Safety Practices Using Organic Solvents in Flame Atomic Absorption Spectroscopy

Application Note

Atomic Absorption

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Introduction

Atomic absorption spectroscopy has found broad acceptance in the analysis of petroleum products and the determination of metals after organic solvent extraction. These analytical methods have been carried out in reasonable safety over many years, but there is no doubt that the combination of an open flame and flammable solvents is potentially very dangerous. Chemists working with this combination are characteristically aware of the potential hazards, and the majority is extremely safety-conscious. But, humans are fallible; even the most careful analyst can sometimes be lulled into a false sense of security; even the most thoughtful chemist can be forgetful; and even the most disciplined worker may suffer a lapse of safety awareness.

With the increasing work burden now being placed on analytical laboratories, it seems opportune to remind analysts of the constant need for safe working practices when using flammable solvents in the presence of a naked flame. We hope that the following notes will help to promote safety awareness in laboratories, and remind analysts of factors that can so easily be overlooked in any analytical program involving flammable solvents.

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Table 1. Organic Solvents Widely Used in Flame Atomic Absorption: (The Flash Point is the Lowest Temperature at Which the Liquid Gives Sufficient Vapor to Form an Ignitable Mixture with Air and Produce a Flame when an Ignition Source is Brought Near the Surface of the Liquid)

Solvent	Flash point (°C)	Boiling point (°C)	Specific gravity
4-methyl-pentan-2-one (MIBK)	22	118	0.79
2-methyl, propan-1-ol	23	108	0.78
m-xylene	29	139	0.80
cyclohexanone	34	155	0.948
kerosene	39–74	175–325	0.78
iso-amylalcohol	43	132	0.81
3-heptanone (ethylbutylketone)	46	148	0.818
Shellsol T	50	186–214	0.75
2, 6-dimethyl,heptan-4-one (DIBK)	60	166	0.806
cyclohexanol	68	161	0.96
tetrahydronaphthalene (Tetralin)	71	207	0.97

1. All relevant safety regulations governing the use of flammable solvents must be strictly followed.
2. When initially selecting an organic solvent, choose a solvent having the highest flash point possible consistent with your analytical requirements. Note that recent methods have been developed for the determination of metals in oils and gasoline, which do not require conventional flammable solvents [6]. The sample is emulsified using Emulsogen LBH, or Emulsifier MS 112 (Hoechst) and the resultant gel can be quickly dispersed in water. The procedure allows aqueous standards to be used, and the emulsions are reported to remain stable for several months.
3. Never use a solvent having a specific gravity lower than 0.75, otherwise the liquid seal can be breached. This can cause a flashback and create an explosion or fire hazard.
4. Never leave uncovered containers of flammable solvent standing near the burner. When aspirating such solvents always use a narrow-necked vessel or a container and feed the capillary tubing through a 2 mm diameter hole in the cover. Always use the smallest volume of solvent consistent with your analytical requirements, ensuring the sample vessel is sealed when the sample is not being aspirated.
5. Never prepare organic samples and standards in or near the atomic absorption spectrophotometer or any other ignition source. Samples should be prepared in another room or on another laboratory bench completely separated from the atomic absorption spectrophotometer. On completion of the sample preparation the organic reagents must be returned to a storage area away from the vicinity of the atomic absorption spectrophotometer. It is advisable to carry out organic sample preparations in a fume cupboard.
6. Never aspirate chlorinated hydrocarbons such as chloroform or carbon tetrachloride since they will readily decompose in the flame to produce phosgene-an extremely hazardous compound.
7. Gasoline, carbon-disulfide and tetrahydrofuran (THF) must not be aspirated directly as these have very low flash points and can create an extreme fire hazard.
8. Use small waste vessels; empty them frequently; ensure that your waste vessel is at a safe distance from the instrument. Locate your waste vessel in an open, ventilated position where you can see it. Never locate the vessel in a confined space. Use Buna N tubing for the drain tube, and always secure the tube to the liquid trap outlet with a hose clamp.
9. A vent tube of solvent resistant material must be connected to the vapor vent on the liquid trap and led to a location where organic solvent vapors can be safely discharged. Secure the tubing to the trap vent with a hose clamp.
10. When your analytical program has been completed:
    a. Turn the flame off exactly as described in your spectrophotometer operation manual.
    b. Remove all samples from the vicinity of the instrument.
    c. Allow the burner to cool.
    d. Use protective gloves and remove the burner from the spray chamber.
    e. Detach the liquid trap from the spray chamber and pour the organic solvent into an organic waste container. Ensure that the liquid trap is emptied.

f. Clean the liquid trap, spray chamber and nebulizer bung thoroughly with alcohol so that all traces of the original organic residues are removed.

g. Re-assemble the spray chamber, liquid trap and nebulizer bung exactly as described in your spectrophotometer operation manual.

h. If you are proposing to leave the instrument unused, fill the liquid trap with water. NEVER leave hazardous solutions standing in the liquid trap. If you are proposing to carry out another analytical program, fill the liquid trap with the same solvent being used for your samples.

i. Empty the waste vessel and ensure that all organic residues are removed.

11. Ensure that nitric or perchloric acid residues are not mixed with organic residues.
12. Always keep the burner slot clear of carbon or other solids. Progressive burner blockage can increase the static pressure in the liquid trap to the point at which the liquid seal is breached.
13. Do not attempt to clean the burner while the flame is burning.
14. Never leave a flame unattended.

Notes

It is thoroughly bad practice to attempt to alternate directly between analyses using organic solvents and aqueous solutions. Always follow procedure number 10 so that the complete spray chamber system will be ready for use with another organic solvent program or an aqueous solution program.

Some organic solvents such as MIBK and xylene may distort the spray chamber and associated polypropylene components. In laboratories where such solvents are continually used, an additional, complete spray chamber should be made available. The two complete spray chamber assemblies can then be used alternately on the shortest practicable working cycle. While one assembly is being employed, the other can be stripped down and left in a fume cupboard for the duration of its off-duty cycle. The drying out process should effectively remove any temporary distortion caused by the absorption of solvent.

All other safety requirements noted in your operation manual must be observed.

References

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