

CHARACTERIZATION OF MG AND FE CONTENTS IN NEPHRITE USING RAMAN SPECTROSCOPY

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Nephrites with similar hues usually have different ranges of saturation and tone, and color determination with the unaided eye can be problematic. The appearance of impurity ions such as Fe^{2+} influences the color of the material. Raman spectroscopy of 99 nephrite samples of different colors from northwestern China and Taiwan identified their main mineral as either tremolite or actinolite. When the $\text{Mg}^{2+}/(\text{Mg}^{2+} + \text{Fe}^{2+})$ ratio was 0.91 or higher, the mineral was tremolite; when the ratio was less than 0.91, the mineral was actinolite. The features in the M-OH stretching vibration region ($3700\text{--}3600\text{ cm}^{-1}$) are important fingerprints of nephrite's composition. Using the relative intensity ratio method, the authors calculated the Mg^{2+} and Fe^{2+} content and the ratio of $\text{Mg}^{2+}/(\text{Mg}^{2+} + \text{Fe}^{2+})$. In this study, the ratio was approximately 1 for white nephrite, 0.98–1 for light green nephrite, 0.90–0.98 for bluish green nephrite, and lower than 0.91 for dark green nephrite. The calculation of relative intensity ratios could provide a standard for nephrite color determination and even the critical evaluation of whiteness.

Nephrite, an amphibole-rich rock, comes in a variety of colors (figure 1). As noted by Zhang (2006), these hues are classified by the Chinese as *baiyu* (white), *qingbaiyu* (white with a slight bluish, greenish, or grayish tint), *qingyu* (bluish green or greenish blue), *biyu* (green to dark green), *huangyu* (yellow), and *tangyu* (brown). Distinguishing the transitions between these color varieties is an important consideration in establishing a nephrite grading standard. Traders once determined the color solely by visual observation, but even a relatively accurate

determination requires decades of experience. Major gemological laboratories still do not have a quantitative or semiquantitative boundary between colors. Since the value of fine white nephrite can be significantly higher than material in other hues, whiteness is the most important parameter in evaluating and classifying nephrite that ranges from white and gray to light green. The boundary between white and light grayish to bluish green nephrite is the hardest to identify, and establishing this difference is the focus of our study.

The amphibole group is comprised of double-chain silicates belonging to the monoclinic crystal system. The group's general chemical formula is $\text{A}_{0-1}\text{B}_2\text{C}_5[\text{T}_4\text{O}_{11}]_2(\text{OH}, \text{F}, \text{Cl})_2$. The components of the formula are described as A, B, C, T, and OH, with T corresponding to the tetrahedral sites in the unit cell. There are four kinds of sites occupied by cations (fig-

In Brief

- Color determination of nephrite jade can be difficult due to ranges of tone and saturation, even within similar hues.
- Since white nephrite tends to be highly valued, establishing a boundary between white and light-colored material is imperative.
- Raman spectroscopy reveals the relative ratio between Mg^{2+} and Fe^{2+} as it pertains to the whiteness of nephrite, providing a measurement for color determination.

ure 2). These sites are C, a composite of five sites made up of two M1, two M2, and one M3 sites; and B, which is two M4 per formula unit. M1 and M3 sites are usually occupied by the Mg^{2+} and Fe^{2+} ions, and M2 sites by a trivalent or tetravalent form of cations with a smaller radius, such as Fe^{3+} . M4 sites are occupied by Ca^{2+} , Na^+ , and K^+ , while A sites are occupied by OH^- and the remaining Na^+ and K^+ cations to balance the valence state. Cations and OH^- that occupy M1, M3, and A sites form an ionic bond. Amphiboles are classified into four subgroups de-

See end of article for About the Authors and Acknowledgments.

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Figure 1. The color varieties of nephrite. Photo courtesy of Beijing Boguan International Auction Co., Ltd.

pending on the occupancy of the B sites: the Mg-Fe-Mn subgroup, the calcic subgroup, the Na-Ca subgroup, and the alkali subgroup (Bragg and Claringbull, 1965; Wang et al., 1982; Leake et al., 1997). Tremolite and actinolite are in the calcic amphibole subgroup.

Nephrite is mainly composed of tremolite, $\text{Ca}_2\text{Mg}_5[\text{Si}_4\text{O}_{11}]_2(\text{OH})_2$, and a variable amount of actinolite, $\text{Ca}_2(\text{Mg, Fe})_5[\text{Si}_4\text{O}_{11}]_2(\text{OH})_2$. When the $\text{Mg}^{2+}/(\text{Mg}^{2+} + \text{Fe}^{2+})$ ratio is 0.91 or higher, the main mineral is tremolite; when the ratio is less than 0.91, it is actinolite (Bragg and Claringbull, 1965; Wang et al., 1982; Leake et al., 1997).

Many researchers exploring the role of Fe and Mg cations in the coordination of tremolite using different spectroscopic methods have pointed out that higher Fe^{2+} content generally produces a deeper green color in tremolite (Liu, 1988; Li and Wu, 1999; Guo and Han, 2002; Lu et al., 2007). On the basis of previous studies and our own Raman spectroscopic investigation of the relative intensity ratio of the spectral features related to M-OH bands, we propose a new method to calculate the relative content of Mg and Fe in the M1 and M3 sites. This method has the potential to define boundaries for nephrite color determination and may allow for evaluation of whiteness.

MATERIALS AND METHODS

Ninety-seven nephrite samples from China (Hetian in the Xinjiang Uygur Autonomous Region and Golmud in Qinghai Province) and two Taiwanese nephrites from Hualian were studied to form a nephrite jade grading standard. Of these, 15 samples showing a range of colors were selected for this study (table 1). One set of nephrite color standards was collected by the Xinjiang Uygur Autonomous

Figure 2. The crystal structure of tremolite. Adapted from Li (2008).

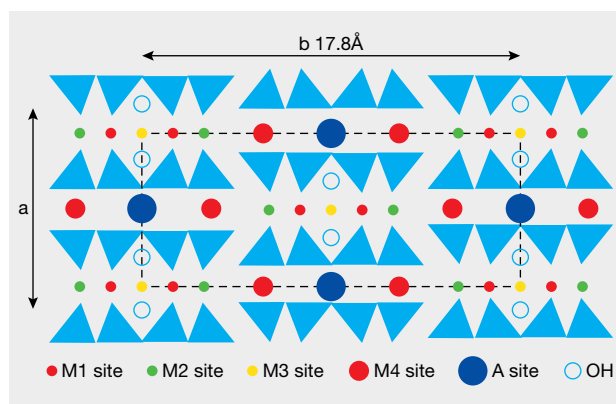


TABLE 1. Chinese nephrite samples examined for color determination.
















Sample number	Locality	Size (mm) L × W × H	Color	Chinese term	
H-01	Xinjiang	30.2 × 20.1 × 9.6	White	<i>Baiyu</i>	
H-02	Xinjiang	30.3 × 19.9 × 10.2	White	<i>Baiyu</i>	
H-03	Xinjiang	30.1 × 20.2 × 10.2	White	<i>Baiyu</i>	
H-04	Xinjiang	30.0 × 20.9 × 10.3	White with a slight greenish tint	<i>Qingbaiyu</i>	
H-05	Xinjiang	29.7 × 20.0 × 10.2	Bluish green	<i>Qingyu</i>	
H-06	Xinjiang	30.3 × 19.8 × 9.5	Dark green	<i>Biyu</i>	
H-07	Xinjiang	29.8 × 20.4 × 9.9	Dark green	<i>Biyu</i>	
H-08	Xinjiang	30.1 × 19.9 × 10.2	Dark green	<i>Biyu</i>	
H-09	Xinjiang	23.0 × 21.4 × 5.3	Dark green	<i>Biyu</i>	
Q-01	Qinghai	24.5 × 3.1	White	<i>Baiyu</i>	
Q-02	Qinghai	24.5 × 3.1	White with a slight grayish tint	<i>Qingbaiyu</i>	
Q-03	Qinghai	24.6 × 3.1	Bluish green	<i>Qingyu</i>	
Q-04	Qinghai	24.7 × 3.1	Bluish green	<i>Qingyu</i>	
TY-01	Taiwan	9.0 × 7.4 × 3.5	Brown-yellow	<i>Biyu</i>	
TY-02	Taiwan	8.6 × 7.1 × 3.4	Brown-green	<i>Biyu</i>	

TABLE 2. Main Raman bands of nephrite and their assignments in the 1200–100 cm⁻¹ region.

Band position (cm ⁻¹)	Assignment
1062	Si-O stretching vibration
1031	Si-O stretching vibration
932	Si-O stretching vibration
741	Symmetric Si-O-Si stretching vibration
675	Symmetric Si-O-Si stretching vibration
530	Si-O-Si and O-Si-O bending
516	Si-O-Si and O-Si-O bending
437	Si-O-Si and O-Si-O bending
418	Lattice vibrations
396	Lattice vibrations
372	Lattice vibrations
353	Lattice vibrations
252	Lattice vibrations
225	Lattice vibrations
179	Lattice vibrations
124	Lattice vibrations

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The nephrite samples were cut into wafers of different shapes. A Renishaw Raman 1000 spectrometer with a 514.5 nm laser was used at a power of 30–40 mW. To observe both the Si-O and M-OH stretching vibrations, Raman data were acquired with a spectral resolution of 4 cm⁻¹ and a scanning time of 20 seconds in the 1200–100 cm⁻¹ and 3700–3600 cm⁻¹ ranges, respectively. Microprobe analyses were performed on a JEOL JXA-8800 electron microprobe analyzer at the Institute of Mineral Resources, Chinese Academy of Geological Sciences in Beijing. Analysis conditions were 20 kV accelerating voltage, 100 nA beam current, and a 5 μm beam.

RESULTS AND DISCUSSION

Raman Spectra. There are two regions of interest in nephrite's Raman spectrum:

1. The 1200–100 cm⁻¹ region: Si-O vibrations are located in this range, where nephrite and other minor minerals can be identified. The main nephrite bands are shown in table 2 (Zou et al., 2002; Lu et al., 2007; Zhao and Gan, 2009). Our samples' Raman spectra essentially match that of tremolite, indicating that this is the main mineral component of these nephrites (figure 3).

The main Raman peaks of tremolite-dominated nephrite are at 1062, 1031, 932, 741, 675, 530, 516, 437, 418, 396, 372, 353, 252, 225, 179, and 124 cm⁻¹ (Zou et al., 2002; Wang et al., 2006).

With higher Fe content, the tremolite component gradually decreases while actinolite increases; at the same time, the Raman peaks shift accordingly (Lu et al., 2007). Some Raman peaks found in white and dark green nephrite (for instance, the peak near 741 cm⁻¹, which is split into double peaks at 748 and 738 cm⁻¹ in white nephrite and shifts to a single 743 cm⁻¹ peak in dark green nephrite) shift about 1–5 cm⁻¹, and their intensity diminishes or even disappears (again, see figure 3).

2. The 3700–3600 cm⁻¹ region: M-OH stretching vibrations are observed in this range. There are one to three bands, depending on the presence of Fe²⁺ in the M1 and M3 sites (Burns and Strens, 1966; Liu, 1988; Guo and Han, 2002; Lu et al., 2007). The number of peaks and their position also depend on the amount of Fe²⁺ occupying these two sites (table 3 and figure 4).

Raman spectra peak positions from M-OH stretching vibration in the nephrite samples are listed in table 4 and are summarized as follows:

Figure 3. Raman spectra of white nephrite (red trace) and dark green nephrite (black trace). In both types of nephrite, the 748 cm⁻¹ peak (see inset A) along with the 530, 516, and 480 cm⁻¹ peaks (inset B) shift about 1–5 cm⁻¹, and their intensity diminishes or even disappears due to the presence of Fe.

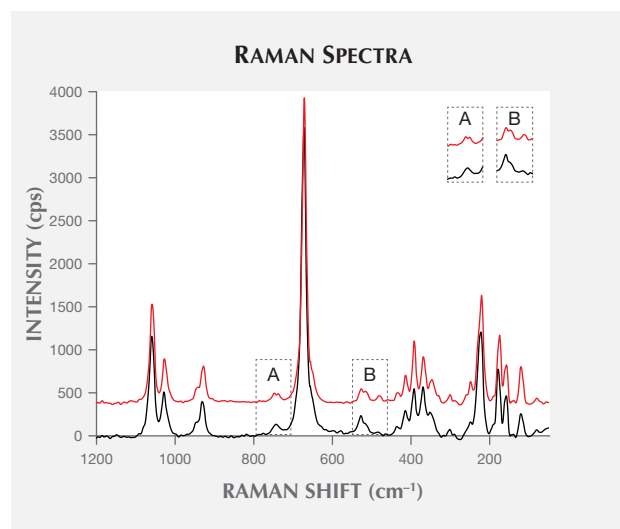


TABLE 3. M-OH stretching bands of nephrite and corresponding cations in the M1 and M3 sites.

Fundamental peak maxima (cm ⁻¹)	3675	3661	3645	
Cation	M1	Mg ²⁺	Mg ²⁺	Mg ²⁺
coordinating with OH ⁻	M1	Mg ²⁺	Mg ²⁺	Fe ²⁺
	M3	Mg ²⁺	Fe ²⁺	Fe ²⁺

Adapted from Burns and Strens (1966), Liu (1988), Guo and Han (2002), and Lu et al. (2007).

TABLE 4. Peaks from M-OH stretching vibration in nephrite.

Peak position (cm ⁻¹)	Presence
3675	All samples
3661	H-04, H-05, H-06, H-07, H-08, H-09, Q-02, Q-03, Q-04, TY-01, and TY-02
3645	H-06, H-07, H-08, H-09, Q-03, Q-04, TY-01, and TY-02

1. The white samples (H-01, H-02, H-03, and Q-01) showed a clear 3675 cm⁻¹ peak but no 3661 or 3645 cm⁻¹ peaks. This indicated very low Fe²⁺ content at the M1 and M3 positions, almost all of which are occupied by Mg²⁺ ions.
2. The slightly greenish white or grayish white nephrite samples (H-04 and Q-02) showed peaks at 3675 and 3661 cm⁻¹. The presence of the 3661 cm⁻¹ peak indicated a larger amount of Fe²⁺ ions, but the 3661 cm⁻¹ peak in these samples was very weak.
3. The bluish green samples (H-05, Q-03, and Q-04) ranged from light bluish green to deep bluish green. Samples from Xinjiang had a

lighter color than those from Qinghai; these lighter-colored samples showed Raman peaks at 3675 and 3661 cm⁻¹ but for the most part no 3645 cm⁻¹ peak. Both green samples from Qinghai exhibited a 3645 cm⁻¹ peak. The 3661 and 3645 cm⁻¹ peaks were more intense in the deep green nephrite, indicating that the green color varied with Fe²⁺ content (Lu et al., 2007).

4. The dark green samples showed the 3675, 3661, and 3645 cm⁻¹ peaks. This indicated a high Fe²⁺ content, which caused the dark green color.
5. Peaks corresponding to the Fe³⁺ ion of M-OH stretching bands, such as those at 3631, 3651, and 3604 cm⁻¹ (Guo and Han, 2002), did not ap-

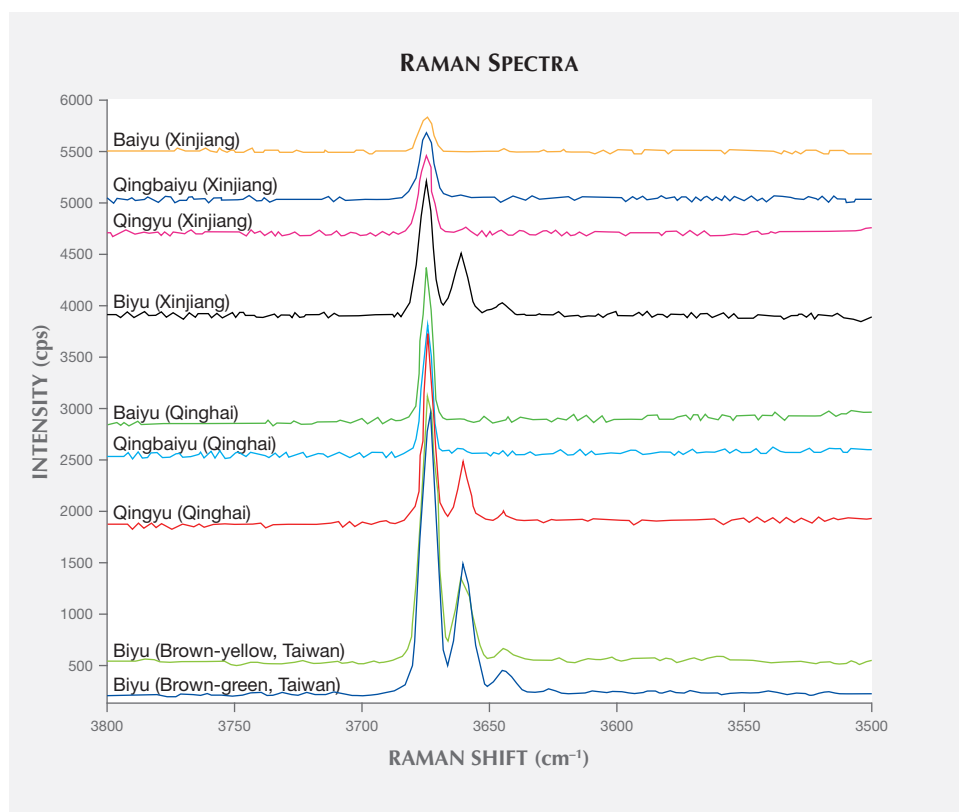


Figure 4. The M-OH stretching bands appear in the 3700–3600 cm⁻¹ region in different nephrite colors.

pear in any of the samples. These samples probably contained little or no Fe³⁺, and Raman spectroscopy could not detect its presence.

The Relative Intensity Ratio Method. We used the relative intensity ratio of the 3675, 3661, and 3645 cm⁻¹ peaks to calculate the relative content of Mg²⁺ and Fe²⁺ and the ratio of Mg²⁺/(Mg²⁺ + Fe²⁺) in the M1 and M3 sites, and to classify the color of nephrite according to the Mg²⁺/(Mg²⁺ + Fe²⁺) ratio.

Raman spectroscopy is a powerful tool for quantitative analysis (e.g., Pelletier, 2003). In a study of titanomagnetites, Zinin et al. (2011) used the relative intensity of the Raman peaks to calculate the Ti concentration. In our study, the intensity of Raman spectra was related to sample composition. Based on the intensity of the spectral peaks, we could calculate the relative content of the corresponding cation. On the basis of previous studies, we used Raman spectroscopy to study the relative intensity ratio of the spectral peak in the M-OH bands and devised a new method to calculate the relative content of Mg and Fe in the M1 and M3 sites (figure 5). The color of each nephrite sample was differentiated according to this ratio.

To calculate the relative Fe²⁺ and Mg²⁺ occupying the M1 and M3 sites, we set the relative intensity of the 3675 cm⁻¹ peak at 1 ($I_1 = 1$) and used this as a ref-

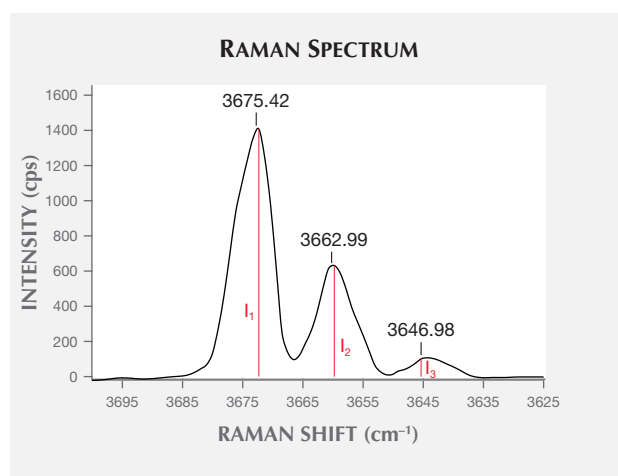


Figure 5. The relative intensities of the three main peaks in the M-OH stretching region are represented by the peak heights as I_1 , I_2 , and I_3 after baseline correction.

erence for calculation. The relative intensity of 3661 to 3675 cm⁻¹ is I_2 ($I_2/I_1 = I_2/1 = I_2$), while the relative intensity of 3645 to 3675 cm⁻¹ is I_3 ($I_3/I_1 = I_3/1 = I_3$). Based on the relative amount of Mg²⁺ and Fe²⁺ cations assigned to each peak position (again, see table 3), the relative amount of Mg²⁺ occupying M1 and/or M3 sites in a certain sample is calculated as $I_1 + 2I_2/3 + I_3/3$, while the relative amount of Fe²⁺ is calculated

TABLE 5. Calculated relative amounts of Mg²⁺ and Fe²⁺ in the M1 and M3 sites and the Mg²⁺/(Mg²⁺ + Fe²⁺) ratio.

Sample no.	Nephrite type	Mg ²⁺ ($I_1 + 2I_2/3 + I_3/3$)	Fe ²⁺ ($I_2/3 + 2I_3/3$)	Mg ²⁺ /(Mg ²⁺ + Fe ²⁺)
H-01	White (Baiyu)	1	0	1
H-02	White (Baiyu)	1	0	1
H-03	White (Baiyu)	1	0	1
H-04	White with a slight greenish tint (Qingbaiyu)	1	0	1
H-05	Bluish green (Qingyu)	1.05	0.02	0.98
H-06	Dark green (Biyu)	1.23	0.13	0.90
H-07	Dark green (Biyu)	1.19	0.11	0.91
H-08	Dark green (Biyu)	1.26	0.17	0.88
H-09	Dark green (Biyu)	1.22	0.15	0.89
Q-01	White (Baiyu)	1	0	1
Q-02	White with a slight grayish tint (Qingbaiyu)	1.02	0.01	0.99
Q-03	Bluish green (Qingyu)	1.22	0.14	0.90
Q-04	Bluish green (Qingyu)	1.21	0.12	0.91
TY-01	Brown-yellow (Biyu)	1.21	0.14	0.89
TY-02	Brown-green (Biyu)	1.31	0.19	0.87

TABLE 6. Chemical composition of nephrite samples by electron microprobe analysis (wt.%).

No.	Na ₂ O	MgO	SiO ₂	Al ₂ O ₃	K ₂ O	CaO	P ₂ O ₅	SO ₃	FeO	MnO	ZnO	Cr ₂ O ₃	NiO	TiO ₂	Total
H-01	0.132	24.940	57.244	0.067	0.138	13.316	0.027	bdl	0.048	0.021	0.040	bdl	0.012	0.019	96.004
H-02	0.128	25.371	56.594	0.188	0.078	13.201	0.024	0.004	0.062	0.028	0.074	0.009	bdl	0.011	95.772
H-03	0.187	24.034	57.683	0.788	0.186	12.785	0.010	bdl	0.275	bdl	0.136	bdl	0.051	0.017	96.152
H-04	0.159	24.271	57.794	0.472	0.102	12.480	0.044	bdl	0.474	0.076	bdl	0.007	0.026	0.002	95.907
H-05	0.337	24.781	57.593	0.639	0.223	12.094	0.031	0.011	0.636	0.090	0.051	0.041	0.074	bdl	96.601
H-06	0.178	23.028	55.798	0.276	0.094	13.131	bdl	bdl	4.454	0.211	0.05	bdl	0.253	bdl	97.473
H-07	0.147	22.809	55.921	0.381	0.019	13.065	0.021	bdl	3.435	0.172	bdl	0.012	0.126	0.032	96.140
H-08	0.239	22.446	56.115	0.534	0.140	13.108	0.033	0.002	4.154	0.117	bdl	bdl	bdl	0.143	97.031
H-09	1.550	22.740	56.790	0.220	0.460	11.380	bdl	bdl	4.360	0.050	bdl	bdl	bdl	bdl	97.550
Q-01	0.085	23.844	58.576	0.124	0.068	13.826	0.044	bdl	0.074	0.035	0.198	bdl	0.004	0.017	96.895
Q-02	0.074	23.692	58.546	0.211	0.043	13.844	0.007	bdl	0.386	0.060	bdl	bdl	bdl	0.044	96.907
Q-03	0.081	23.233	56.235	0.565	0.021	12.334	0.013	bdl	3.894	0.127	bdl	bdl	bdl	bdl	96.503
Q-04	0.113	23.173	56.298	0.526	0.021	12.920	bdl	bdl	3.725	0.153	bdl	0.024	0.069	bdl	97.022
TY-01	0.080	19.180	59.900	bdl	bdl	12.870	0.010	bdl	3.880	0.170	bdl	0.010	0.030	0.020	96.240
TY-02	0.030	21.300	57.270	0.170	0.010	12.590	0.020	bdl	4.900	0.320	bdl	0.030	0.110	bdl	96.750

bdl: below detection limit

as $I_2/3 + 2I_3/3$. We calculated the relative amounts of Mg^{2+} and Fe^{2+} in all samples and the $Mg^{2+}/(Mg^{2+} + Fe^{2+})$ ratio. The results are summarized in table 5.

According to table 5, the value of $Mg^{2+}/(Mg^{2+} + Fe^{2+})$ varies in samples by color. White nephrite has a value of approximately 1, light green nephrite 0.98–1, bluish green nephrite 0.90–0.98, and dark green

nephrite below 0.91. The lower the value of the $Mg^{2+}/(Mg^{2+} + Fe^{2+})$ ratio, the deeper the green color.

Comparison with Electronic Microprobe Data. To confirm the reliability of the relative intensity ratio method, we analyzed the chemical component of these samples using electron microprobe. The results

TABLE 7. Cation numbers of Mg^{2+} and Fe according to electron microprobe (wt.%) and the $Mg^{2+}/(Mg^{2+} + Fe)$ ratio.

Sample no.	Sample color	Mg^{2+} content	Fe content	$Mg^{2+}/(Mg^{2+} + Fe)$
H-01	White (Baiyu)	0.6186	0.000668	0.999
H-02	White (Baiyu)	0.6292	0.000863	0.999
H-03	White (Baiyu)	0.5961	0.00383	0.994
H-04	White with a slight greenish tint (Qingbaiyu)	0.6020	0.00660	0.989
H-05	Bluish green (Qingyu)	0.6146	0.00885	0.986
H-06	Dark green (Biyu)	0.5711	0.0620	0.902
H-07	Dark green (Biyu)	0.5657	0.0492	0.922
H-08	Dark green (Biyu)	0.5567	0.0578	0.907
H-09	Dark green (Biyu)	0.5640	0.0607	0.899
Q-01	White (Baiyu)	0.5914	0.00103	0.998
Q-02	White with a slight grayish tint (Qingbaiyu)	0.5876	0.00537	0.991
Q-03	Bluish green (Qingyu)	0.5762	0.0542	0.914
Q-04	Bluish green (Qingyu)	0.5747	0.0518	0.917
TY-01	Brown-yellow (Biyu)	0.4757	0.0540	0.899
TY-02	Brown-green (Biyu)	0.5283	0.0682	0.887

are shown in table 6. According to the data, the Fe and Mg^{2+} content and the $Mg^{2+}/(Mg^{2+} + Fe)$ ratio can be calculated, as shown in table 7.

Comparing the $Mg^{2+}/(Mg^{2+} + Fe^{2+})$ ratio obtained using the Raman relative intensity method with electron microprobe results shows good agreement between the two methods, but there is some variance. The variance may be caused by several factors, starting with the different nature of the two methods. Second, the Fe content measured by electron microprobe includes both Fe^{3+} and Fe^{2+} , while the spectral features reflect only Fe^{2+} . In addition, the analyzing spot cannot be exactly the same, because the two instruments do not use the same magnification. Taking this into consideration, the difference of the ratio in these samples is less than 3%, an acceptable variance.

CONCLUSIONS

Raman spectroscopy can help distinguish the color varieties of nephrite (again, see figure 4 and table 5) using the relative ratio of Mg^{2+} to Fe^{2+} concentration. The Fe^{2+} concentration is lowest in white nephrite and highest in dark green nephrite.

Since white nephrite (figure 6) tends to be highly valued, determining a boundary between white and light-colored nephrite is of importance to the trade. According to three relative peak intensity ratios in the M-OH stretching vibration bands, we can classify the color of nephrite to a certain degree. When the Raman spectrum only exhibits a 3675 cm^{-1} peak, the nephrite contains little Fe^{2+} . If the peak is more intense, the nephrite is whiter. The presence of a 3661 cm^{-1} peak is associated with a green tone; it is this peak that separates white nephrite from slightly greenish white, bluish green, and dark green material. The $Mg^{2+}/(Mg^{2+} + Fe^{2+})$ ratio indicates the color of nephrite. The lower the ratio, the deeper the green color. When the relative intensity of the 3661 cm^{-1} peak is stronger, the nephrite has a darker color. The 3645 cm^{-1} peak only



Figure 6. This $30 \times 30 \times 13\text{ cm}$ carving of high-quality Chinese nephrite from Hetian is shaped like a peach, symbolizing longevity. The design pattern comes from the Chinese fairy tale “Magu Xianshou.” Courtesy of Zhiguang You.

appears in dark green and bluish green nephrite. Thus, the method of relative peak intensity ratio of 3675 , 3661 , and 3645 cm^{-1} and the value of the relative intensity ratio are useful in evaluating nephrite color.

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