Exsolution and Al-Si disorder in alkali feldspars: Their analysis by infrared spectroscopy

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ABSTRACT

An experimental method for the determination of Ab-Or exsolution and Al-Si ordering in alkali feldspar is described. Powder infrared spectroscopy was used to measure spectra in the spectral range between 50 and 1500 cm⁻¹ using about 5 mg of sample material. The spectra were analyzed using reference spectra of uniform samples with various Ab-Or compositions and several degrees of Al-Si order. The application of this method is demonstrated for two examples of exsolved feldspar minerals, and it is shown that detailed characterization of alkali feldspars using IR spectroscopy leads to new insight into their structural details.

INTRODUCTION

Infrared spectroscopy is a sensitive tool for the determination of both the chemical composition of a sample and its structural state (Salje 1992, 1994). Previous work concentrated on the determination of the local degree of Al-Si order in sodium feldspar by using line shifts and intensity changes in the spectral region between 500 and 700 cm⁻¹ (Salje et al. 1989; Zhang et al. 1996), the separation of T₁-T₁ ordering from T₁₀-T₁m ordering in potassium feldspar (Harris et al. 1989), study of the structural phase transition I₁-P₁ in calcium feldspar (Redfern and Salje 1992), investigation of the kinetics of the monoclinic to monoclinic phase transition in BaAl₂Ge₂O₈ feldspar (Malcherek et al. 1995), and of Al-Si ordering in strontium feldspar (Benna et al. 1995). This paper presents an easy-to-handle experimental method for the quantitative characterization of alkali feldspars. Systematic applications of this approach will be published in a separate paper.

EXPERIMENTAL METHOD

Infrared technique

Infrared spectroscopy was performed using a standard Bruker 113 V Fourier-transform infrared spectrometer. The conventional IR pellet technique with about 5 mg of sample material was used. The sample preparation and experimental details were reported previously (e.g., Zhang et al. 1996). Each spectrum was calculated by Fourier transformation of 512 scans. All spectra were then recorded as absorbance A, with A = −log₁₀(Sample/Reference), where S is the single-beam transmission intensity.

In general, a typical procedure for the quantitative analysis of an unknown alkali feldspar sample involves the following basic steps: (1) preparation of standard samples, including the acquisition of reference spectra and analysis of reference spectra to determine the spectral bands suitable for better quantitative analysis; (2) calculation of a calibration curve using the reference spectra; and (3) acquisition of spectra of the unknown sample, followed by an analysis of its chemical composition and degree of Al-Si order. These steps will now be described in some detail.

Standard samples for calibration

Synthetic alkali feldspars with various Ab-Or compositions were prepared as reported in Zhang et al. (1996). Lattice parameters were determined by X-ray Guinier powder diffraction. The chemical compositions of the products were confirmed using the lattice parameters published by Kroll et al. (1986). In addition, a fully disordered synthetic Or₉₀.₁ sample (Kroll et al. 1986) was used as a standard.

To obtain standard spectra for different degrees of Al-Si order, Amelia albite was annealed at 1353 K for annealing times between 0.5 h and 9 d. Similar samples were studied previously (Salje et al. 1989) by IR spectroscopy in the region between 450 and 1500 cm⁻¹. The spectral range has now been extended to 50–1500 cm⁻¹.

Analyzed samples

Two natural samples were chosen to demonstrate the application of the analytical method. One sample, a microperthite, GGU140021, originated from the Klokken syenite intrusion, southern Greenland. It contains ordered albite-microcline intergrowths with an average composition of Or₄₇. Details of the bulk composition, lamellar exsolution, and microstructures were reported by Brown et al. (1983). The second example is a feldspar from Sri Lanka, VSL510, which was previously described by Voll et al. (1994). Further details of the two samples and some samples used for calibration are given in Table 1.
Spectral analysis of standard samples

Al-Si disordering in alkali feldspars leads to a continuous decrease of band intensities, shifts of band frequencies, and an increase of linewidths (Salje et al. 1989; Harris et al. 1989). The peak profiles of the IR spectra are also affected by changes of chemical composition (Zhang et al. 1996). For the analysis of unknown samples, their IR spectra are compared with two different sets of reference spectra. The first set is called the “primary reference spectra.” It consists of a series of spectra of natural and synthetic samples with well-defined chemical composition and degree of Al-Si order. These spectra were published by Zhang et al. (1996). The second set is called the “secondary reference spectra.” These spectra were not measured directly but calculated by superposition of primary reference spectra. They are needed for the reconstruction of the spectra of unknown samples and provide a much finer grid for the analysis than provided by the primary reference spectra. A useful subset of secondary reference spectra was constructed by the superposition of spectra of fully ordered Or$_{100}$ and Ab$_{100}$ (Fig. 1). These were used for the analysis of unknown samples with high degrees of Al-Si order. In this case, the spectral weights of the two primary reference spectra were changed to obtain a best fit with the unknown spectra.

IR modes sensitive to Ab-Or composition

Exchange of Na and K causes specific spectral features to vary in ordered alkali feldspars. The most obvious variation is in the stretching bands between 700 and 800 cm$^{-1}$, with four separated bands in the Na-rich end-member and two bands in K-rich samples (Fig. 1). In the spectral region between 350 and 500 cm$^{-1}$, the effects of composition on the line profiles can be seen near 400 and 470 cm$^{-1}$. The most significant spectral variations caused by exchanging Na and K, or varying Ab-Or concentration, occur in the spectral region below 300 cm$^{-1}$. This spectral region was found to be most useful for the analysis of exsolved samples. For samples with high degrees of Al-Si order, the low-frequency bands include the K-O band at 114 cm$^{-1}$ and the Na-O band at 184 cm$^{-1}$ (Fig. 2). Spectral analysis of mechanical mixtures of samples and
exsolved binary alkali feldspars with coarse exsolution lamellae shows that the band intensities change linearly with composition. For a quantitative measurement, the relative absorbance $A_{114}$ of the K-O band at 114 cm$^{-1}$ and the absorbance $A_{184}$ of the Na-O band at 184 cm$^{-1}$ for the constructed secondary reference spectra were measured, using a horizontal linear baseline through 240 cm$^{-1}$. The effect of Ab-Or composition on the value of the absorbance near this wavenumber is weak. For the case of samples with high degrees of Al-Si disorder, the bands are located near 115 and 202 cm$^{-1}$. The ratios of $A_{114}$ and $A_{184}$ and of $A_{115}$ and $A_{204}$ correlate linearly with Ab-Or content (Fig. 3). The curves were obtained from fitting the measured data to the following function:

$$X_{ab} = \frac{k_1 + k_2 r}{1 + k_3 r}$$  

where the coefficients $k_1$, $k_2$, and $k_3$ were determined using the primary reference samples and $r$ is the ratio of the absorbances. For the case of highly Al-Si ordered samples, the numerical values are: $k_1 = 2.8$, $k_2 = 99$, $k_3 = 0.1$, and $r = A_{114}/(A_{114} + A_{204})$. For the case of Al-Si disordered material, the corresponding parameters are: $k_1 = 1.6$, $k_2 = 90$, $k_3 = 0$, and $r = A_{115}/(A_{115} + A_{204})$.

In addition to phonon signals in the far-infrared regime, some bands in the mid infrared were used to determine the Ab-Or composition of each exsolved phase or, alternatively, of the homogeneous synthetic alkali feldspars. The infrared bands between 500 and 680 cm$^{-1}$ were examined; these bands were previously used to extract
The effect of various Ab and Or components and different degrees of Al-Si order on the infrared spectra of alkali feldspars in the region between 500 and 680 cm\(^{-1}\). The spectra are normalized for better comparison with (a) primary reference spectra of unexsolved samples (\(Q_{od} = 1\)) with compositions given in Table 1; (b) secondary reference spectra of exsolved samples with various concentrations of Ab\(_{100}\) and Or\(_{100}\) components (\(Q_{od} = 1\)); (c) primary reference spectra of Ab\(_{100}\) with various values of \(Q_{od}\).

![Figure 4](image)

Structural information from sodium and potassium feldspars (Salje et al. 1989; Harris et al. 1989; Zhang et al. 1996). The spectra of homogeneous synthetic feldspars in Figure 4 show the effects of Ab-Or content and the degree of Al-Si order on the infrared bands in this spectral region. The spectra in this frequency region are normalized to show the differences more clearly. From Figure 4 it is seen that the band at 650 cm\(^{-1}\) does not exhibit significant changes in peak position with chemical composition. The band at about 540 cm\(^{-1}\) changes its frequency with composition (Figs. 4a and 4b) and also varies with degree of Al-Si order (Fig. 4c). This means that for the 540 cm\(^{-1}\) band both the Ab-Or content and the degree of Al-Si order cause similar spectral effects. The band at 580 cm\(^{-1}\) exhibits less order-disorder dependence and shows a stronger composition dependence than the bands at 540 and 650 cm\(^{-1}\). The peak position of the band at 580 cm\(^{-1}\) shows a nearly linear correlation with chemical composition. The peak profile analysis was performed for the IR bands between 500 and 680 cm\(^{-1}\). Lorentzian functions were used for the bands at 540, 607, and 650 cm\(^{-1}\); a mixture of Gaussian and Lorentzian functions (with 30–40% Lorentzian) was used for the peak at 580 cm\(^{-1}\). In the case of samples with high degrees of Al-Si order, the position of the peak near 580 cm\(^{-1}\) is plotted as a function of bulk composition in Figure 5. In this figure, solid symbols represent peak positions from unexsolved standards and open symbols correspond to those from secondary reference spectra, which are equivalent to those of exsolved samples. The line in Figure 5 connects the data of Ab\(_{100}\) and Or\(_{100}\) and represents all intermediate compositions rather well. The large linear shift of the peak positions allows a fairly accurate determination of the bulk composition of the sample once the position of the 580 cm\(^{-1}\) band has been determined.

Effects of Al-Si (dis)order

The degree of Al-Si order is quantified by the two order parameters \(Q_{od} = (t_{in} - t_{in})(t_{in} + t_{im})\) and \(Q = (t_{i} - t_{j})(t_{i} + t_{j})\) where \(t_{i}\) denotes the occupancy of Al on the crystallographic position indicated (Salje 1985; Salje et al. 1985; Carpenter and Salje 1994). The effect of Al-Si ordering is to sharpen the line profiles and shift the phonon bands (Salje et al. 1989; Harris et al. 1989). The time evolution of infrared spectra of Ab\(_{100}\) annealed at 1353 K (Fig. 6) shows a gradual change from well-resolved
sharp bands in low albite to broad absorption bands in high albite, resulting from a decrease in the degree of Al-Si ordering, \( Q_{od} \). The effect of decreasing order in albite on the infrared spectra is seen clearly in several spectral regions. Between 1000 and 1200 cm\(^{-1} \), the Si(Al)-O stretching bands show significant broadening with increasing degree of Al-Si disorder. The bands in the region between 700 and 800 cm\(^{-1} \) cannot be resolved for disordered samples. The Si-O-Si(Al) phonon absorption near 334 cm\(^{-1} \) exhibits a remarkable decrease in absorbance. Low values of \( Q_{od} \) affect the Na-O bands at low frequencies so much that they all become broad, whereas the ratio of the height of 184 cm\(^{-1} \) and that of 200 cm\(^{-1} \) decreases with decreasing \( Q_{od} \). The fact that the spectral features in the region of 370–500 cm\(^{-1} \) broaden makes it possible to estimate the degree of Al-Si order directly from the appearance of these peak profiles. Such changes do not depend on the composition of individual phases or on bulk composition of mechanical mixtures of two alkali feldspars with different composition (Salje et al. 1989; Harris et al. 1989; Zhang et al. 1996). The degree of Al-Si order can be estimated quite simply by superposition of spectra with \( Q_{od} = 0 \) and \( Q_{od} = 1 \) with the same bulk chemical composition. A spectrum \( S \) with unknown \( Q_{od} \) can be simulated using two spectra \( S_{ordered} \) and \( S_{disordered} \) with \( Q_{od} \) of 1 and 0, respectively:

\[
S = Q_{od}^1 S_{ordered} + (1 - Q_{od}^1) S_{disordered}
\]

The rationale behind this formula is that all spectral features such as line position, linewidth, and peak intensities in alkali feldspars scale with the square of the order parameter. For small spectral changes the entire spectrum can be reconstructed by interpolation of the spectra of fully ordered and fully disordered material (e.g., see Salje and Bismayer 1997 for details).

**FIGURE 5.** Calibration curve for bulk composition and peak position of the 580 cm\(^{-1} \) band in alkali feldspars with \( Q_{od} = 1 \). (solid circles = standard spectra; open circles = superposition of the spectra of the two end-members).

**FIGURE 6.** Infrared spectra of low albite annealed at 1353 K for various times. With increasing annealing time, the degree of Al-Si order decreases and the peaks broaden gradually.

The broadening of phonon signals because of decreasing \( Q_{od} \) is quantitatively different from that caused by heterogeneous distributions of K and Na. As an example, the Na-O bands at 186 and 201 cm\(^{-1} \) and the K-O band at 113 cm\(^{-1} \) do not change much by chemical mixing but broaden because of Al-Si disorder. Similarly, the linewidth of the band at 650 cm\(^{-1} \) is very sensitive to changes of \( Q_{od} \) while being less sensitive to Na-K exchange. For fully ordered samples (with or without exsolution), this band shows a linewidth of 11 cm\(^{-1} \), whereas the variation of the width (full width at half maximum (FWHM)) because of chemical change is only about 0.7 cm\(^{-1} \) (Fig. 7). For the case of \( Q_{od} = 0 \), the band is nearly 10 cm\(^{-1} \) wider than for the case of \( Q_{od} = 1 \). This band does not overlap with other bands and can be easily analyzed. With \( \Gamma_{650,cm} \propto Q_{od}^2 \) (where \( \Gamma_{650,cm} \) is the FWHM of the mode at 650 cm\(^{-1} \)) and using a quadratic function to represent the relation between composition and linewidth for \( Q_{od} \approx 0 \) and 1, we obtain the bandwidth as functions of \( Q_{od} \) for various compositions.

**RECIPIES**

To analyze natural samples, different IR regions and different analytical methods were needed to determine the various structural and chemical parameters. The following “recipe” is commonly used for the analysis. (1) The degree of Al-Si order in samples is estimated to decide if the reference spectra of ordered or disordered standard...
spectra should be used in the next stage. (2) The sample is checked for exsolution. (3) The Ab-Or components of each phase are obtained for the case of exsolved samples. These steps are now described in detail.

**Determination of the degree of Al-Si order, \( Q_{od} \)**

The linewidth of the band near 650 cm\(^{-1} \) is determined in the following way: Fit the bands between 500 and 680 cm\(^{-1} \) to Lorentzian functions, apart from the band at 580 cm\(^{-1} \), which is fitted using Voight profiles. A free linear baseline should be used. From the linewidth of the band at 650 cm\(^{-1} \), the corresponding value of \( Q_{od} \) for the unknown sample follows from the line width \( G \) as:

\[
Q_{od} = \frac{G_{disordered} - G_{ordered}}{G_{ordered}}
\]

where \( G = 20.6 \) cm\(^{-1} \) and \( G = 12 \) cm\(^{-1} \) are the composition-averaged linewidths of the band near 650 cm\(^{-1} \) for fully ordered and fully disordered materials.

For further refinement of the analysis, \( Q_{od} \) is determined by reconstructing the sample spectra using the primary reference spectra. The method was described in a previous section.

For exsolved samples, this method leads to an averaged value of \( Q_{od} \) for Ab-rich and Or-rich phases. Methods for the determination of \( Q_{od} \) for each phase are described below.

**Identification of exsolution**

Exsolved and homogeneous samples show very different IR spectra in the range between 70 and 250 cm\(^{-1} \) (Fig. 8). Such low-frequency phonons relate mainly to vibrations between the large cations and the surrounding structure. The reason for large spectral variations stems from the fact that spectra of binary feldspars in this low-frequency region are not simply the superposition of the spectra of the end-members but contain additional phonon signals resulting from the non-ideal mixing of alkali feldspars. The analysis consists of curve-fitting to the relevant IR bands between 70 and 250 cm\(^{-1} \) with Lorentzian line profiles, followed by matching the phonon frequencies to those of the primary reference samples. Most conveniently, the bands at 113 and 138 cm\(^{-1} \) for Or-rich phase and the bands at 184 and 216 cm\(^{-1} \) for Ab-rich phases are used for the quantitative analysis.

**Determination of bulk composition**

Three methods are generally applied. In the first method, the unknown spectra of exsolved samples are decomposed by curve-fitting or spectral subtraction in the spectral region between 90 and 250 cm\(^{-1} \). It is then assumed that the measured spectrum is the superposition of two spectra that correspond to the two exsolved phases. Often the curve-fitting can be limited to the modes near 114 and 184 cm\(^{-1} \). The absorbances of these bands may then be evaluated as for the case of \( Q_{od} = 1 \).
The simultaneous existence of absorption bands at 114 and 185 cm\(^{-1}\), but none of the characteristic bands of hypersolvus anorthoclase, indicates that the sample is exsolved. To determine the compositions of the two phases, the regions of the spectrum between 85 and 250 cm\(^{-1}\) were compared with superpositions of pairs of secondary reference spectra of fully Al-Si ordered alkali orthoclase and albite. Limits and errors

High-quality spectra are required for any analysis because the variations of peak positions in alkali feldspar are generally relatively small. As a typical example, the total change of the peak position of the 580 cm\(^{-1}\) band between Ab\(_{100}\) and Or\(_{100}\) is about 10 cm\(^{-1}\) for Q\(_{sd}\) values close to unity. Calibrated reference samples are usually run just before or after measurements of the unknown samples to reduce any instrumental errors. When Q\(_{sd}\) is of intermediate value, the analysis based on peak position becomes less straightforward because a band frequency may be affected by both Al-Si disorder and composition changes. Maximum errors of 0.2 in Q\(_{sd}\) and X were found by repeated, independent measurements.

Another potential source of uncertainty is the construction of the secondary reference spectra. To check for systematic errors, the powders of Ab\(_{100}\) and Or\(_{100}\) end-members were mixed in various concentrations and the spectra of the mixtures recorded. Then the spectra were compared with the calculated reference spectra. The spectra were virtually identical showing that this potential error is not relevant in our experiments.

Further potential sources of error exist, such as the following. (1) Significant errors are introduced by uncertainties in the concentrations of the calibration samples, e.g., the amount of sample in the pellets (weighing errors). (2) Different grain sizes of various samples may result in erroneous line broadening of the spectra. Insufficient grinding can cause variations in analysis of the degree of exsolution and the degree of Al-Si ordering as it affects the bandwidths and absorbance of IR bands. Grain sizes of less than 2 \(\mu\)m are recommended. (3) Changes in the spectrometer settings may also affect the peak profiles. All spectra were analyzed in exactly the same way as the standard samples (e.g., using the same experimental parameters, detector, aperture, number of interferograms, scan speed, beamsplitter, matrix materials, and so forth). (4) The existence of impurity phases such as anorthite or quartz may lead to additional errors in the analysis. First, these impurities may affect the weight of the materials in the pellets. Second, anorthite contains spectral features similar to those of alkali feldspar (Redfern and Salje 1993). The IR bands of An\(_{100}\) may affect the real position of the infrared absorptions at 530 and 650 cm\(^{-1}\) as well as the band near 580 cm\(^{-1}\). Infrared spectroscopic study of the plagioclase feldspar solid solution (Thompson and Milton 1957) has shown that the infrared bands at 640 cm\(^{-1}\) in low albite shift to lower frequencies with increasing Ca content whereas the band at 540 cm\(^{-1}\) shifts to higher frequencies. Such impurities are easily recognized because An\(_{100}\) has a characteristic isolated absorption peak at 238 cm\(^{-1}\) and quartz has sharp absorption peaks at 264 and 697 cm\(^{-1}\) that do not overlap with the IR peaks of alkali feldspars.

Analysis of a microperthite

As the first example, the IR spectra of a microperthite from the Klokken syenite (sample GGU140021, Brown et al. 1983) were analyzed. A TEM image of the microtexture in this sample was shown by Brown et al. (1983, Fig. 6b). It is a low albite, low-microcline braid cryptoperthite. The spectra in Figure 9 show well-resolved phonon bands, which indicate that the sample has a high degree of Al-Si order. A quantitative analysis is based on curve-fitting of the bands between 500 and 680 cm\(^{-1}\). The line width of the band at 640 cm\(^{-1}\) was found to be 14.7 cm\(^{-1}\). The corresponding value of Q\(_{sd}\) follows from Equation 2 with the numerical value Q\(_{sd}\) = 0.83. This estimate of the overall degree of Al-Si order shows that the sample is highly ordered.

\[
X_{ab} = \frac{a_1 H_{114}}{a_1 H_{114} + a_2 H_{184}} \quad \text{(in wt%)} \quad (3)
\]

\[
X_{ab} = \frac{a_2 H_{184}}{a_1 H_{114} + a_2 H_{184}} \quad \text{(in wt%)} \quad (4)
\]

with \(a_1 = 6.57\) and \(a_2 = 6.07\). \(H_{114}\) and \(H_{184}\) are the heights of the bands at 114 and 184 cm\(^{-1}\). For samples with low degrees of Al-Si order, the coefficients \(a_1\) and \(a_2\) are modified. In this case, a full spectral analysis for comparison with the primary and secondary reference spectra is recommended. Alternatively, composition is determined using the relationship in Equation 1 with the coefficients interpolated between those of an Al-Si ordered end-member and an Al-Si disordered end-member. In this case, a full spectral analysis for comparison with the calculated reference spectra is required.
feldspars. Closest agreement between the sample spectrum and the superposed secondary reference spectra was obtained for a combination of Ab$_{100}$ and Or$_{90}$Ab$_{10}$ for the two phases (Fig. 10). Systematic deviations were found when the composition of the two phases changed by more than ±10%, which is the estimate for an upper limit of the analytical uncertainties.

**Analysis of a sample with intermediate value of $Q_{od}$**

From X-ray diffraction analysis the sample VSL510 is monoclinic (i.e., $Q_{od} = 0$ on a macroscopic scale); the T$_1$-T$_2$ ordering is rather high, with $Q_{od} = 0.42$ (Voll et al. 1994). Even a cursory inspection of the IR absorption spectra of this sample shows that the characteristic phonon signals are much sharper than expected for a Al-Si disordered sanidine with $Q_{od} = 0$ (Fig. 9). Although some spectral features are broad, the bands at 370, 400, and 460 cm$^{-1}$ are well resolved. This indicates that the samples are partially ordered. The samples are exsolved as indicated by the bands at 114 cm$^{-1}$ and near 200 cm$^{-1}$. Peak profile analysis in IR regions between 500 and 680 cm$^{-1}$ yields the linewidths of the band near 640 cm$^{-1}$ as 18.9 cm$^{-1}$. This value corresponds to short-range order equivalent to $Q_{od} = 0.4$. Comparing this result with that of the X-ray diffraction analysis shows that the sample has no long-range triclinic order whereas there is a high degree of short-range order.

The degree of short-range order (which is equivalent to $Q_{od}$ on a macroscopic scale) was also determined by constructing secondary reference spectra. Before this could be done, the composition of at least one of the exsolved phases had to be determined. From the analysis of the peak positions for the bands between 70 and 170 cm$^{-1}$ in terms of secondary reference spectra, the Or-rich phase was identified as Or$_{94}$Ab$_{6}$. The uncertainty of the analysis is ±5%. Absorption from the Ab-rich phase was too weak to be analyzed in detail. No close match with any reference spectrum of alkali feldspars was found, which indicates that the Ab-rich phase contains an important An component. Chemical analysis of the macroscopic sample (Voll et al. 1994) led to an overall composition, which can now be broken down into Or$_{90}$Ab$_{10}$ for the Or-rich phase and Or$_{90}$Ab$_{6}$An$_{36}$ for the Ab-rich phase.

The degree of short-range Al-Si order was then determined for the Or-rich phase. In Figure 11 the first derivatives of the measured spectrum (dots) are compared with the calculated derivatives of the secondary reference spectra for potassium feldspars with different degrees of Al-Si order. The closest agreement was found for $Q_{od} = 0.53$. The uncertainty of this analysis is estimated to be ±0.2. The value of $Q_{od}$ of the Or-rich phase is only slightly larger than the overall value for the sample, as esti-
estimated from the line width of the 640 cm\(^{-1}\) phonon signal. The reason for this tendency is that any degree of disorder or variation in the chemical composition (e.g., An content) causes an increase in the line width of the high-frequency phonon (and leads to a lower value of \(Q_{od}\)). The phonons at low wavenumbers are specific for the Or-rich phase and are much less susceptible to impurity phases. In summary, the sample VSL510 was found to be exsolved; the Or-rich phase has a composition close to Or\(_{90}\) and has considerable short-range Al-Si order.

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