

Article

## Raman Microscopy Applied to the Analysis of the Pigments Used in Two Persian Manuscripts

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As gravuras de dois manuscritos Persas, Ms Pers 1 (UCL) "Anatomia do Corpo" e Ms Pers 2 (UCL) "Elogio à Poesia", da coleção da Biblioteca D.M.S. Watson da University College London, foram analisados por microscopia Raman. Os seguintes pigmentos foram identificados: sulfeto de mercúrio vermelho, HgS, óxido de chumbo vermelho, Pb<sub>3</sub>O<sub>4</sub>, negro de fumo e negro marfim (à base de carbono amorfo), branco de chumbo, 2PbCO<sub>3</sub>.Pb(OH)<sub>2</sub>, orpimento, As<sub>2</sub>S<sub>3</sub>, hidróxido de ferro hidratado, FeO(OH).nH<sub>2</sub>O e lazurita, Na<sub>8</sub>[(AlO<sub>2</sub>)<sub>6</sub>(SiO<sub>2</sub>)<sub>6</sub>]S<sub>n</sub>.

The palettes of two Persian manuscripts Ms Pers 1 (UCL) 'Anatomy of the Body' and Ms Pers 2 (UCL) 'Poetry in Praise' from the collection in the D.M.S. Watson Library at University College London were analyzed by Raman microscopy. The pigments vermilion HgS, red lead Pb<sub>3</sub>O<sub>4</sub>, lamp black and ivory black (based on amorphous carbon), white lead 2PbCO<sub>3</sub>.Pb(OH)<sub>2</sub>, orpiment As<sub>2</sub>S<sub>3</sub>, hydrated ferric hydroxyoxide FeO(OH).nH<sub>2</sub>O, and lazurite Na<sub>8</sub>[(AlO<sub>2</sub>)<sub>6</sub>(SiO<sub>2</sub>)<sub>6</sub>]S<sub>n</sub> were identified.

**Keywords:** Raman microscopy, pigments, Persian manuscripts

### Introduction

Raman microscopy is recognized as a non-destructive analytical technique which has both high sensitivity and high spatial resolution<sup>1-3</sup>, and is therefore very suitable for the *in situ* analysis of various artistic masterpieces<sup>4-8</sup>. It is nowadays often used to characterize the palette of illuminated manuscripts<sup>9-11</sup>. The D.M.S. Watson Library at University College London has a valuable collection of rare books and manuscripts, many of codicological and paleographic interest, but none having had its palette characterized by modern instrumental methods of analysis. Two Persian manuscripts (Ms Pers 1 (UCL) 'Anatomy of the Body' and Ms Pers 2 (UCL) 'Poetry in Praise') were chosen for the analysis of their pigments by Raman microscopy in order to characterize their palette, and to find out possible similarities between them, information about their manufacture, the social categories of their initial owners<sup>12</sup>, and the possible transformations undergone by the pigments during their lifetime.

There is very little information about these two Mss. The Ms Pers 1 is mentioned, without commentary, by J.D. Pearson as an "anatomical treatise illustrated with rather crude drawings"<sup>13</sup>. Only a library label gives information

about the former owner of Ms Pers 2 (UCL) 'Poetry in Praise'. Some photographs and microphotographs from each Ms are presented.

### Experimental

The analytical samples consist of the pigments from particular areas of a miniature, decoration or colored letter selected from folios of each manuscript. An Olympus BH-2 microscope was coupled to a Dilor XY triple-grating spectrometer which had an intensified photodiode array detector. Excitation lines of wavelengths 488.0, 514.5, 568.2, and 647.1 nm were obtained from Coherent Radiation 52-krypton ion and Coherent Innova 70-argon ion lasers and, in conjunction with narrow band-pass filters from Ealing Electro Optics, were used to excite Raman spectra of selected samples of pigment. Laser powers of 3 - 10 mW were used in order to avoid burning the sample. Integration times of 2 - 30 s and 50 - 200 accumulations were used. Small single crystals or stains (spots) of pigment, 1-10 μm in diameter, were analyzed using a 100x objective in conjunction with a 10x eyepiece. At this magnification, even the smallest movement of a sample due to vibration of the manuscript page or any shrinkage of the parchment (paper)

in the atmosphere of the laboratory would put the sample out of focus and greatly diminish the signal-to-noise ratio of the resulting spectrum. To eliminate these effects, as well as any stray light reflected by the sample, special weights were made to keep the area to be investigated stable without restricting the movement of the whole page. A microscopic stage (42 x 31 cm) was designed and constructed, which can easily be adapted to a standard Olympus microscope on which manuscripts as large as 30 x 21 x 8 cm can lie open under secure, stable conditions. The micrograin selected can be focused on the cross wires to  $\pm 1 \mu\text{m}$  by using the X-Y micrometers attached to the microscope stage<sup>14</sup>. Microphotographs were taken (at 100x magnification) using a camera attached to the Olympus microscope.

### Ms Pers 1 (UCL) 'Anatomy of the Body'

#### *About the manuscript*

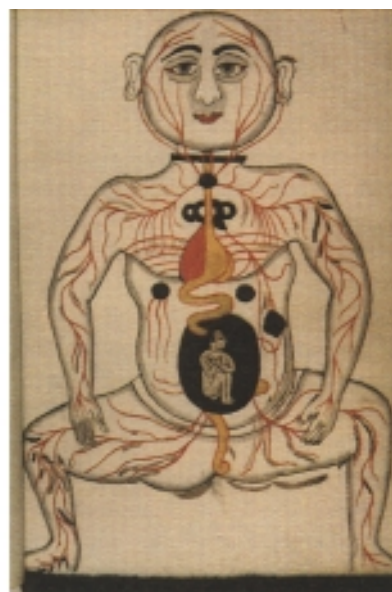
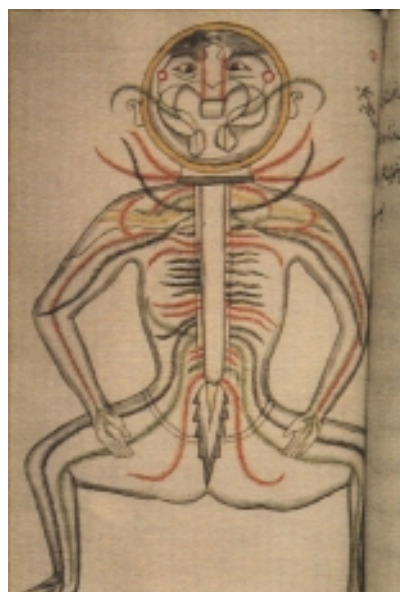
From the label pasted on the cover it is evident that this Ms was given to the UCL library by the Emeritus Professor of Anatomy and Physiology, William Sharpey, in 1874. The Ms is written in Persian calligraphic script on 54 fs of paper (recto and verso) of size 15.6 x 21.5 cm, in a blind (not drawn) frame of size 10.2 x 16.5 cm. The text consists of 12 rows per page. Two fly leaves after f 54 are made of the same paper as the Ms. A water mark is easily observed through the paper of these fly leaves on fs 50 - 52. It shows the coat of arms and the letters 'GIOR. MAGNANI' which pertain to a paper factory existing in 1798 in the Grand Duchy of Tuscany (cat. no. 1754 'supplementary water-marks outside of the Austro-Hungarian empire',

Eineder)<sup>15</sup>. Two more fly leaves of another type of paper, in front of f 1 and after f 56, have a water mark in which the inscription 'SHEL GROVE' can be seen. An *ex libris* written on the verso of the front fly leaves mentions that the Ms is 'Tushrih - ul - aza, or The Anatomy of the Body ("members")', and shows a dedication to Professor Sharpey from one of his students. The binding is of cardboard, covered with tanned thin red leather, without inscription apart from a series of yellow rectangles centered on the same point in the middle of the cover. There are six crudely drawn and simply colored diagrams of the human body on fs 20v, 30r, 33v, 42v, 46r and 54v.

Comment: The watermark proves that Ms Pers 1 'Tushrih - ul - aza' (UCL) is a 19<sup>th</sup> C copy of an older one. There are very close similarities between the body diagrams drawn in this Ms and those of two older versions (Ms 450 of the University Central Library, Tehran, and Ms 14709 of the Islamic Museum, Cairo, see plates 100 and 101, respectively, from Seyyed Hossein Nasr<sup>16</sup>) of 'Tashrih - i - Mansuri' or 'Mansur's Anatomy', written in the 9<sup>th</sup> C by Mansur ibn Muhammad ibn Faqih Ilyas, and later often copied, improved and widely used in the Islamic world during the 11-19<sup>th</sup> C<sup>16</sup>.

#### *The characteristic palette*

The text and drawings are in black ink (Photographs 1a, 1b); the grey underlay of the drawings is of a dilute black pigment. The human body diagrams are colored in black (ink) and brown (f 54v), red 1 (fs 20v, 30r, and 46r), red 2 (f 46r), yellow 1 (f 20v), and the nerves in f 30r, yellow 2



**Photograph 1.** a) (left) Ms Pers 1 (UCL) 'Anatomy of the Body', f 30r, the nervous system (schematic). The colors black, red 1, yellow 1, yellow 2, and pale green can be seen. b) (right) Ms Pers 1 (UCL) 'Anatomy of the Body', f 54v, the circulatory, digestive and reproductive systems (schematic). The fetus is 3-5 months old, in the upward position. The brown pigment is evident.

(digestive system f 54v), and pale green (nerves in f 30r) see Photographs 1c, 1d. No blue pigment was used in this Ms, and only restricted use was made of any green pigment (for some nerves on f 30r).

## Results and Discussion

The red 1 is vermilion<sup>17,18</sup> HgS CI 77766 PR 106; its Raman spectrum (Fig. 1a) has three main bands<sup>10,11</sup> at c. 247, 275, and 336  $\text{cm}^{-1}$ . The red 2 is red lead<sup>19</sup>  $\text{Pb}_3\text{O}_4$  CI 77578 PR 105, which has a spectrum shown in Fig. 1b. It has characteristic bands<sup>9,10,20</sup> at c. 48, 58, 116, and 147  $\text{cm}^{-1}$ . The brown pigment is a variety of ochre<sup>21,22</sup> or sienna<sup>23</sup> containing  $\text{FeO}(\text{OH}) \cdot n\text{H}_2\text{O}$  CI 77492 PBR 6 and 7. Its spectrum contains a band at 386  $\text{cm}^{-1}$  (Fig. 1c) which is characteristic of iron hydroxide, *cf.* the spectrum of a reference sample from Winsor and Newton. The yellow 1 is orpiment<sup>24</sup>  $\text{As}_2\text{S}_3$  CI 77086 PY 39. Its spectrum (Fig. 1d) shows characteristic bands<sup>10,19</sup> at 130, 148, 195, 286, 303, 348, and 376  $\text{cm}^{-1}$ .

It is remarkable that neither malachite nor azurite was used for the pale green pigment, which does not give a Raman spectrum. The green is probably a natural organic green such as sap green (*Rhamnus*)<sup>18,24,25</sup>, tempered with orpiment. Yellow 2 gave a very strong and specific fluorescence spectrum but could not be identified. Yellow 2 is possibly Indian yellow<sup>18,26</sup>, a Ca and Mg salt of euxanthic acid CI 75320 natural dye. The black pigment does not give a Raman spectrum. Some grains of red lead have been identified in the brown pigment, suggesting that the painter washed the brush carelessly.

## Conclusions

The paper of the Ms is imported from Tuscany, and is of low quality. The black ink was used to write as well as to color (Photograph 1b). Vermilion, ochres, and orpiment were cheap pigments in comparison to others such as indigo, saffron, ultramarine, etc<sup>27,28</sup>. Pigments such as azurite or malachite, which were extensively used in European Mss, were not used in this Ms. The diagrams of the body are very crudely drawn, suggesting that the Ms is a cheap copy of an older one made by a student for personal use.

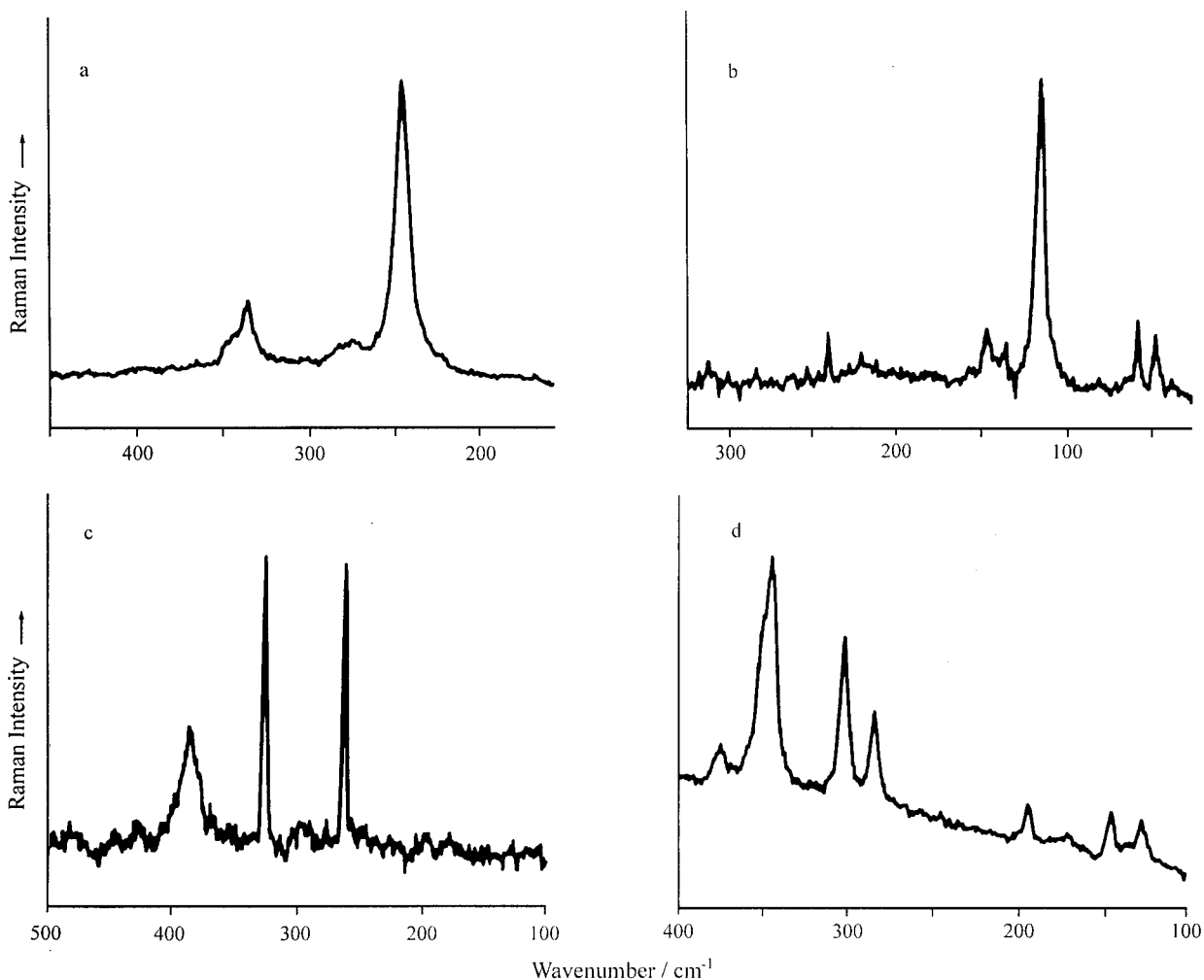
## Ms Persian 2 (UCL) 'Poetry in Praise'

### *About the manuscript*

This precious and very richly illuminated and decorated manuscript comes from the former collection of Arthur Strong (Oriental Library, 1905). The Ms consists of 177 fs of very thin, good quality paper, of size 11.1 x 18.3 cm, without a watermark. An area of 10.3 x 17.3 cm of each page is illuminated with burnished gold (fs 1v, 2r, 2v, 3r, see for example Photograph 2a) or stamped with a (symmetrical) repetitive characteristic pattern (a flower, two buds and two leaves, on a small stem surrounded by an elliptical frame of four leaves) (see the folios beyond 3v, for example Photograph 2b). The stamped illumination is limited by a 0.3 cm wide border. Another rectangular frame of 6.3 x 11.8 cm is limited by a 0.4 cm wide border and divided in two by a 0.6 cm band. Very finely painted garlands of small flowers, alternately red and blue, decorate this frame (see the details in Photograph 2b). Two columns of nine rows of Persian text are calligraphic and written in



**Photograph 1.** c) (left) A detail (100x magnification) of the nervous system shown in Photograph 1a at the crossing point of the yellow and red nerves on the right-hand side of the body. d) (right) A detail (100x magnification) from the circulatory and digestive systems shown in Photograph 1b, from near the upper thorax (esophagus).



**Figure 1.** a) The Raman spectrum of vermilion,  $\text{HgS}$ , used as red 1 in Photographs 1a and 1b. b) The Raman spectrum of red lead,  $\text{Pb}_3\text{O}_4$ , used as red 2 in Photograph 1b. c) The Raman spectrum of  $\text{FeO}(\text{OH}) \cdot n\text{H}_2\text{O}$ , a component of brown ochre evident in Photograph 1b. The lines at  $264$  and  $327 \text{ cm}^{-1}$  arise from plasma. d) The Raman spectrum of orpiment,  $\text{As}_2\text{S}_3$ , used as yellow 1 and to temper the green used in Photograph 1a.

black ink on a white background, which is itself bordered by a very fine black curve. The poem titles are written in blue ink on a golden background within a rectangle which has a thin border variously colored in the Ms, and is decorated with small crosses (Photograph 2b). The first two fs contain red and black script on paper and white script on gold. There is a flyleaf at both the front and the back. *The ex libris* are written in Persian on f 1r in black ink. On the label of the library is written 'Mir Samihud - al - din, F. Dehlvi (Delhi), Masnavi, (Poetry in Praise)'.

Floral miniatures arranged in various symmetries are used to compose the very rich decorations characteristic of Islamic art in which human or zoomorphic representations are forbidden. All fs are numbered in Persian at the bottom of the page (see the left corner of Photographs 2a, 2b) in such a way as to destroy a little of the symmetry of the page, (for the reason that a very correct and complete symmetry is attributed only to God not to

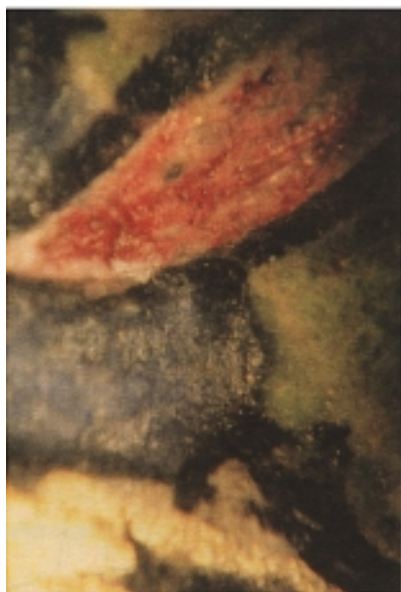
human beings), but without reducing its artistic impression. For the same reason, the smaller decorated frame which contains the text is not centered exactly in the middle of the page.

The binding is original, made from two 0.3 cm thick wooden boards (heavy hardwood), and covered with very thin tanned leather which has on the external faces of the front and back covers a red background and on the internal faces a green background. A rich, very fine floral decoration and illumination is tooled on both faces of the covers. This decoration is finally covered with a thick layer of varnish (lac), at present a little cracked.

Comment: The very rich illumination of the page is characteristic of Persian Mss of the 14-16<sup>th</sup> C<sup>29</sup>. The Ms is very well preserved, and similar in decoration to Ms Persian 35 in the Ryland Library, Manchester<sup>30</sup>. It can easily be seen that a simple page (Photograph 2a) in this Ms was made in successive operations, first by drawing the frame

and illuminating it with gold. The microscopic observations suggest that a roller or press was used to apply the gilded pattern as well as the main frame of the page onto the paper (Photograph 2b), and that later the gold leaves were burnished to illuminate the smaller internal frame (Photographs 2a and 2b). Second, the black script was

written in black ink. Third, the white background was completed around the text and border with a fine curved line, and then the other decorations followed. The details of the finest drawn lines of the floral miniatures can be observed only under a microscope at 100x magnification (see Photographs 2c and 2d).



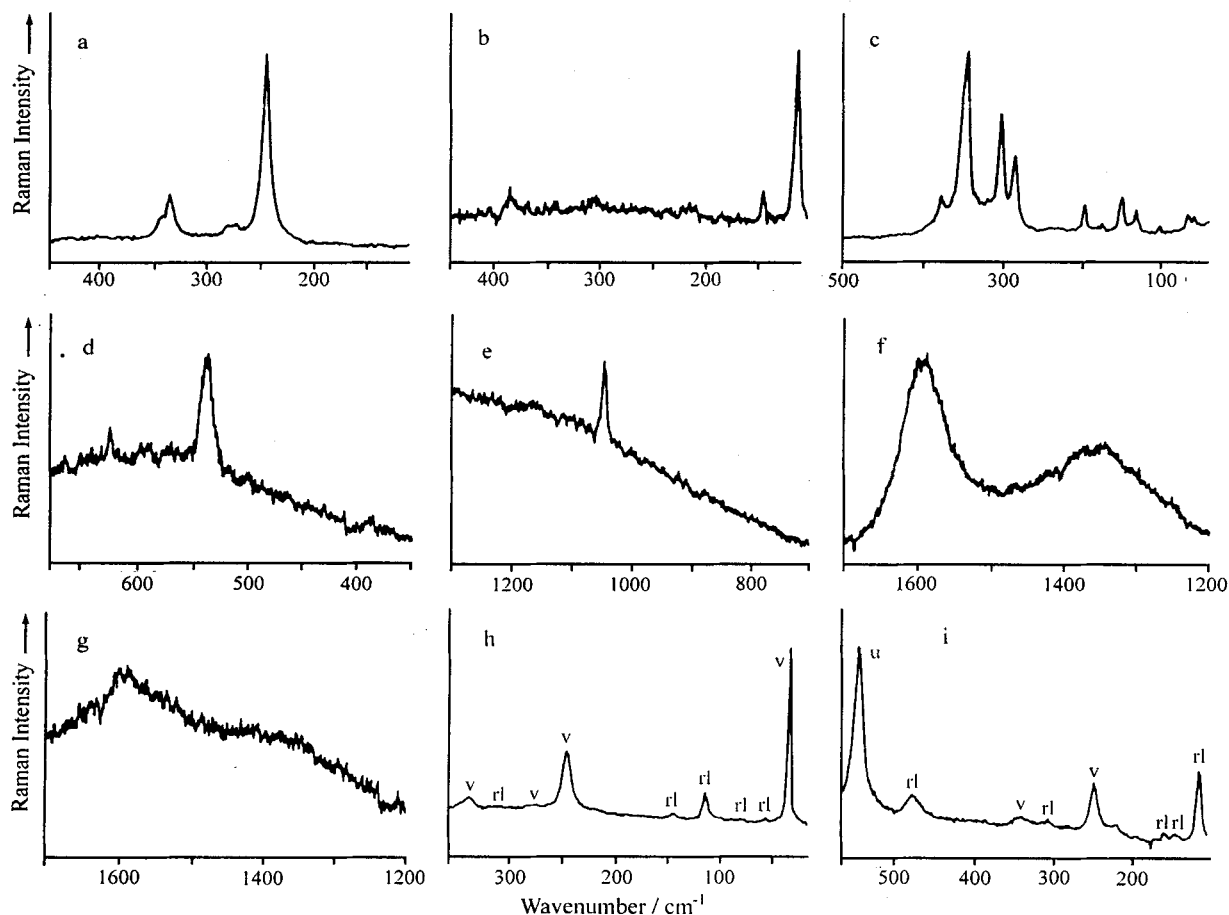
**Photograph 2.** a) (upper, left) Ms Pers 2 (UCL) 'Poetry in Praise' f2v. On a surface with burnished gold, white 1 (script), white 2 (the background of the black script), and a richly colored floral decoration on a blue background can be seen. This decoration has a pattern of variable symmetry. Black 1 (intense) and black 2 (matt) can be seen in the thin border of the blue arabesques with flowers and in the large rectangular frame of the page. b) (upper, right) Ms Pers 2 (UCL) 'Poetry in Praise' and a typical page (beyond f3v). It contains nine rows of black script on the white 2 background, and blue script of the title of the poem on burnished gold. The gilded floral symmetrical pattern was tooled (rolled, stamped) onto all the pages of this Ms. In the lower left corner of the tail, the number of the page (in Persian) with a characteristic golden border can be seen. This should be compared with that on the page in Photograph 2a. c) (bottom, left) A detail (100x magnification) in Photograph 2a. The mixture of red 1 and red 2, blue, green, and yellow can be seen. d) (bottom, right) A detail (100x magnification) of Photograph 2a. The red 1, red 2, brown, and black of the very small flowers were painted with great finesse, particularly in the drawing of the black lines of widths 0.02 - 0.1 mm.

### The characteristic palette

Black, blue, white (white 1), and red inks were used in this Ms. Another white pigment (white 2) was used as a background to increase the contrast of the black script. The background for the blue and white inks is burnished gold, and that for red ink is the white of the paper (flv, 2r). The decorations on fs 2v and 3r contain two types of black pigment different from the ink, the intense black (black 1) of the thin border line of the arabesque decoration, and the matt black (black 2) of the main decorated frame (see Photograph 2a). There are two types of red pigment, red 1 and red 2. The yellow, blue, green, and brown pigments (the latter rarely) can be observed in the detail of the floral decorations (Photographs 2c and 2d). Composite colors like purple, pink, deep red, deep green, light blue etc., seem to be made by tempering those listed.

### Results and Discussion

Figure 2a shows the spectrum of vermilion, which was used as a red ink and also in the floral decorations. Red lead was used either as a principal hue or tempered with vermilion and other pigments (Photographs 2c and 2d). It has the spectrum shown in Fig. 2b. The yellow pigment is orpiment, as shown by the spectrum in Fig. 2c. That shown in Fig. 2d is from the blue pigment and has a band at  $548\text{ cm}^{-1}$ , characteristic<sup>10,31</sup> of lazurite (ultramarine)<sup>32,33</sup>,  $\text{Na}_8[(\text{AlO}_2)_6(\text{SiO}_2)_6]\text{S}_n$ , CI 77007 PBI 29, which was extensively used in the decorations as well as for the blue script. The white 1 pigment is white lead<sup>18,34</sup>,  $x\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$  ( $x = 1.6\text{--}2.8$ ) CI 77597 PW 1, which gave the spectrum (Fig. 2e) with the main band<sup>35,36</sup> at  $1050\text{ cm}^{-1}$ . The other white pigment used as a background for the black script did not give a Raman spectrum, but only a very strong, characteristic fluores-



**Figure 2.** a) The Raman spectrum of vermilion used as red ink and as red 1 pigment. b) The Raman spectrum of red lead used as a red 2 pigment. c) The Raman spectrum of orpiment used as the yellow pigment and to temper the green one (see Photograph 2c). d) The Raman spectrum of ultramarine  $\text{Na}_8[(\text{AlO}_2)_6(\text{SiO}_2)_6]\text{S}_n$  used as a blue pigment in the decorations and as ink for the blue script. e) The Raman spectrum of white 1 used as white ink on gold at the center of Photograph 1a. f) The Raman spectrum of black ivory used to draw the fine line of the arabesque in Photograph 2a. g) The Raman spectrum of lamp black used in the decoration of the main frame in Photograph 2a. h) The Raman spectrum of a mixture of vermilion (v) and red lead (rl). i) The Raman spectrum of a mixture of red lead (rl), vermilion (v), and lazurite (u).

cence spectrum. The spectra given in Figs. 2f and 2g are characteristic of ivory black and lamp black, respectively<sup>24</sup>, and are similar to those obtained from reference samples (Winsor and Newton). Their main bands at 1346 and 1600  $\text{cm}^{-1}$  are named D (diamond) and G (graphite)<sup>37</sup>. The spectrum of black 2 has the same two bands, attenuated due to a larger degree of disorder of the carbon atoms in the structure<sup>37-39</sup>. The spectra of some samples of the red pigments show that they arise from mixtures, the component grain sizes of which must be  $< 1 \mu\text{m}$ , the diameter of the focussed laser beam, e.g. those shown in Fig. 2h (red lead and vermilion) and Fig. 2i (red lead, ultramarine and vermilion). An ambiguity may arise concerning the very strong band at c. 547  $\text{cm}^{-1}$  in this spectrum. It is a common band in the spectra of red lead,  $\text{Cr}_2\text{O}_3$  and ultramarine. However, further experiments showed that the band at c. 545  $\text{cm}^{-1}$  in the spectrum of red lead is only half as intense as the main band at c. 119  $\text{cm}^{-1}$ , and that  $\text{Cr}_2\text{O}_3$  is only a very weak Raman scatterer; thus the band at 547  $\text{cm}^{-1}$  must be the  $\nu_1$ ,  $\text{S}_3^-$  ion, band of lazurite (ultramarine)<sup>31</sup>. The green pigment seems to be natural organic<sup>18,24,25</sup>, and gave only a strong, fluorescence spectrum. The brown pigment did not give a Raman spectrum.

## Conclusions

Various mineral and natural organic pigments were used in Ms Pers 2 UCL 'Poetry in Praise'. It is remarkable that, as was the case for Ms Pers 1 UCL 'Anatomy of the Body', malachite and azurite were not used in the decorations, but rather a natural organic green (a pigment different from that used in Ms Pers 1) and, quite extensively, ultramarine blue. The white lead was used as an ink, as a main hue, and to temper other pigments so as to obtain various shades. The other white pigment, which did not give a Raman spectrum, was used only as a background for the black script. Intimate mixtures of pigments were obtained by finely grinding the grains to a diameter of  $< 1 \mu\text{m}$ , which is the size of the focused laser beam. In this way it is understandable that superimposed spectra were obtained (Figs. 2h and 2i). Expensive ultramarine was associated with gold<sup>40</sup> to increase the artistic value of the book. It is very probable that powerful magnifiers were used to draw, paint and illuminate the symmetric floral decorations (see Photographs 2c and 2d). All of these features, combined with the very good calligraphy and the richness of the binding, lead to the conclusion that Ms Pers 2 (UCL) 'Poetry in Praise' was produced by several very skilful artists at the order of a rich Islamic person.

## Acknowledgment

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