

## Cathodoluminescence microscopy and spectroscopy of plagioclases from lunar soil

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### ABSTRACT

Cathodoluminescence (CL) microscopy and spectroscopy of single plagioclase grains from lunar soil show that plagioclases from Luna 20 (highland) have more or less homogeneous CL with both blue or green colors, whereas plagioclase grains sampled by Luna 24 (mare) luminesce dominantly green with partially distinct oscillatory zoning. The three main emission bands in the blue (~450 nm), green (~560 nm), and red-IR (~690 nm), mimic the most common emission bands in terrestrial feldspars. Mn<sup>2+</sup> is the most important activator element in lunar plagioclases. Variations in the amount of structurally incorporated Mn<sup>2+</sup> cause variations in the intensity of the green emission band at 560 nm, in some cases resulting in zoning of the CL intensity within single crystals. Calculations by a combination of quantitative spectral analysis of CL emission and PIXE measurements yield Mn concentrations of 7–47 ppm. The intense intrinsic emission band at 450 nm (probably an Al-O-Al center), which was especially prominent in Luna 20 plagioclases, causes their blue CL color. The occurrence of a CL emission band at ~690 nm in plagioclases from Luna 24 samples confirms that Fe<sup>3+</sup>-activated CL is common in these grains. The results indicate that at least some of the Fe in Luna 24 plagioclases is Fe<sup>3+</sup>, whereas all Luna 20 plagioclases have Fe<sup>3+</sup>-near the CL detection limit of about 0.1 ppm.

### INTRODUCTION

Plagioclase is a principal mineral in lunar rocks and soil. It is generally assumed that Fe, the most abundant transition element in calcic plagioclases of lunar rocks and soil (regolith), occurs predominantly as Fe<sup>2+</sup>, because Mössbauer measurements, optical studies, and wet chemical analysis did not detect Fe<sup>3+</sup> (e.g., Herzenberg and Riley 1970; Forester 1973; Burns et al. 1973; Bell and Mao 1973). However, electron spin resonance (ESR) and luminescence excitation spectroscopy studies have indicated the presence of Fe<sup>3+</sup> along with Mn<sup>2+</sup> (Weeks 1973; Niebuhr et al. 1973; Telfer and Walker 1975). To further constrain concentrations of Fe<sup>3+</sup> on the moon, single plagioclase grains from Luna 20 and Luna 24 samples, which represent material of predominantly highland origin and material mostly consisting of pure mare soil, respectively, were characterized by spatial resolved CL spectroscopy and spectral analysis of CL emission. In addition, attempts were made to quantify the concentration of the most important activator for CL (Mn<sup>2+</sup>) in these lunar plagioclases.

### MATERIALS AND METHODS

#### Sample material

The landing site of Luna 20 is located in the highland region between Mare Fecunditatis and Mare Crisium (3°32'N and 56°33'E). The Luna 20 soil consists of loose-grained, light grey regolith with about 20% of the grain size >0.45 mm and

about 50% <0.083 mm. The sample material consists of about 50% degraded bedrock and thus is mature soil (Taylor et al. 1973). Lithic fragments are mainly anorthositic norite and troctolite with calcium plagioclase, pyroxene, olivine, and only minor basaltic constituents (Cameron et al. 1973).

The landing site of Luna 24 is located in the south-eastern part of Mare Crisium, which fills most of the Crisium multiringed basin (Vinogradov 1972). The Luna 24 regolith is a relatively immature, pure mare soil with minor exotic components and a near bimodal size distribution with maxima at the 200–94 and 40–10 µm fractions (Laul et al. 1987). The soil is dominated by monomineralic particles (Ca-rich feldspar, Ferich pyroxene of mare origin, olivine) with low agglutinate, lithic, and glass contents. Whereas the monomineralic population has a minor highland component, the fraction of glass fragments contains a significant proportion of clasts that are of highland origin (Laul et al. 1987).

Monomineralic grains of plagioclases investigated here mainly consist of transparent, pure plates and splinters with a grain-size of 100–300 µm. All investigations were carried out on polished thin sections of the separated single plagioclase grains, which were produced using low-fluorescing epoxy resin.

#### Experimental methods

We used a “hot cathode” CL microscope at 14 kV and with a current density of ~10 µA/mm<sup>2</sup>. To prevent the build-up of electrical charge, the thin sections were coated with carbon. Luminescence images were captured “on-line” during CL operations by means of an adapted digital video-camera (KAPPA 961-1138 CF 20 DXC with cooling stage). CL spectra were

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obtained using an EG&G (Princeton Research) digital triple-grating spectrograph (resolution of 0.5 nm at 600 nm) with liquid nitrogen cooled, Si-based charge-coupled device (CCD) detector. The CCD camera was attached to the CL microscope by a silica-glass fiber guide (Neuser et al. 1995). Lateral resolution is about 30  $\mu\text{m}$ . CL spectra were measured in the range 320 to 800 nm. Wavelengths were calibrated using a Hg lamp. Intensity was calibrated through comparison with an external standard (iceland spar). To prevent any falsification due to electron bombardment, all spectra were taken on non-irradiated sample spots.

Chemical compositions were determined using a JEOL JXA-8900 RL electron microprobe. Beam conditions were 15 kV, 10 nA, and a defocused beam with a diameter of 20  $\mu\text{m}$ . Counting times for each spot were 20 s for Si, Al, Ca, and Na; 100 s for Ti, Fe, Mg, Ba, Sr, and K; and 500 s for Mn. To minimize volatilization of Na, this element was analyzed during the first sequence. Background intensities on both sides of the measured element peaks were analyzed with each half of the counting times. Reference intensities were obtained from standard materials including anorthite and pure metal specimens of Ti and Mn.

Concentration of  $\text{Mn}^{2+}$  was determined by quantitative high-resolution spectroscopy of CL emission (QHRS-CL). The method was calibrated for feldspar using an albite sample from Spruce Pine, U.S.A.; (see Götze et al. 1999a), which was simultaneously analyzed by proton-induced X-ray emission (PIXE) and CL spectroscopy (Habermann et al. 1998). The micro-PIXE analyses were done at the Dynamitron Tandem

Laboratorium at the Ruhr-University Bochum (for details see Meijer et al. 1994). At an average spot size of 10  $\mu\text{m}$  and a proton energy of 3 MeV, the detection limit for Mn is 5–10 ppm. At present, the calculated detection limit of QHRS-CL is in the range of 0.1 ppm. To keep the calculated limit of detection (LOD) of micro-PIXE, QHRS-CL and moreover the quantitative ionoluminescence (Homman et al. 1994) comparable it was calculated on the same way using the GUPIX software package (Maxwell et al. 1995).

## RESULTS

Luna 20 plagioclase compositions from  $\text{An}_{91}$  to  $\text{An}_{99}$  dominate (Fig. 1) and no Fe-rich plagioclase fragments were found. The low-Fe (0.014–0.065 wt% FeO), low-K (0.005–0.084 wt%  $\text{K}_2\text{O}$ ) compositions of the Luna 20 plagioclases have been interpreted as to reflect early crystallization of plagioclases from a relatively low Fe magma (Roedder and Weiblein 1973).

Plagioclases of Luna 24 soil contain some more Na (Fig. 1) and some of the plagioclase grains significantly vary in chemical composition. One plagioclase grain (Luna 24-1) is intergrown with a grain of  $\text{SiO}_2$ , which is clearly discernible due to its blue CL. Furthermore, Fe-rich pyroxene inclusions were detected in Luna 24 plagioclase (Fig. 2f), which is a characteristic feature of mare origin (Laul et al. 1987). In general, the concentrations of Fe and K are more scattered than in Luna 20 plagioclases (0.234–0.671 wt% FeO; 0.005–0.529 wt%  $\text{K}_2\text{O}$ ).

The Mg contents range from 100 to 3000 ppm, which is similar to data reported by Steele et al. (1980). Mg tends to correlate

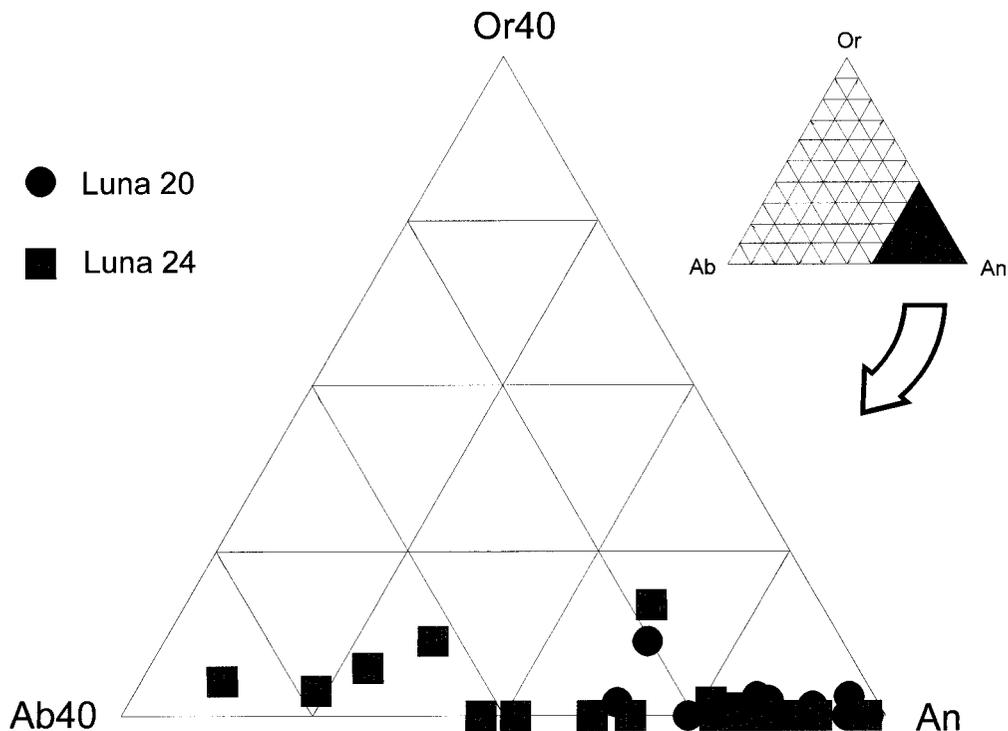
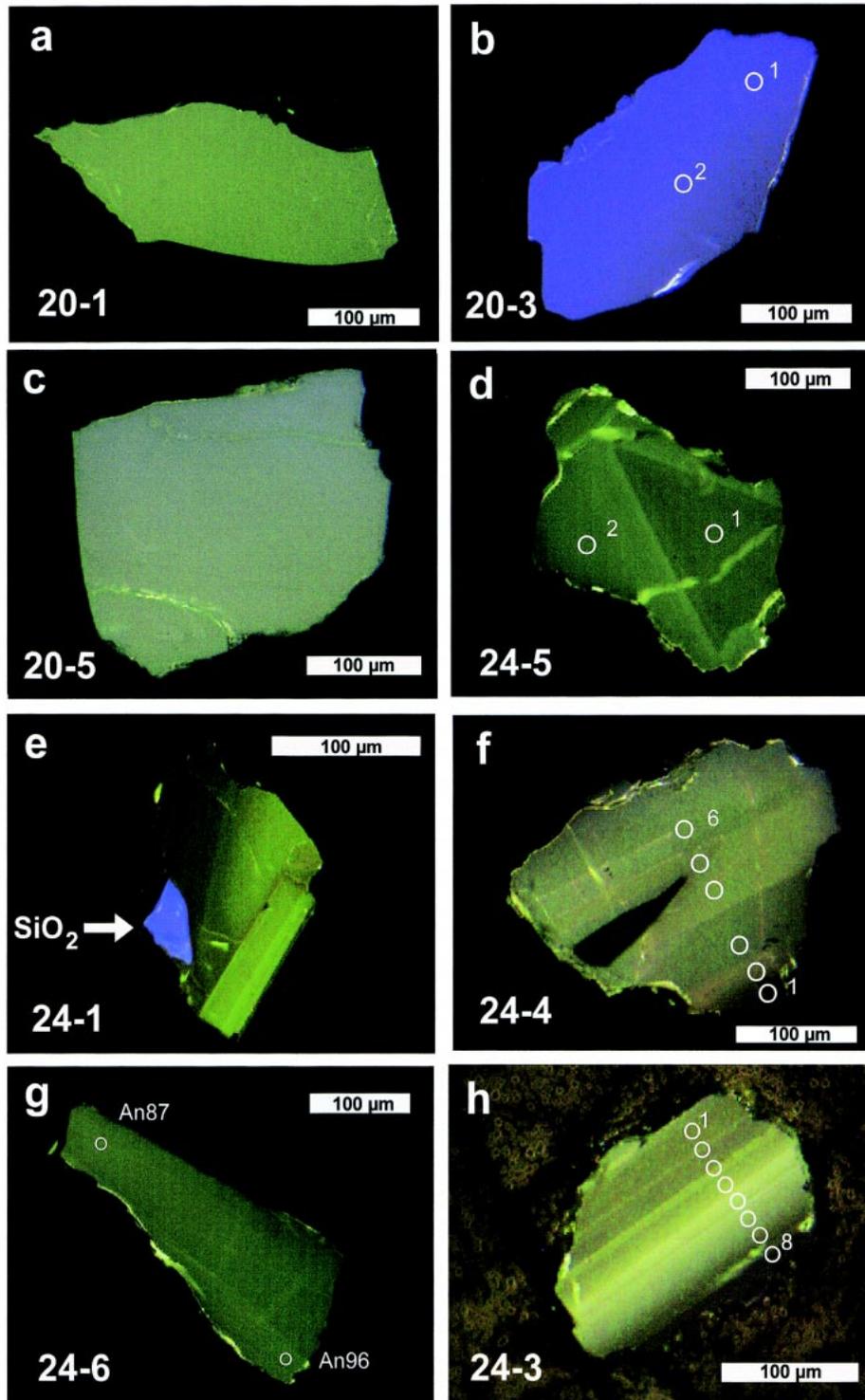


FIGURE 1. Chemical composition of Luna 20 and Luna 24 plagioclases plotted into the Or-Ab-An ternary. Luna 20 samples represent exclusively An-rich feldspars, whereas Luna 24 feldspar compositions are more scattered.



**FIGURE 2.** CL micrographs of lunar plagioclase grains. (a–c) Luna 20 samples show more or less homogeneous CL distribution within single grains without distinct zoning. The grains exhibit both blue and green CL. (d–h) Luna 24 plagioclases luminesce exceptionally greenish and show partially distinct oscillatory zoning. The blue luminescing grain in plagioclase 24-1 (see arrow in 2e) is SiO<sub>2</sub>. Fe-rich pyroxene, which was interpreted by Laul et al. (1987) as characteristic feature of mare origin, is present as non-luminescent, splinter-shaped inclusion in plagioclase 24-4 (Fig. 2f). The circles and numbers in f and h represent spot positions of microchemical analysis (Fig. 3).

with Ca in plagioclases. We did not detect Ba (LOD 90 ppm BaO) and Sr (LOD 310 ppm SrO) was observed only once.

The colors range from pale blue to green (Fig. 2). The plagioclase crystals of Luna 20 appear more or less homogeneous under CL, showing both blue and green CL colors. Only irregular areas with slightly different CL colors are detectable within the bluish luminescing plagioclase grains (compare Fig. 2b), but no regular zoning is visible.

In contrast, Luna 24 plagioclases exhibit only greenish luminescence colors and moreover show partly distinct oscillatory zoning under CL (Figs. 2d–h). In Luna 24–3 (Fig. 2h) the zone with the brightest CL is Ca rich ( $An_{95}$ ) grading to  $An_{71}$  in the zone with the dullest CL. However, other plagioclase grains with significant variations in the chemical composition show no differences in the CL behavior (e.g., Luna 24–6, see Fig. 2g). Therefore, it is doubtful that the luminescence characteristics of the plagioclases are primarily caused by variations of major components. Instead, the CL intensity shows a correlation with the variations in the Mn concentration within the plagioclase grains (Figs. 3, 2f, and 2h).

Spectral analysis of the CL emission revealed that the plagioclases of Luna 20 and Luna 24 show in general three main CL emission bands in the blue around 450 nm, in the green at ~560 nm, and in the red/IR at ~690 nm (Figs. 4–6). These are also the most common emission bands found in terrestrial plagioclases (e.g., Geake et al. 1973; Marshall 1988; Götze et al. 1999b). The intensities of these emission bands vary between plagioclases of the different samples as well as within single feldspar grains causing the different CL colors and CL zoning (Fig. 2).

### DISCUSSION

The green peak at ~560 nm is the most dominant emission in all samples (Figs. 4–6). According to Geake et al. (1972,

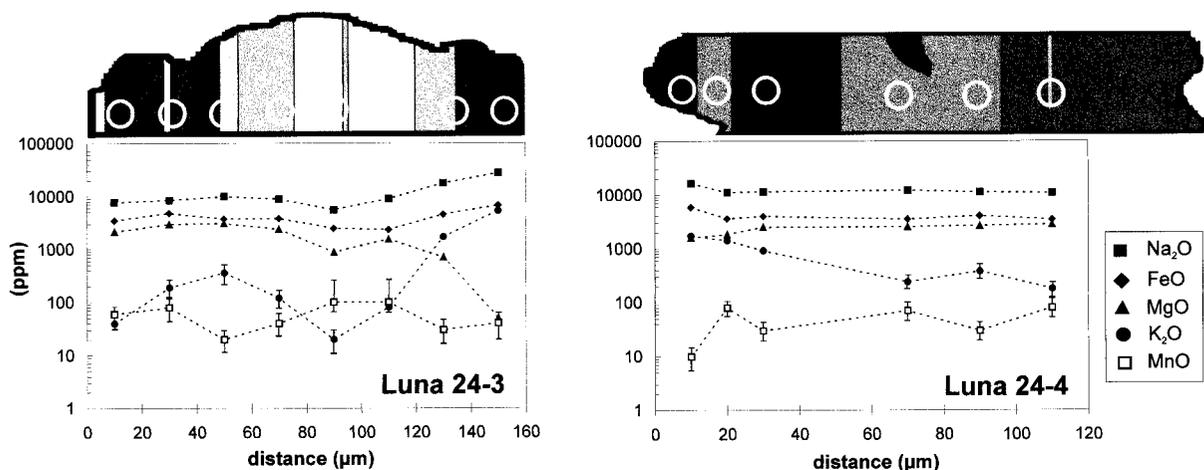
1973) this emission band centered around 560 nm is due to  $Mn^{2+}$  in M sites (most probably  $Ca^{2+}$  sites). They reported this luminescence emission in almost all plagioclases from Apollo samples and meteorites and concluded that  $Mn^{2+}$  is the dominant activator in lunar and other extra-terrestrial material because of its wide availability and the common substitution for Ca and Mg. Measurements on synthetic anorthites (Telfer and Walker 1978) and natural plagioclases (Mora and Ramseyer 1992; Götze et al. 1996) have shown a clear correlation between the intensity of the green emission band and the Mn content.

Based on these results, we attempted to determine the Mn concentration by quantitative analysis of the CL emission intensity. Using the method of Habermann et al. (1998) the intensities of the 560 nm band in the lunar plagioclases were quantified by a noncommercial software, and Mn concentrations were calculated from a reference standard albite sample. Mn concentrations of 7–47 ppm are indicated. The CL zoning in plagioclase grain Luna 24–3 (compare Figs. 2h and 5) is caused by variations of the Mn content between 7 and 31 ppm.

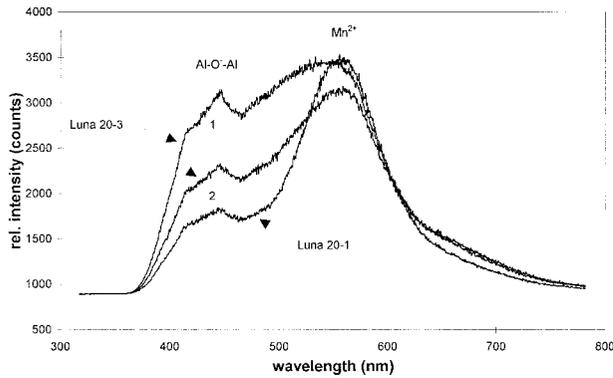
The intensity of the blue emission at ~450 nm in most lunar plagioclases is relatively weak. Only some of the Luna 20 anorthites show a strong emission in the blue (Figs. 2b and 4). In these samples, both the blue and green emission bands determine the integral CL intensity and the resulting visual CL color of the plagioclase grains.

The origin of the blue emission in feldspars has been extensively discussed (cf. Marshall 1988). Although initial results of Mariano and Ring (1975) connected the blue emission with Ti in Al sites ( $Ti^{3+}$  electron center), it is not clear yet whether Ti is an activator or a sensitizer enhancing intrinsic CL. Here, Ti shows no relation to the CL intensity. Similar results were reported from CL investigations on synthetic and meteoritic anorthite (Steele et al. 1997).

Walker (1985) related the blue emission to structural de-



**FIGURE 3.** Compositional profiles. Top, schematic illustrations of the spot positions of plagioclases Luna 24-3 and Luna 24-4 in relation to the CL emission intensities (compare Figs. 2f and 2h). The zones with the brightest CL correspond to the highest Mn concentrations. Bottom shows various elemental profiles. Symbols are at the right side. Error bars ( $2\sigma$  standard deviation) are marked for  $K_2O$  and  $MnO$ , for the other elements the error bars are within the range of the symbol size.



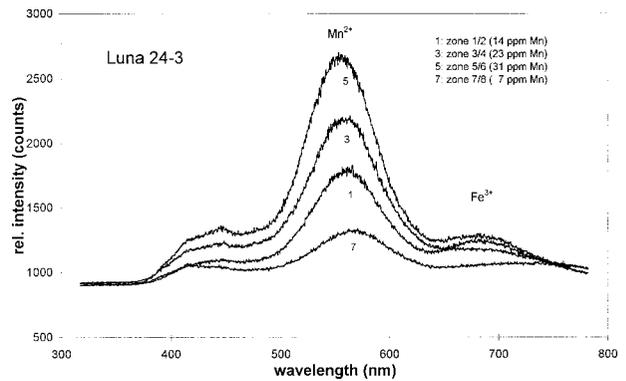
**FIGURE 4.** CL emission spectra of plagioclase grain Luna 20-1 (visible green CL, compare Fig. 1a) showing dominant  $\text{Mn}^{2+}$  activated emission at 560 nm and grain Luna 20-3 (visible blue CL, compare Fig. 2b) with an additional strong emission band in the blue at ~450 nm (probably Al-O-Al center). The calculated Mn concentration of grain Luna 20-1 is 47 ppm. Variations in the visible blue CL color in grain Luna 20-3 (zone 1 and 2 in Fig. 2b) are caused by variations in the intensity ratios of the blue and green emission bands.

fects (electron-hole recombinations at various hole centers) similar to those in other silicates. The CL emission band around 450 nm may be caused by the substitution of  $\text{Al}^{3+}$  for  $\text{Si}^{4+}$  in the feldspar structure. Variations in the peak position are associated with non-equivalent sites for the impurity Al resulting in different types of Al-O-Al centers (Kirsh et al. 1987). The Al-O-Al (or  $\text{O}^{1-}/^{27}\text{Al}$ ) center forms with two Al atoms, one of which is “structural Al” and the other “impurity Al” (e.g., Marfunin 1979). The formation of short-range disordered domains is due to Al-O-Al clustering associated with structural defects in their local environment (Petrov et al. 1993). Thus, the strong blue CL emission in some of the Luna-20 plagioclases indicates abundant lattice defects.

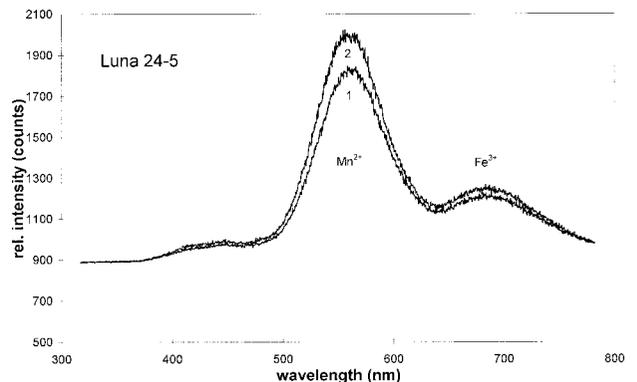
In some Luna 24 plagioclase crystals an emission band in the red-IR at ~690 nm was clearly detected (Figs. 5 and 6). This is of special interest because the red emission band was not detected with any certainty for Luna 20 (see Fig. 4). Sippel and Spencer (1970) found no evidence of a ~690 nm peak in any of more than one hundred investigated lunar plagioclase grains.

Geake et al. (1973), Telfer and Walker (1975, 1978), and White et al. (1986) showed that the red emission band around 700 nm is due to  $\text{Fe}^{3+}$ , which occupies  $\text{Al}^{3+}$  tetrahedral sites in the feldspar lattice. The deep-red luminescence is due to the  ${}^4\text{T}_1 \rightarrow {}^6\text{A}_1$  electronic transition of tetrahedral  $\text{Fe}^{3+}$  (Telfer and Walker 1978; White et al. 1986). The sensitivity of the red peak position to anorthite content in terrestrial feldspars has been previously reported (e.g., Sippel and Spencer 1970; Geake et al. 1973; Telfer and Walker 1975; Mora and Ramseyer 1992; Götze et al. 1996). Both the structural state of the plagioclase and the tetrahedral site occupancy of  $\text{Fe}^{3+}$  can affect the position of the red peak (Telfer and Walker 1978; Petrov et al. 1989). The position of the red peak in the investigated Luna 24 plagioclases centered at 690 nm correlates well with the results of Ca-rich terrestrial plagioclases.

The weak or absent red emission band in lunar plagioclases



**FIGURE 5.** CL emission spectra of different zones within plagioclase grain Luna 24-3, which are clearly discernible by different CL intensities and shades of green CL (compare Fig. 2h). The visible CL zoning is mainly caused by varying intensities of the  $\text{Mn}^{2+}$  emission at 560 nm. The intrinsic blue emission band and the red band due to  $\text{Fe}^{3+}$  show only slight variations. The Mn contents calculated from quantitative spectral analysis of CL emission are 7 ppm (zone 7/8), 14 ppm (zone 1/2), 23 ppm (zone 3/4), and 31 ppm (zone 5/6)



**FIGURE 6.** CL emission spectra taken from two spots within plagioclase grain Luna 24-5 showing a clear emission at ~690 nm, which can be related to  $\text{Fe}^{3+}$ . The two spectra of spot 1 and 2 (compare Fig. 2d) only slightly differ in the intensity of the  $\text{Mn}^{2+}$  emission (calculated Mn concentrations are 18 and 20 ppm, respectively).

has been ascribed to low  $f_{\text{O}_2}$  during plagioclase crystallization (Geake et al. 1972, 1973). Nevertheless, the occurrence of the CL emission band at 690 nm indicates that at least some of the Fe in lunar plagioclases is ferric.

Niebuhr et al. (1973) concluded from ESR studies of lunar plagioclases that one percent of the total amount of Fe in investigated anorthite was  $\text{Fe}^{3+}$  in tetrahedrally coordinated  $\text{Al}^{3+}$  sites. Considering this, calculations from microchemical analyses in this study would provide contents of  $\text{Fe}^{3+}$  in the range of a few up to about 100 ppm. Comparison of our spectral CL measurements with results of extensive measurements on terrestrial plagioclases (Götze et al. 1999b) indicate that the  $\text{Fe}^{3+}$  concentrations in Luna 24 plagioclases are below 100 ppm. However, the quantification of the red emission peak is difficult because much of the Fe chemically measured as traces in

the lunar feldspars is likely incorporated as Fe<sup>2+</sup> or present in accessory phases.

The possible sources of Fe<sup>3+</sup> in lunar material are widely discussed. Ferric iron could give an indication of free oxygen available during formation. For lunar mare basalts p(O<sub>2</sub>) was estimated to be 10<sup>-15</sup> to 10<sup>-16</sup> at 1000 °C, whereas p(O<sub>2</sub>) for terrestrial anorthosite (e.g., Stillwater anorthosite) was suggested to be ~10<sup>-12</sup> (Schürmann and Hafner 1972). It was concluded that the Fe<sup>3+</sup>/Fe<sub>tot</sub> ratio in plagioclase can reflect the p(O<sub>2</sub>) during crystallization. On the other hand, secondary processes may have changed the Fe<sup>2+</sup>/Fe<sup>3+</sup> ratio. Niebuhr et al. (1973) suggested that during subsequent recrystallization substantial cation redistributions due to intra- and intercrystalline exchange occurred that may have also effected the ferric to ferrous iron ratio. Agrell et al. (1972) detected goethite in a weakly recrystallized lunar breccia (Apollo 14) indicating that fine lunar surface materials may have been oxidized on the moon. Griscom and Marquardt (1972) also detected traces of Fe<sub>2</sub>O<sub>3</sub> in lunar soil that they related to oxidation processes from cometary impacts or endogenous sources. It is not clear yet whether the Fe<sup>3+</sup> is of primary origin or due to other secondary processes (e.g., radiogenic oxidation), although the absence of Fe<sup>3+</sup> in Luna 20 samples but the presence in Luna 24 material indicates primary origin.

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