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## **Report on the GC-MS analyses of binding media from the painting by Petr Brandl (O 578), the National Gallery, Prague, CZ**

### **Introduction**

Three samples of organic materials were analysed for the presence of lipids and resinous media by gas chromatography - mass spectrometry technique. The analytical procedure for the analysis of lipids is based on the transesterification of fatty acids and the determination of their relative ratios to identify particular lipids and the analytical procedure for the analysis of resinous binding media is based on the esterification of resinous acids followed by the identification of particular resins according to their resinous acid methyl esters.

## **Experimental**

### Analytical procedure for lipids and resins:

#### *Transmethylation with Meth-Prep II:*

The solid reference standards and the samples were placed in vials with conical inserts and then treated with a 0.2M methanolic solution of Meth-Prep II (30 µl) and a solvent mixture (methanol: toluene, 1:2; 70µl). The sealed vials were heated to 60°C for 1 hour, removed from the heat, cooled to room temperature, and centrifuged. The clear solutions were replaced into new vials and 1µl injected into a GC inlet at a temperature of 300°C.

#### Apparatus:

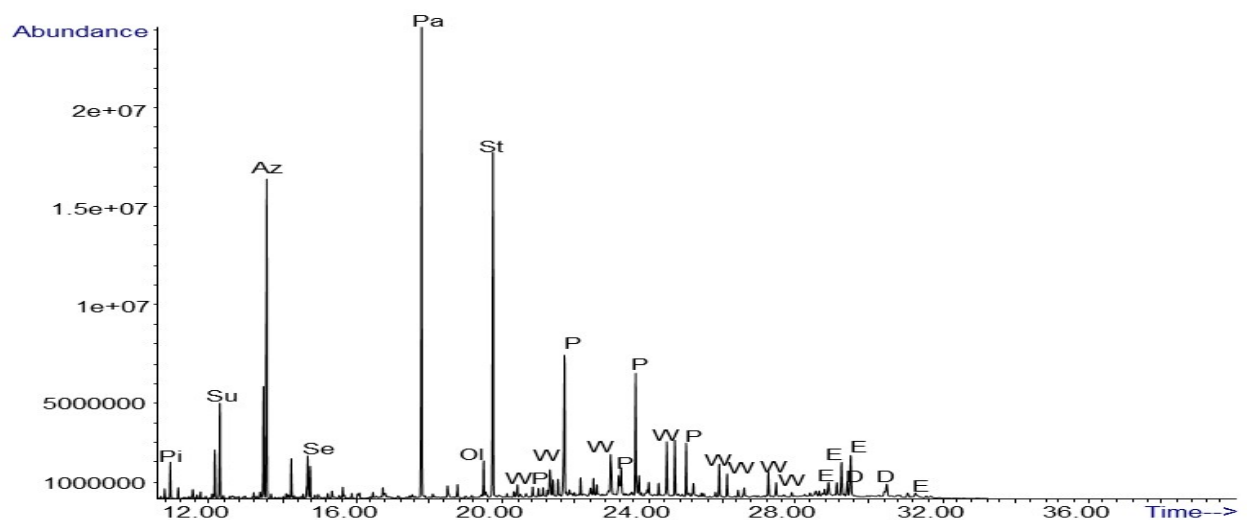
GC-MS analyses were performed on a 6890N gas chromatograph connected to a quadrupole mass spectrometer, model 5973N (both Agilent Technologies, USA).

## **Results and discussion**

The results are summarised in Table 1 and depicted in Figures 1-3:

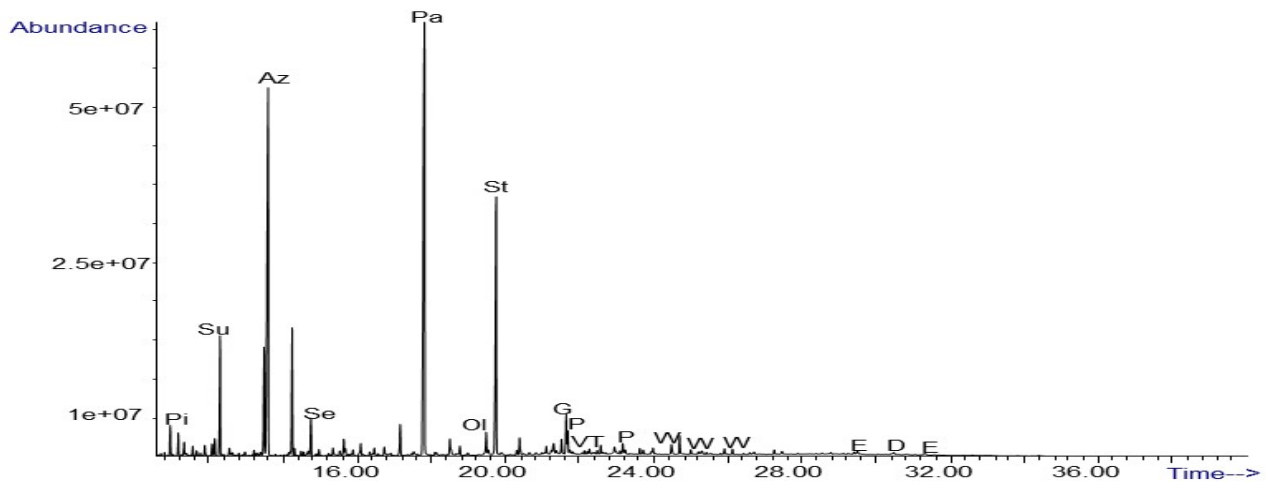
sample	sample description	lipids	resins	wax
1	Simeon s Ježíškem/ podklad z okraje obrazu (dark red thicker piece)	- <u>linseed oil</u> (Pa/St~1.4)	- pine resin - traces of elemi - traces of dammar	- beeswax
2	Simeon s Ježíškem/ pastózní malba – noha Ježíška (pastel beige-pink layer + varnish)	- <u>walnut oil</u> (Pa/St~2.4)	- traces of pine resin - traces of elemi - traces of dammar	- traces of beeswax
3	Simeon s Ježíškem/ pastózní malba s lazurní vrstvou – ruka Ježíška (pastel beige-pink layer + varnish)	- <u>walnut oil</u> (Pa/St~2.8)	- traces of pine resin - traces of dammar	- traces of beeswax

**Table 1: The composition of the samples derived by GC-MS analyses**



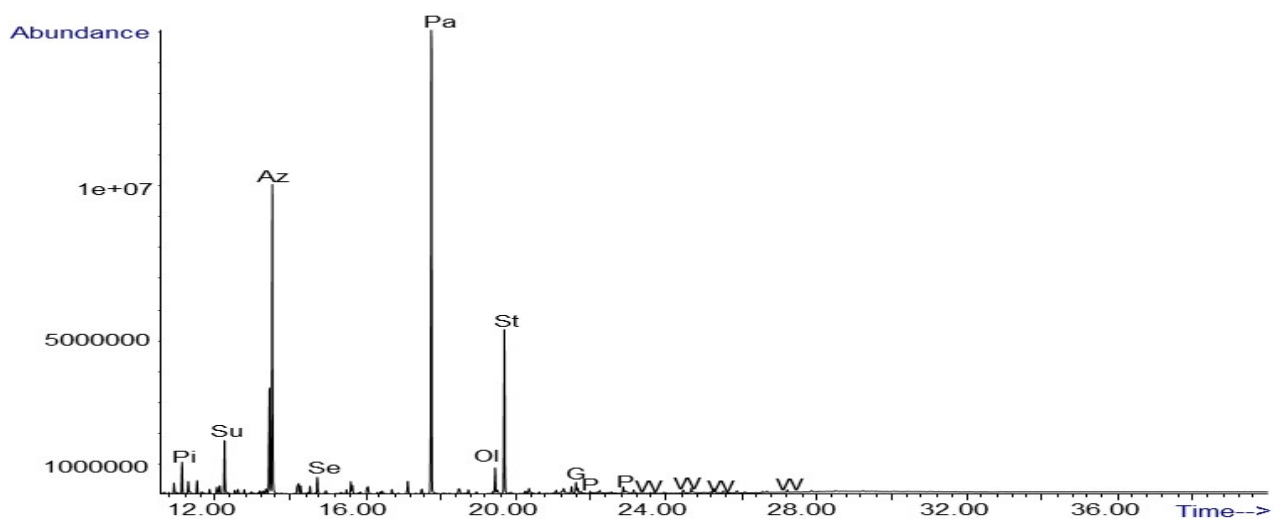
**Figure 1. The chromatogram of the organic material from sample 1 after methylation**

Note: fatty acids from linseed oil (Pi...pimelic acid, Su...suberic acid, Az...azelaic acid, Se...sebacic acid, Pa...palmitic acid, Ol...oleic acid, St...stearic acid); P...pine resin diterpenes; W...long chain esters and alkanes from beeswax; E...elemi components; D...dammar triterpenes.



**Figure 2. The chromatogram of the organic material from sample 2 after methylation**

Note: glycerol (G) and fatty acids from walnut oil (Pi...pimelic acid, Su...suberic acid, Az...azelaic acid, Se...sebacic acid, Pa...palmitic acid, Ol...oleic acid, St...stearic acid); G...glycerol; P...pine resin diterpenes; VT...Venice turpentine, W...long chain esters and alkanes from beeswax; E...elemi components; D...dammar triterpenes.



**Figure 3. The chromatogram of the organic material from sample 3 after methylation**

Note: glycerol (G) and fatty acids from walnut oil (Pi...pimelic acid, Su...suberic acid, Az...azelaic acid, Se...sebacic acid, Pa...palmitic acid, Ol...oleic acid, St...stearic acid); G...glycerol; P...pine resin diterpenes; W...long chain esters and alkanes from beeswax.

## Conclusions

All three samples are based on drying oil. In particular, **sample 1** (see Fig 1) is based on linseed oil, while **samples 2** and **3** are composed of walnut oil (see Figs. 2 and 3). Drying oils were identified according to the ratios of fatty acids, namely palmitic acid/ stearic acid (Pa/St), which is a marker to characterise the particular drying oil.

Besides the drying oils, a series of resins, originating probably from the varnish, were also detected. Pine resin (*Pinus* species) was identified according to the presence of diterpenes like dehydro-abietic acid (DHA) and its oxidation products. Larixol is one of the markers typical for Venice turpentine (larch trees, *Larix* species), triterpenes  $\alpha$ -amyrin and  $\beta$ -amyrin helped to confirm the presence of elemi resin (*Burseraceae*), and oxidised dammar resin (*Dipterocarpoideae*) was found according to the triterpenoid compounds like 20,24-epoxy-25-hydroxy-3,4-seco-dammaren-3-oic acid methyl ester.

Long chain esters and alkanes are typical for beeswax, which comes from a previous restoration treatment.