - An aliquot of the extracted DNPH reagent is prepared and analyzed according to the procedure in Sec. 5.4.3 to ensure that the background in the reagent is acceptable for field use.

12.0 METHOD PERFORMANCE

Method performance evaluation - The expected method performance parameters for precision, accuracy, and detection limits are provided in Table 3.

13.0 REFERENCES

1. 40 CFR Part 60, Appendix A, Test Methods.

2. Martin, R.M., "Construction Details of Isokinetic Source-Sampling Equipment", U.S. Environmental Protection Agency, Research Triangle Park, NC, Air Pollution Technical Document (APTD) 0581, April 1971.

3. Rom, J.J., "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment", U.S. Environmental Protection Agency, Research Triangle Park, NC, Air Pollution Technical Document (APTD) 0576, March 1972.

4. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials (ASTM), Philadelphia, PA, 1974, pp. 617-622.

TABLE 1

APPROXIMATE AMOUNT OF CRYSTALLINE DNPH USED
TO PREPARE A SATURATED SOLUTION

Amount of Moisture in DNPH	Weight Required per 8 L of Solution
10 weight percent	36 g
15 weight percent	38 g
30 weight percent	46 g

TABLE 2

OPTIMUM STACK DETECTION LIMITS[a] AND REAGENT SAMPLING CAPACITY[b] FOR FORMALDEHYDE ANALYSIS

Analyte	CAS No.	Detection Limit[a] (ppbv)	Reagent Capacity	
			ppmv	mg/m^3
Formaldehyde	50-00-0	36	7.5	9
Acetaldehyde	75-07-0	34	7.5	14
Acetone	67-64-1	30	7.5	17
Propionaldehyde	123-38-6	30	7.5	17
Butyraldehyde	123-72-8	30	7.5	21
Valeraldehyde	110-62-3	30	7.5	25
Isovaleraldehyde	590-86-3	28	7.5	25
Hexaldehyde	66-25-1	26	7.5	30
Benzaldehyde	100-52-7	28	7.5	33
Acetophenone	98-86-2	28	7.5	37
o-Tolualdehyde	529-20-4	26	7.5	37
m-Tolualdehyde	620-23-5	26	7.5	37
p-Tolualdehyde	104-87-0	26	7.5	37
2,5-Dimethylbenzaldehyde	5779-94-2	24	7.5	41
Isophorone	78-59-1	24	7.5	42

[a]Detection limits are determined based on 400 mL of reagent and 10 times the instrument detection limit using hydrazones in solvent, and therefore, represent the optimum capability of the method.

[b]Based on 400 mL of reagent, a reagent concentration of 10 mM DNPH, a 1.3 cubic meter sample size and a safety factor of 10.

TABLE 3

EXPECTED METHOD PERFORMANCE BASED ON DUAL TRAINS

Compound	Precision (%RPD)[1]	Accuracy (%)[2]	Detection Limit (ppbv)[3]
Formaldehyde	±21	±10	±90
Acetaldehyde	±17	±21	±40
Propionaldehyde	±49	±23	±60
Acetophenone	±44	±10	±10
Isophorone	±9	±8	±10

[1] Relative percent difference limit for dual trains.
[2] Limit for field spike recoveries.
[3] The lower reporting limit having less than 1% probability of false positive detection.

FIGURE 1
SAMPLING TRAIN FOR ALDEHYDES AND KETONES

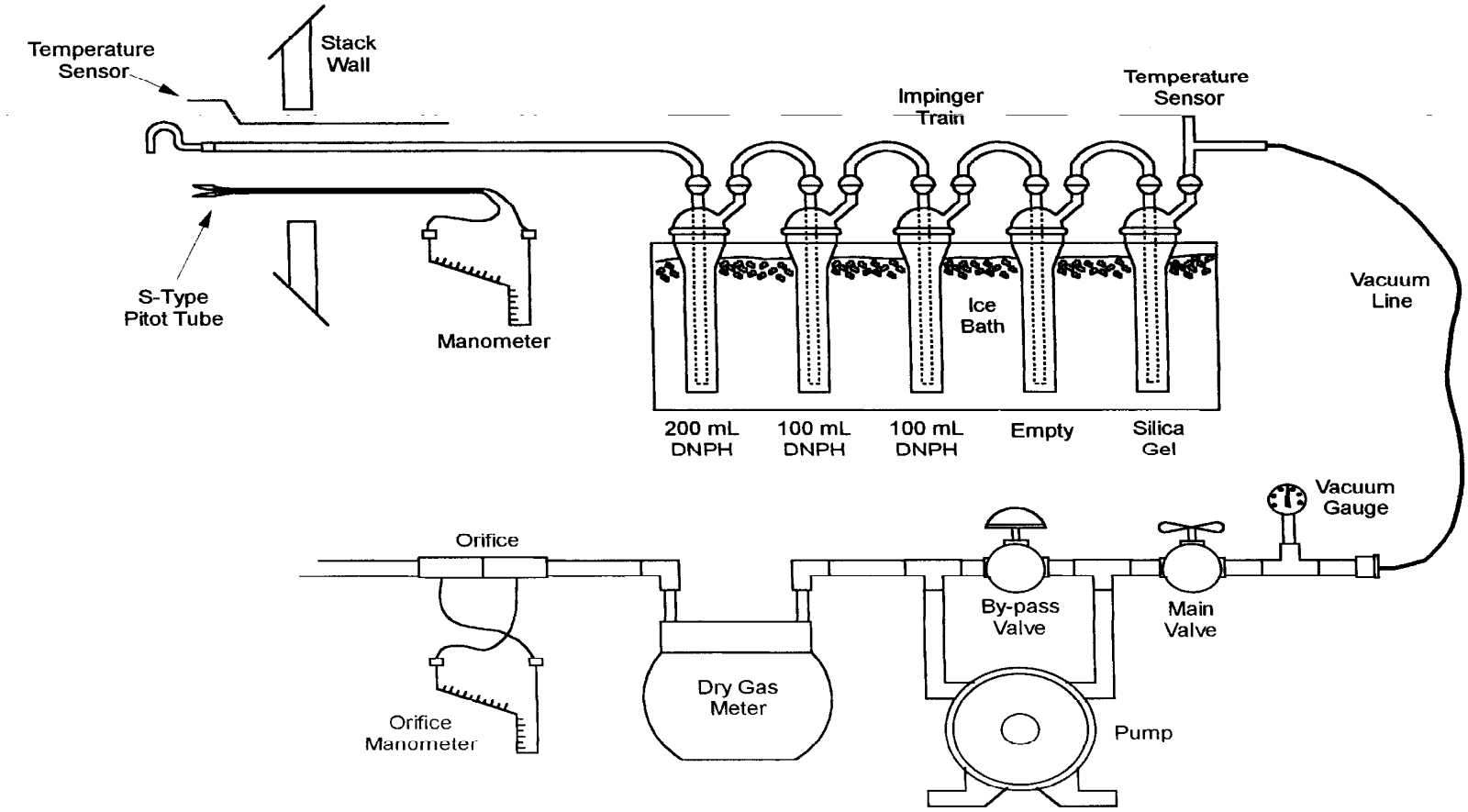

FIGURE 2
FIELD SAMPLING DATA FORM

Plant _____

Location _____

Operator _____

Date _____

Run Number _____

Sample Box No. _____

Meter Box No. _____

Meter H@ _____

C Factor _____

Pitot Tube Coefficient C_p _____

Schematic of Stack Cross Section

Ambient Temperature _____

Barometric Pressure _____

Assumed Moisture % _____

Probe Length _____ m (ft)

Nozzle Ident. No. _____

Average Calibrated Nozzle Diameter _____ cm (in)

Probe Heating Setting _____

Leak Rate _____ m^3/min (cfm)

Probe Liner Material _____

Static Pressure _____ mm Hg (in.Hg)

Filter No. _____

Traverse Point Number	Sampling Time (Min.)	Vacuum mm Hg (in. Hg)	Stack Temperature (T_s) °C (°F)	Velocity head (ΔP) mm (in) H_20	Pressure Differential Across Orifice Meter mm (H_20) (in. H_20)	Gas Sample Volume m^3 (ft^3)	Gas Sample Temp. at Dry Gas Meter		Filter Holder Temp. °C (°F)	Temp. of Gas Leaving Last Impinger °C (°F)
							Inlet °C (°F)	Outlet °C (°F)		
Total							Avg	Ave		
Average										

APPENDIX A

ADDITION OF A FILTER TO THE FORMALDEHYDE SAMPLING TRAIN

As a check on the survival of particulate material through the impinger system, a filter can be added to the impinger train either after the second impinger or after the third impinger. Since the impingers are in an ice bath, there is no reason to heat the filter at this point.

Any suitable medium (e.g., paper, organic membrane) may be used for the filter if the material conforms to the following specifications:

1) The filter has at least 95% collection efficiency (<5% penetration) for 3 µm dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

2) The filter has a low aldehyde blank value (<0.015 mg formaldehyde/cm^2 of filter area). Before the test series, determine the average formaldehyde blank value of at least three filters (from the lot to be used for sampling) using the applicable analytical procedures.

Recover the exposed filter into a separate clean container and return the container over ice to the laboratory for analysis. If the filter is being analyzed for formaldehyde, the filter may be recovered into a container or DNPH reagent for shipment back to the laboratory. If the filter is being examined for the presence of particulate material, the filter may be recovered into a clean dry container and returned to the laboratory.