Purpose: To determine the presence of a flammable liquid in fire debris.

Responsibility: Section analyst or designee

Materials:

1. Heating mantles- capable of heating a pint, quart or gallon can
2. Thermometer - 0-100 C (reference only)
3. Vacuum source (mechanical pump)
4. Cans- unused, lined paint cans: pint, quart, gallon and five gallons
5. Standard laboratory glassware
6. Reagent grade or better pentane and acetone
7. Appropriate standards and controls
8. Activated charcoal tubes
9. Gas chromatograph- suitable for temperature programming. Set up for using capillary GC columns, and equipped with a flame ionization detector (FID).
10. Data system - capable of reintegrating raw data and providing a hard copy printout.

Procedures:

I. Initial Examination

1. Open the submitted item (can etc.), and observe the contents.

2. Sniff the contents to determine if a flammable liquid odor or other unique odor is present.

3. Note on the worksheet a description of the material, if a unique odor is present and the amount of the contents (if in a can).

4. Write the lab case number, the item number and the analyst's initials on the top or side of the can.

5. NOTE: If materials are present which require subsequent testing, the nature of the materials and the type of testing should be considered before heating.

II. Recovery of organic compounds

A. Heated headspace

1. Punch a hole in the lid of the submitted can.

2. Cover the hole with a piece of plastic adhesive tape

3. Heat the can until a bulge appears under the tape.

4. Remove the can, using a disposable 3.0 ml syringe; inject 1.0 ml (cc) of headspace into the GC.

B. Adsorption/elution

1. Check the outside of the can for dirt etc. If present, clean the can with water.

2. Punch 1-4 evenly spaced holes around the circumference of the can near the bottom. If there is water in the can, place the holes approximately 1.0 inch above the water level.

3. Replace the original lid with a lid adapted to hold a charcoal tube. Snip the ends of the charcoal tube and place it into the Swagelok fitting. Insert the tube with the "open" end in the holder.

4. Insert the can into the heating mantle (the temperature is approximatel80 C). In some cases it may be necessary to insert a dial thermometer through the lid until it touches the contents of the can (determined by analyst).

5. Connect the end of the charcoal tube to the vacuum line, and turn on the vacuum pump.

6. After an appropriate period of time ie. when condensation appears in the open area of the tube; or if the thermometer is used, when the temperature of the contents reaches 40-50 C. CAUTION: Avoid sample temperatures above 80 C to minimize the stripping of "lighter" components of flammable liquids.

7. Insert the "full" end of the charcoal tube into a small (4.0 ml) glass vial, and using a Pasteurpipette, add 0.5 ml of pentane to the "open"end of the charcoal tube.

8. Elute the pentane through the charcoal tube. Repeat the procedure with a second 0.5 ml of pentane.

9. If the sample extract cannot be analyzed the same day, store the extract in the refrigerator or freezer.

10. If a petroleum distillate or gasoline odor is not discernible, concentrate the sample by using a stream of nitrogen gas. Concentrate the sample to approx. 100 uL.

11. Inject approximately 1.0 ul of the extract into the GC for analysis. Clean the syringe according to SOP

12. Thoroughly rinse the bottom of the modified can lid and the interior of the Swagelok fitting with acetone. This removes condensed water and any organics from the lid and is necessary to prevent cross contamination of samples.

C. Solvent Extraction

1. Fill a suitable beaker half way with all, or a portion of, the contents of the submitted item, and place in the fume hood.

2. Add reagent grade pentane. The amount needed will vary with the amount of debris. Add the solvent slowly, washing the debris in the process. Swirl gently to make sure the solvent comes in contact with all the debris.

3. Filter the extract and collect in a second beaker.

4. Concentrate (either by heating or evaporation) until most of the C5 is removed.

5. Transfer to a 4.0 ml glass vial and concentrate as above.

6. Inject 1.0 ul into the GC and analyze.

Sample preservation:

After each sample is analyzed, solvent is added to the sample vial and it is returned to the submitted item can. The can is then sealed with evidence tape and initialed by the analyst.

References:

ASTM Standard Methods: Designation E 1387-95 (modified).
GC-MS Guide to Ignitable Liquids
ASTM Methods: E 1386, E 1388, E 1413