

such as (for example) P^{5+} serving as a 'carrier' for Al^{3+} and together proxying for $2Si^{4+}$. It may be implied that Be^{2+} could accept interstitial sites, which is feasible, but it would be much less likely that Mn^{2+} would do so.

Energetically, what appears to be most likely is that a stuffed derivative is formed rather than adding ions in interstitial sites. Thus, Be^{2+IV} replaces Si^{4+IV} with Mn^{2+} , Fe^{2+} and Fe^{3+} being added in the stuffing site to balance the loss of charge.

Alternatively, Fe^{3+} , Be^{2+} for Si^{4+} with Mn^{2+} , Fe^{2+} (and remaining Fe^{3+} if any) in the stuffing site.

If Fe^{2+} were in lattice sites rather than Fe^{3+} it would require that twice as many total charges be realized by the stuffing ions. In fact, the analysis of Fig. 5 cannot be quantitatively explained on the basis of any of these schemes, since the atomic concentration of Be is too high to be compensated for by putting all the iron and manganese in the stuffing site. If this amount of Be^{2+} is actually incorporated, almost certainly one should detect by infra-red spectroscopy appreciable $(OH)^-$ proxying for O^{2-} .

The absorption due to Fe^{3+} , Fe^{2+} or Mn^{2+} in the stuffing site will, of course, not be the same as the absorption in other commoner crystal fields and hence one cannot extrapolate from the color of known compounds to the 'color' of an ion in an interstitial or stuffing site.

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THERMOLUMINESCENCE MEASUREMENTS WITH RAPID HEATING

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Recent papers in the *American Mineralogist* have described refined thermoluminescence measuring apparatus for minerals (Ashby and Kellagher, 1958; Lewis, *et al.*, 1959). The heating rate used for these is about $2^\circ C.$ per second or less. Slow heating rates were first used in this laboratory (Saunders, 1953), but a fast heating instrument has been found to be more sensitive to low levels of thermoluminescence and increases the number of determinations which can be made per hour. A consequence of the fast-heat feature, however, is a decrease in temperature control and precision. Houtermans, *et al.* (1957) has also described a fast heating thermoluminescence instrument and discusses its relative advantages.

The apparatus described here normally employs a heating rate of 20

to 30° C. per second, but if required, heating rates of 90° C. per second may be attained. Although the total number of thermoluminescence photons is independent of the rate of heating, a fast heating rate releases more photons per second, thus leading to a more easily detected, intense light. The time of heating is only a few seconds and the whole

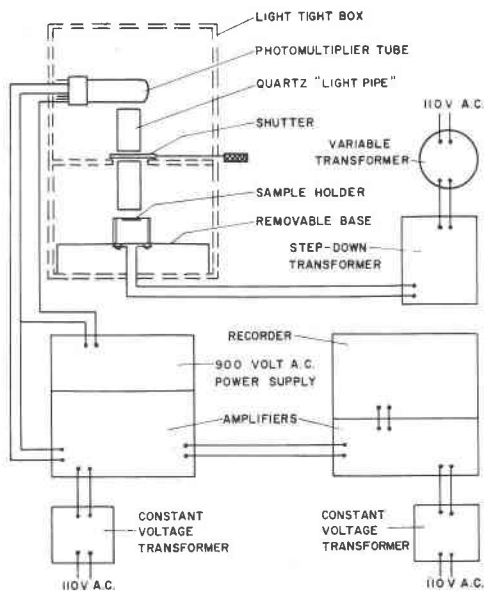


FIG. 1. Fast-heat thermoluminescence measuring device.

operation can be completed in about 5 minutes per sample, including grinding, loading, heating and cooling.

The apparatus is shown in Fig. 1 (Ammentorp, 1957). The heater is a strip of nichrome ribbon $1\frac{1}{2}$ inches long, $1\frac{1}{4}$ inches wide and 0.027 inch thick, mounted with spring tension on a transite base. Above it is a vertical light pipe of quartz provided with a shutter. A photomultiplier tube (1P21) receives thermoluminescent light emitted by heated crystals and the current is amplified with a circuit designed by Professor Robert J. Parent and built by Mr. Donald M. Pryor in this laboratory (Fig. 2).

The photoelectric current is recorded on a rapid recorder, Sanborn Model 127, equipped with a built-in D.C. amplifier Model 126B. Paper speed is 25 mm. per second and the light intensity curve is traced by a heated stylus on color-changing paper. The light intensity-temperature (time) graph is the "glow curve."

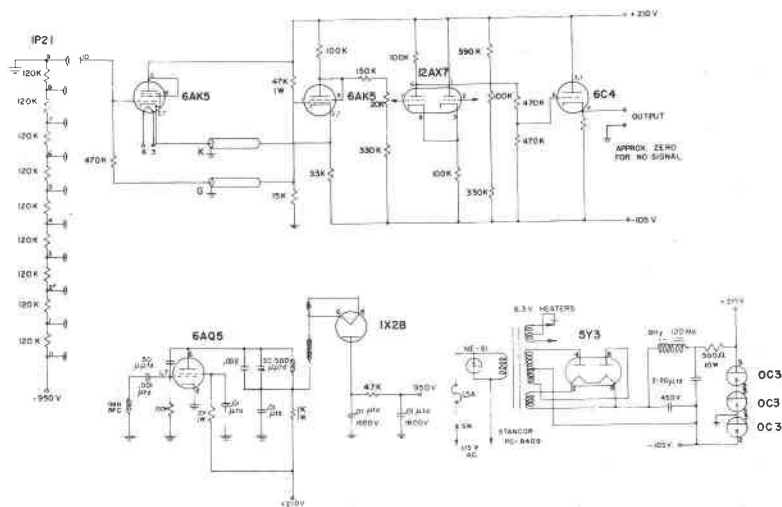


FIG. 2. Photomultiplier and amplifier.

The nichrome heater is supplied power through a 1.5 kilowatt step down transformer and the heating rate is controlled with a Variac. Temperatures from the start of heating are determined by using heat-sensitive crayons. A curve of temperature is then plotted against time for each heater power setting. The calibration curves are near linear. After red heat has been reached, power is cut off and the nichrome heating ribbon is quickly cooled by an air blast or with dry ice, ready for the next sample.

Sensitivity to thermoluminescence may also be controlled by the area and volume of sample exposed to the photomultiplier tube. A simple control technique is to pack a powdered sample (100–200 Tyler) into shallow ring molds. Compaction of the powder against the sides of the ring holds the sample in place and permits transportation of the sample for weighing and loading onto the furnace. The molds make uniform sample discs with one side in intimate contact with heating elements and the other side exposed to the photomultiplier. Sample volume, geometry and heating environment may be varied according to the dimensions of the ring mold.

By adjusting sample size and/or regulating the heating rate a wide range of thermoluminescent intensities may be measured and recorded. The effect of faster heating on improving instrumental sensitivity is shown in Fig. 3.

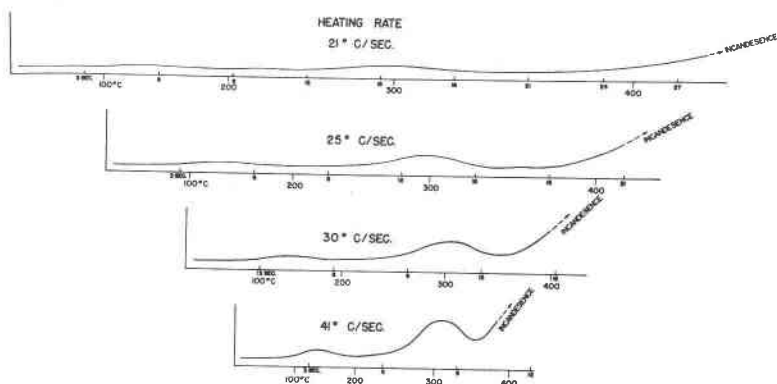


FIG. 3. Resolution of low level thermoluminescence in NaCl by fast heating.

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ADSORPTION OF ETHYLENE GLYCOL AND GLYCEROL BY MONTMORILLONITE*

(Clay-Organic Studies, Part V†)

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The swelling of montmorillonite immersed in ethylene glycol or in glycerol has been used for many years to identify montmorillonite in

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† Part IV. See Hoffmann and Brindley (1961).