

THERMAL CHARACTERIZATION OF METALLIC PAINTS AND BALL POINT PEN INKS ON PAPER WITH A MULTIPURPOSE PTV INJECTOR

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Aims

The ATAS OPTIC 3 is a multipurpose PTV injector. In addition to the normal modes of GC injector operation it features a thermal desorption mode for solid samples. The upper desorption temperature limit being as high as 600 degrees the injector can also be used as a pyrolyzer.

The aim of the study was to look at the applicability of the injector in the thermal characterization of metallic paints and ball point pen inks focusing on its sensitivity and performance compared to dedicated desorbers and pyrolyzers. In theory, better overall sensitivity would be obtained with in-line desorption compared to that obtained with external desorption units.

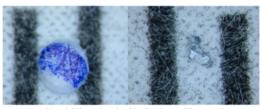


The OPTIC injector can accommodate large diameter liners. Sample particle is place in to an insert tubes that fits into the liner.

Methods

Microdots of ball point pen inks on paper were punched out of paper and placed in the insert tubes of the OPTIC 3 injector. Paint chips were placed as such in the insert tubes. The study targeted especially in the determination of smallest sample sizes possible. For ink samples relatively low TD temperatures (around 200 °C) were used while the paint samples were pyrolyzed at temperatures near the upper injector limit.

The OPTIC 3 was installed on an Agilent 6890/5973 GC/MS. The desorbed analytes were separated with an HP-5 column (Agilent, 30m x 0.25mm x 0.25 μ m)



A paper dot with an ink line and a paint chip placed on a millimetre scale.

Results

The dead volume of the OPTIC injection area remains low enough to enable the transfer of the desorbed compounds into the capillary in a splitless mode in a reasonable time. This provides with excellent overall sensitivity. The disadvantage of many dedicated pyrolyzers or desorbers is that they have to be operated in split mode due to their large dead volumes. Sensitivity is lost unless cryotrapping is arranged for before the split valve.

The aim of many forensic analyses is not only to determine the chemical composition of samples but to establish the chemical similarity of a sample pair. Thermal analysis provides with a lot of differentiation power in cases where the sample size or low organic content of the sample render the measurements difficult with other means of instrumental analysis.

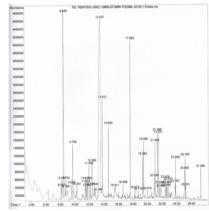
Metallic paints were chosen for the study because of their low organic binder content. This emphasizes the demand for sensitivity. The paints studied were of the wheel chrome type and randomly chosen from among identically coloured samples in the NBI paint collection.

Ball point pen ink lines on paper represent another example of where sensitivity is called for.

Results are encouraging in that the paints and inks can be differentiated as effectively as with the Pyrola 2000 pyrolyzer that was used as a reference. More importantly, the sample sizes required for the OPTIC 3 are considerably smaller than those for the Pyrola. This extends the applicability of thermal characterization and pyrolysis in trace evidence case work where the actual very small sample sizes often prevent it. Reproducibilities matching those with Pyrola 2000 can be reached.

The differentiation power of thermal analysis was highlighted in a study that involved a set of 10 different wheel chromes with roughly matching hues of silver. All were easily differentiated with the OPTIC3.

A correct answer was reached with the OPTIC3 in a collaborative test organised by the European Network of Forensic Science Institutes in 2009 that involved the differentiation of 8 ball point ink lines on paper.



TD operation for metallic paints: Temperature 500 °C
Desoption time: 3 min
Desorption Column flow: 3 ml/min
Column flow: 1 ml/min

Conclusions

The multipurpose OPTIC 3 PTV injector provided with excellent overall sensitivity in the thermal characterization of a set of metallic paints and ball point pen inks on paper.

OPTIC 3 extends the sample size range that thermal analysis can be applied to in NBI.