The K content of the K-feldspars being measured in optical dating or in thermoluminescence dating

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Mejdahl (1983, 1985) suggested the use of separated potassium feldspars for dating, and showed the feasibility of this. We address here the question of what is the correct potassium content to use to evaluate the β dose rate from the potassium within the grains.

Pure K-feldspar, $KAlSi_3O_8$, has 14.0 wt. % K. The grains in a sediment generally consist of a highly inhomogeneous collection and one can expect grains to be present with a range of K contents between 0 and 14%. The laboratory procedures designed to separate K-feldspars are not perfect and usually result in the presence of other minerals such as quartz and plagioclase feldspars. The situation is further complicated by the fact that different mineral grains have different luminescence sensitivities and it is not unusual to find that most of the light that is being measured arises from a small fraction of the grains of an aliquot. The one observation that appears to make the problem tractable is that it is generally observed that there is a positive correlation between luminescence sensitivity and potassium content.

Our approach to the problem has been to note that 14% is the maximum possible value of the K content, and that a minimum value can be determined from the average K content of the separated grains.

The grains are separated using standard procedures. These are an HCl treatment to dissolve carbonates, sieving to select a grain size, a 6 minute HF etch to clean, but not dissolve the grains, an HCl treatment to dissolve precipitated fluorides, sieving to remove small fragments, selection of the < 2.58 g cm^{-3} fraction using an aqueous solution of sodium polytungstate, and magnetic separation. After optical dating measurements the grains are recovered and about 0.3 g sent for commercial atomic-absorption analysis; the K contents so obtained for a variety of samples are given in the table.

We have sought to obtain the fraction of the separated grains that is K-feldspar by making maps of element concentrations of Si, Al, Na and K using a scanning electron microscope. A monolayer of grains is sprinkled onto an aquadag-coated carbon target, the element maps are made, and then an ordinary white-light photograph made. About 100 grains are then identified and classified. Grains showing only Si are classified as SiO_2. Those with Si and Al usually contain either K or Na or both. Such a grain is classified as K-feldspar if K is clearly dominant and Na feldspar if Na is clearly dominant. Where neither is dominant a grain is classified as mixed. Some grains showed Si and Al but neither K nor Na, and these are listed as "other Si & Al" and may be low-Na plagioclases. The table shows the grain counts and fraction identified as K-feldspar; the latter ranges from 15% to 97%. Here a mixed K & Na grain is counted as 1/2 a K-feldspar grain.

On the assumption that all the K is in the grains identified as K-feldspar, the K content of these grains is calculated, and given in the last column of the table. With two exceptions these figures cluster about a value of about 13% and this figure would appear to be generally appropriate. Two considerations, however, indicate that this value may be too high. The classification of the grains causes bias because of any K in those grains classified as Na feldspar. For some samples this cannot be a large effect because when there is a large fraction that is not K-feldspar, this is mainly quartz. The second consideration is that analyses of single mineral samples of K-feldspar generally yield K contents in the range 10-13% K (Huntley et al, 1988; Spooner, 1992, Prescott and Fox, 1993), and unless museum samples have lower K contents than K-feldspar grains found in sediments, a typical value of 13% for the latter seems unlikely and a slightly smaller value is warranted.

Although this establishes typical K contents of K-feldspar grains it does not take into account the fact that most of the measured luminescence often arises from a small fraction of the grains, and these could have lower or higher K contents. Prescott and Fox

(1993) have shown that there is a high degree of correlation between thermoluminescence sensitivity and K content for a range of K-feldspars, those with over 11% K being the brightest. A similar indication was found by Spooner (1992) for the 1.4 eV (infrared)-excited luminescence. Thus one can expect that the light will be coming from high-K feldspars.

Sample	Separated grains K wt. %	grain counts						K-feldspar fraction	K of K-feldspar grains wt. %
		total	SiO₂	K-feldspar	Na-feldspar	mixed K&Na	other Si&Al		
TTS	4.8	97	19	43	35	0	0	0.44 ± 0.05	10.9 ± 1.2
TTS3	9.05	102	23	69	10	0	0	0.68 ± 0.05	13.3 ± 1.0
PATSI	7.2	71	11	26	21	11	2	0.52 ± 0.06	13.8 ± 1.6
CBTS2	9.6	113	9	77	19	8	0	0.68 ± 0.05	14.1 ± 1.0
NRTS	8.29	185	49	82	27	17	10	0.49 ± 0.04	16.9 ± 1.5
FHTS-3	4.7	184	14	42	76	45	7	0.35 ± 0.04	13.4 ± 1.7
KHTS-1	5.7	160	22	61	35	24	18	0.46 ± 0.04	12.5 ± 1.2
KHTS-2	3.9	160	14	33	78	25	10	0.28 ± 0.04	13.4 ± 1.7
ZCTS	3.8	192	28	64	50	43	6	0.44 ± 0.04	8.4 ± 0.8
SW6-01	7.6	100	38	55	6	1	0	0.55 ± 0.05	13.8 ± 1.2
SAW94-32	6.2	107	45	39	7	11	5	0.47 ± 0.05	13.2 ± 1.4
SAW94-37	8.3	106	29	42	5	26	4	0.64 ± 0.04	13.0 ± 0.8
SAW94-62	2.4	83	41	29	9	4	0	0.37 ± 0.05	6.5 ± 0.9
MELVL93-5	10.7	128	9	95	24	0	0	0.74 ± 0.04	14.5 ± 0.8
CPIW	6.0	98	37	32	2	11	16	0.44 ± 0.05	13.6 ± 1.5
TAG2	7.9	60	20	33	3	15	9	0.60 ± 0.06	13.2 ± 1.3
CTL2	2.3	72	31	11	21	0	9	0.15 ± 0.04	15.3 ± 4.0
DY24	11.5	117	3	104	1	9	0	0.93 ± 0.02	12.4 ± 0.3
SN30	11.9	88	0	83	1	4	0	0.97 ± 0.02	12.3 ± 0.3
SN55	11.4	74	0	69	1	4	0	0.96 ± 0.02	11.9 ± 0.3
SN4d	3.4	96	63	27	2	4	0	0.28 ± 0.05	12.1 ± 2.2

Table: Grain counts, K contents, and deduced K contents of the K-feldspar grains for 21 sediment samples from 7 geographically distinct areas. The first 8 samples are tsunami-laid sands from British Columbia and Washington State (Huntley and Clague, 1996; Baril, 1997). ZCTS is a fluvial sediment from the same region. SW6-01, SAW94-32 & -37 are interdune and dune sands from the Great Sand Hills, Saskatchewan. SAW94-62 is a loess from the Cypress Hills nearby. MELVL93-5 is from California. CPIW and TAG2 are a sand wedge and buried fluvial deposit respectively from the Mackenzie River delta. CTL2 is from near Merritt, British Columbia. DY24 is from a site by the Lena River near Yakutsk, Siberia. The SN samples are from Sandy Neck, Cape Cod, Massachusetts, U.S.A.

There is, however, usually a small fraction of high-Na feldspar (albite) amongst our separated grains, and the same authors show that these can be as bright as the high-K feldspars. There is thus the possibility that these may contribute significantly to the measured luminescence, and in one sample for which we obtained spectra we found this to be the case (Ollerhead et al, 1994). There is, fortunately, an easy remedy for this; since the main K-feldspar emission is at 3.1 eV (400 nm), whereas that of Na-feldspar is at 2.2 eV (570 nm), the use of a blue filter in the measuring system can readily eliminate the latter. In this respect, the quantum efficiency of the photomultiplier tubes commonly used is significantly lower at 2.2 eV than 3.1 eV and this goes some way to accomplishing the same objective.

From the combination of information above a suitably conservative estimate of the K content to use would appear to be 12.5 ± 0.5%. At 95% confidence this covers the range 11.5 - 13.5% which would seem to cover the possible range. The β dose rate from the potassium within the grains can be calculated using the table of Mejdahl (1979).

The two low values of 6.5 and 8.4% K in the table are cause for concern. If we assume that the K analyses are correct they indicate that for some sediments the K-feldspars are not 13% K; for such samples one will have to assume the relevant K content could lie anywhere between the determined value and 14%, but recognize that most of the luminescence could still be from feldspars with ~ 13% K.

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Reviewer
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Comments

The paper deals with the intricate problem of determining the correct K concentration in K and Na feldspar when the separation of feldspars from other minerals, especially quartz, is incomplete. The procedure described is a mapping of minerals in five groups: SiO_2, K-feldspar, Na-feldspar, mixed K + Na, other (Si and Al) by means of a scanning electron microscope.

This method seems to work well and as a result of their discussion the authors conclude that a typical, suitably conservative estimate of the K content in K-feldspar would be 12.5±0.5% ;