THE IDENTIFICATION OF TURQUOISE BY INFRARED SPECTROSCOPY AND X-RAY POWDER DIFFRACTION

By Th. Lind, K. Schmetzer, and H. Bank

A combination of infrared spectroscopy and X-ray powder diffraction methods is suggested for the identification of natural, treated, and synthetic turquoise as well as imitation turquoise. Both techniques require powdering only a very small quantity of the specimen (approximately 3 mg), which means minimal damage to the piece. New experimental results on treated turquoise and imitation turquoise are given.

Turquoise has been subjected to various methods of treatment in order to improve its value as a gem material, for example, enhance color or reduce porosity. In addition, a number of turquoise imitations are found on the gemstone market. Although many of these are called turquoise (Galia, 1977), at present, the so-called Gilson synthetic turquoise, which contains crystalline turquoise material as a component, is the only true synthetic available. The separation of natural, untreated turquoise from its treated counterpart, and the unequivocal identification of imitation or synthetic turquoise, is difficult with the routine gemological methods generally used. However, by applying techniques that are commonly used in mineralogy, e.g., X-ray powder diffraction methods and infrared spectroscopy, the gemologist can obtain the data necessary to sufficiently characterize the material (Banerjee, 1972; Arnould and Poirot, 1975; Williams and Nassau, 1976–1977; Schmetzer and Bank, 1980, 1981).

MATERIALS AND METHODS

The recognition of treated turquoise by infrared spectroscopy was comprehensively described by Banerjee (1972). Since that time, the practice of treating natural turquoise, especially the so-called stabilization techniques, has increased dramatically. Consequently, more and more samples have been submitted for investigation to determine whether or not the material is, indeed, natural turquoise, and, if so, whether it has been treated in any manner. In an attempt to solve the questions posed by the gemstone industry, we initiated a systematic study of the turquoise and turquoise-like material available on the market in order both to develop criteria to characterize these products and to augment the information available in the literature. A summary of the various methods of treating natural turquoise that are currently used is given in table 1. Two of the most common methods, paraffin treatment and plastic impregnation, were included in this study.

In addition to the 30 specimens of unknown composition submitted by the industry for characterization, we investigated 15 samples of natural turquoise from the United States (Arizona, Nevada), Mexico, Iran, and China; 10 of plastic- and paraffin-impregnated turquoise; 4 of Gilson "synthetic turquoise"; and 5 of "reconstructed turquoise." Figure 1 illustrates some of the different types of stones examined for this study.

X-ray powder diffraction and infrared spectroscopy were used. For the X-ray investigations, powder photographs were prepared using the Debye-Scherrer method. The infrared spectra were recorded on a Perkin Elmer 180 Infrared Spectrometer using the KBr pressed-pellet technique. Both techniques mentioned require the powdering of a very small quantity of the specimen. Normally, Debye-Scherrer photographs can be taken with less than 1 mg of powdered material; for the preparation of a KBr pellet for infrared spectroscopy, 2 mg of the sample were used.

ABOUT THE AUTHORS

Mr. Lind and Dr. Schmetzer are research associates, and Dr. Bank is an honorary professor, at the Mineralogisch-Petrographisches Institut der Universität Heidelberg, D-6900 Heidelberg, Federal Republic of Germany.

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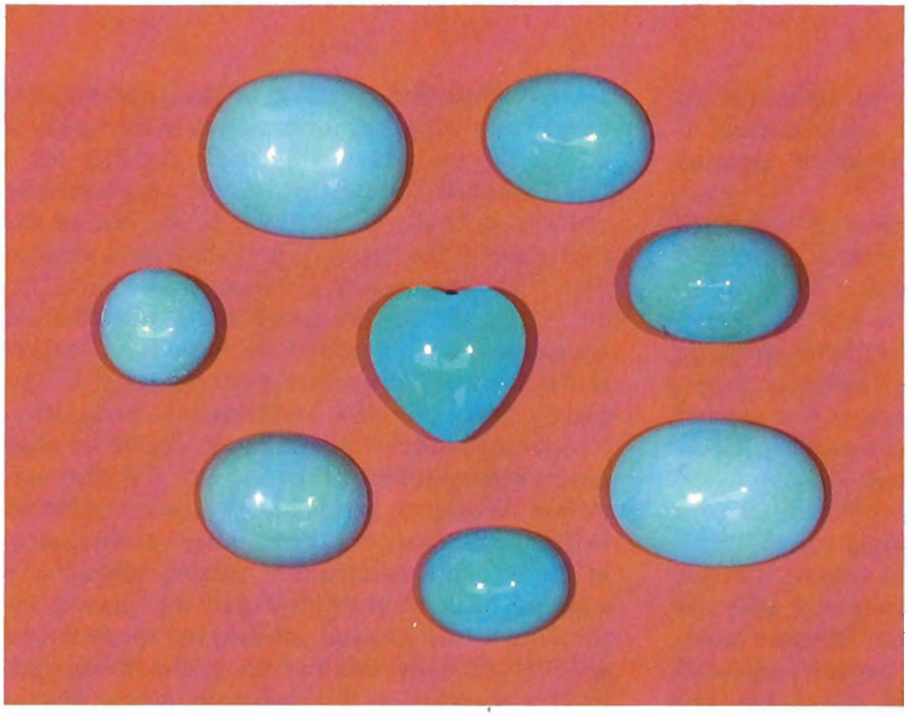

Figure 1. Samples of the different types of turquoise—natural, treated, synthetic, and imitation—studied by the authors. The center, heart-shaped cabochon is a Gilson synthetic, 2.10 ct. Clockwise, starting with the small stone to the left of the center specimen, the others are: paraffin impregnated, from Iran (1.38 ct); Gilson synthetic (4.91 ct); plastic impregnated, from Arizona (2.03 ct); natural, from Iran (2.79 ct); natural, from Arizona (4.50 ct); paraffin impregnated, from Arizona (1.79 ct); plastic-impregnated gibbsite (2.68 ct). Photo by Mike Havstad.

RESULTS

The results of the systematic investigations are summarized in table 2 and discussed in detail below.

Treated Turquoise. When plastic impregnation was first used, in the late 1960s, the so-called stabilized turquoises that resulted (which have nothing to do with the reconstructed turquoises described later in this article) were considered to be of relatively poor quality. At that time, primarily turquoise too porous for cutting was plastic-impregnated to improve the hardness of the specimen (see Banerjee, 1972; Galia, 1977). Currently, good-quality turquoise is also treated by plastic impregnation in order to improve the durability of the material, since natural turquoise is very sensitive to chemicals and has been known

TABLE 1. Treatment procedures used on natural turquoise.[a]

Procedure	Treatment substance	Color change	Other purposes of treatment	Penetration	Advantages/ disadvantages
Dyeing, varnishing	Colored organic or inorganic compounds, mixed with epoxy or other resin	Light colors become darker		Surface	
Paraffin treatment	Paraffin of different melting points	Light colors become darker	Impregnation	1–2 mm	Very uniform colors
Stabilization treatment (hardening by plastic impregnation)	Plastics (colorless or blue-dyed) with a polyester or polyacryl base	Light colors become darker	Impregnation, restoration of natural colors, improvement of durability for material of all qualities	>4 mm	Colors vary; enables cutting of weathered and porous material (chalk)
Stabilization treatment (hardening by use of inorganic mineral salts)	Inorganic mineral salts, e.g., colloidal silica		Same as for stabilization treatment by plastic		

[a] *For further details, see Galia (1977) and Gübelin (1981).*

to suffer damage from simple perspiration. At present, plastic impregnation (stabilization) is thought to be the best method of treating turquoise.

The X-ray powder diffraction of stabilized turquoise shows additional diffraction lines which do not belong to turquoise. These additional lines are also observed in the diffraction pattern of some specimens of Gilson synthetic turquoise. The d-values of these lines are identical to those of the strongest lines of the mineral berlinite, the chemical formula of which is $AlPO_4$ (ASTM 10-423). When we took several X-ray powder diffraction photographs of material from different areas of a single sample of stabilized turquoise, we observed that the intensity ratios of the berlinite lines varied compared with the intensities of the turquoise lines. In some cases, areas with great percentages of berlinite adjoined areas in which no berlinite was observed by the X-ray diffraction method.

The formation of an $AlPO_4$ phase with cristobalite structure after an exothermic reaction produced by heating turquoise to 840°C was described by Manly (1950). The substance investigated also showed some relicts of an earlier berlinite structure. Banerjee (1972) described the formation of an amorphous phase in turquoise after heating it to 400°C. With further heating (to between 740° and 775°C), an exothermic reaction identical to that described by Manly (1950), in which the $AlPO_4$ phase with cristobalite structure is formed, was observed. To clarify whether the $AlPO_4$ phase causing the additional X-ray lines found in the diffraction pattern of plastic-impregnated turquoise might be formed by the stabilization procedure, we conducted heating experiments on natural, untreated turquoise (180°C for 24 hours and 250°C for 24 hours). The X-ray powder photographs of every area investigated in the treated samples showed the strongest diffraction lines of berlinite in addition to the turquoise lines. It appears from these experiments that the $AlPO_4$ phase with berlinite structure forms at lower temperatures than those previously described in the literature; that is, berlinite can also be formed in the stabilization treatment. The fact that lower temperatures and shorter heating periods are usually used in the plastic-impregnation procedure explains why berlinite forms in some areas of the stabilized turquoise and not in others.

In the infrared spectrum of the six plastic-impregnated turquoises investigated in this study, a strong infrared absorption band at 1725 cm^{-1} was observed in addition to the characteristic absorption bands of turquoise in the area of the vibrations of the hydroxyl and phosphate groups. The absorption band between 1450 and 1500 cm^{-1}, described by Banerjee (1972) in stabilized turquoise, was not found during our investigations. The infrared spectrum of one sample of the plastic used for the stabilization procedure, which was made available to us, showed a very strong absorption band at 1725 cm^{-1}. Additional strong absorption bands of the plastic are found in the spectral area of the turquoise bands; that is, in the infrared spectrum of treated turquoise a superposition of turquoise and the plastic absorption bands is found. Only in the spectral area at 1725 cm^{-1}, in which no turquoise absorption band is observed, is the absorption of the plastic distinctly separated from the absorption of the turquoise. The absorption spectrum of the plastic used for the impregnation procedure is not identical to the spectra reported by Banerjee (1972). The strongest absorption band in our plastic was found at 1725 cm^{-1}, but no absorption band was observed between 1450 and 1500 cm^{-1}. Our industry sources have advised us that in the more than 10 years since Banerjee's article was published, new types of plastic have been adopted for the stabilization of turquoise.*

In the X-ray powder diffraction pattern of turquoise that has been paraffin impregnated (see table 1), the additional lines due to berlinite found in plastic-impregnated samples were also observed incidentally. Additional absorption bands in the infrared spectrum were not found when the KBr pressed-pellet method was used. This method of treatment is not limited to high-quality turquoise specimens as described by Galia (1977). Unfortunately, the use of this procedure is not always identified when the material is sold.

Synthetic Turquoise. At present, Gilson's synthetic turquoise is the only synthetic product found on the market in which the powder pattern

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**The kind of plastic investigated in this study, which is commonly used for the stabilization of turquoise, is known to the authors. We respect, however, our source's request that we keep this information confidential.*

of crystalline turquoise is observed by X-ray investigations (Williams and Nassau, 1976–1977; Schmetzer and Bank, 1980, 1981). In comparison to the turquoise pattern, however, in all of the samples of Gilson's synthetic product that we investigated, we observed additional X-ray diffraction lines caused by one or more additional crystalline phases. In two of the samples, the diffraction lines of berlinite were also found. When the specimens were examined using spectroscopy, we observed an absorption band at 1725 cm^{-1} in two of them as well, in addition to the absorption bands characteristic for turquoise. This band indicates that the synthetic material has also been treated.

Reconstructed and Imitation Turquoise. In addition to systematic experiments with natural, treated, and synthetic turquoise, we also investigated imitation turquoises. We used samples submitted to us by the trade to determine the composition of the specimens (compare Banerjee, 1972; Williams and Nassau, 1976–1977; Galia, 1977; Gübelin, 1981).

Dyed magnesite has been known for some years to be used as a turquoise substitute. Dyed calcite and dolomite were also observed recently. All three were found among the imitations examined (interestingly, dyed howlite, another common imitation, was not present among the samples studied). X-ray diffraction investigations revealed that most of the specimens called "reconstructed turquoise" in the trade were free of any turquoise component. γ-Al(OH)$_3$ (as a mineral, gibbsite) was the only crystalline phase found in these samples. In the infrared spectrum, the absorption at 1725 cm^{-1}, known already from the plastic component of stabilized turquoise, was observed. According to Galia (1977), true reconstructed turquoise is produced from finely powdered and cleaned turquoise and has crystalline turquoise as the main component. The "reconstructed turquoise" investigated in our laboratory, however, contained no turquoise; therefore, "reconstructed turquoise" is thought to be a misnomer for gibbsite that has been dyed and plastic-impregnated.

CONCLUSION

This investigation of natural and treated turquoise, of Gilson synthetic turquoise, and of various imitation turquoises suggests that most "turquoise" products found on the market can be identified by a combination of X-ray powder diffraction and infrared spectroscopy. Only paraffin-impregnated turquoise could not be positively identified by the two methods in all instances. Both methods require only very small amounts of powdered substance, which normally can be obtained from cut specimens without causing undue damage. We believe that, for the purpose of gemological nomenclature, the mineral name *turquoise* should be restricted to natural and synthetic turquoise only. In samples without a component of crystalline turquoise, the use of the name *turquoise* without the supplement "imitation" or "simulant" is misleading. In our opinion, the fact that a specimen of natural turquoise has been treated, regardless of the method used, should be disclosed in the trade.

TABLE 2. Results of X-ray powder diffraction and infrared spectroscopy tests on natural (untreated), treated, synthetic, and imitation turquoise.

Sample	X-ray powder diffraction pattern	Infrared spectrum
Turquoise, untreated	Turquoise	Turquoise
Turquoise, plastic impregnated	Turquoise or turquoise + berlinite	Turquoise + absorption band at 1725 cm^{-1}
Turquoise, paraffin impregnated	Turquoise or turquoise + berlinite	Turquoise
Gilson "synthetic turquoise"	Turquoise + several diffraction lines of an unknown phase or turquoise + several diffraction lines of an unknown phase + berlinite	Turquoise or turquoise + absorption band at 1725 cm^{-1}
Some "reconstructed turquoise" specimens from the trade	Gibbsite	Gibbsite + absorption band at 1725 cm^{-1}
Different imitation turquoises[a] of the trade	Magnesite, calcite, or dolomite	Not investigated

[a] *The X-ray powder diffraction patterns of other imitation turquoises were published by Williams and Nassau (1976–1977).*

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