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THERMOLUMINESCENCE OF ROCKS AND MINERALS

PART I. AN APPARATUS FOR QUANTITATIVE MEASUREMENT

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Abstract

Equipment which permits quantitative recording of the thermoluminescence emitted by samples with a high degree of reproducibility and sensitivity has been developed. The equipment operates over the temperature range from 25° C. to 600° C. at rates from 10° C. per minute to 100° C. per minute. The apparatus has high light-detection sensitivity and broad spectral response. The construction combines reliability and flexibility with simplicity of operation. Not more than 50 milligrams of sample is ordinarily required.

An analysis of the instrumental and physical factors which determine the precision of making glow curves is made to evaluate the requirements for each section of the apparatus.

INTRODUCTION

Measurements of thermoluminescence have been made for several purposes, such as fundamental physical studies (Halperin, Braner, and Alexander, 1957), radiation dosimetry (Daniels, Boyd, and Saunders, 1953), studies of meteorites (Houtermans, Jäger, Schön, and Stauffer, 1957), applications to stratigraphy (Parks, 1953; Saunders, 1953; Bergstrom, 1956; Pitrat, 1956), age estimation in sediments (Zeller, Wray, and Daniels, 1957), and the identification or determination of certain minerals (Lewis, 1956). The demands of each application require equipment designed to meet the specific needs of the problems for which the thermoluminescence data are to be used. The equipment described in this paper has been designed to measure and record the thermoluminescence of rocks and minerals with sufficient accuracy and repeatability that records may be quantitatively compared over long periods of time. Operation of this equipment has been made as simple and convenient as possible, consistent with the other requirements of the measurements. The equipment is the culmination of several years' experience in measuring the thermoluminescence of natural samples.

The present equipment measures the thermoluminescence of a powdered sample of not more than 50 mg. over the temperature range from 25° C. (room temperature) to 600° C. The rate of temperature rise of the sample is constant over the operating temperature range and may be varied from 10° C. to 100° C. per minute in steps of 10° C. per minute.

The interrelation of the various sections of the equipment is shown in the block diagram (Fig. 1). The constancy of rate of temperature in-

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crease is accurately maintained by continuous comparison in the program controller of the voltage produced by a thermocouple in the sample heater with a prescribed voltage which corresponds to an exactly constant rate. Any deviation of sample temperature from what has been prescribed at a given instant will modify the amount of heater power which is supplied by the magnetic amplifier.

This method of controlling the heating rate of a sample has been found generally useful and has been adapted to other applications, such as programing temperatures of samples in x-ray diffraction studies (Rowland, Weiss, and Lewis, 1959).



FIG. 1. Block diagram of thermoluminescence equipment showing functional interrelation of various sections.

The glow of the thermoluminescence is detected by a photomultiplier tube operated so that the output current is accurately proportional to the light intensity. Since the intensity of thermoluminescence varies greatly from sample to sample, this detector must operate from the lowest possible levels of light intensity up to the highest levels at which the current output is still proportional to intensity. The limit of low-level operation is determined by the dark current, which is principally produced by thermally liberated electrons in the photomultiplier tube. This dark current is reduced substantially by keeping the tube at -80° C., the temperature of dry ice. By operating in this manner, a range of light intensities of a million to one can be covered quantitatively. The photomultiplier type selected has the broadest spectral response available, since the color of the thermoluminescence varies considerably from one type of mineral to another.

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The current from the photomultiplier tube is amplified to a convenient level for recording on a strip-chart recorder. This equipment uses an X-Y recorder indicating light intensity as a function of temperature.

DISCUSSION OF PHYSICAL PRINCIPLES OF THERMOLUMINESCENCE Nature of Thermoluminescence (Garlick, 1949)

When an alpha particle, gamma ray, or other high-energy radiation passes through matter, it transfers some of its energy to this matter by



FIG. 2. Typical thermoluminescence glow curve of calcite following gamma irradiation with Cobalt-60. Heating rate 80° C. per minute. Light intensity in arbitrary units.

producing clouds of excited electrons which are dissociated from their original atoms. In metals and semiconductors, these electrons have high mobility in the crystal and dissipate their excess energy as heat. In insulating crystals, however, some of these excited electrons become localized in the crystal at structural defects. If the temperature of the crystal is raised, the probability of escape increases rapidly. As these trapped electrons are released, some of them drop to lower energies by emitting a photon of light. This light emission produced by thermal liberation of stored electrons is called thermoluminescence.

If a sample containing trapped electrons is heated, light is emitted in a series of discrete maxima as the sample temperature rises. Each maxi-

mum in the light intensity corresponds to a certain energy of trapped electrons and is related to the detailed structure of the crystal. A curve of the thermoluminescence light intensity as a function of the sample temperature is called a glow curve. A typical glow curve for calcite following gamma irradiation in the laboratory is shown in Fig. 2. Samples are irradiated in a cylindrical cobalt-60 gamma ray source as previously described (Lewis, 1956; Saunders, Morehead, and Daniels, 1953).

Glow-Curve Equation

The shape of a glow curve can be described mathematically if certain simplifying assumptions are made as follows:

1) The rate of release of trapped electrons is proportional to the number of electrons present.

2) The electrons giving rise to one particular glow-curve peak all have the same energy.

3) When a trapped electron has been released, the probability of its returning to a lower energy is much greater than the probability of its being retrapped.

If any of these assumptions is changed, a different mathematical expression will be necessary to describe the glow curve. However, these assumptions are physically reasonable in many systems and lead to the following equation for each glow-curve peak:

$$I(T) = Cn_0 s \exp\left[-\int_0^T (s/\beta) e^{-E/kT} dT\right] e^{-E/kT}$$
(1)

where

I = thermoluminescence light intensity

T = absolute temperature, degrees Kelvin

C = geometrical constant

 n_0 = initial number of trapped electrons

s =frequency factor in seconds⁻¹

 $\beta =$ the heating rate, $\beta = dT/dt$, degrees per second

E =trap energy of electrons, electron volts

 $k = \text{Boltzman's constant}, 8.62 \times 10^{-5} \text{ ev/degree}$

In order to be able to solve this equation, it is necessary to know how the heating rate, β , depends on temperature. The simplest condition is that of constant heating rate, or $\beta = dT/dt = \text{constant}$. Since the intensity depends exponentially on the heating rate, small changes in heating rate will distort the glow curve. For the present equipment a departure of 2° C. from the prescribed ideal linear program is the maximum allowable deviation.

The range of heating rates was selected such that for the size of sample particle regularly used for these studies (40 mesh or finer) and for the chemical compositions normally encountered (mainly ionic solids) there should not be a temperature spread of more than 2° C. from the top to the bottom of the sample. The maximum rate of 100° C. per minute is within this range and establishes the power requirements of the magnetic amplifier which supplies current to the sample heater.

The temperature range of interest can include the lowest temperatures at which experimental work could be done up to that at which the incandescence of the sample masks any luminescence. Since this equipment is largely directed toward the natural thermoluminescence of samples which have been at geologic formation temperatures for long times, room temperature is a convenient lower limit. The temperature at which the incandescence of the heater dominates the glow of the system depends on the sensitivity at which the detector is operated. This always occurs before 600° C. which fixes the upper limit of operation.

The intensity of the emitted thermoluminescence depends on the following factors, as described by equation (1):

1) The number, n_0 , of electrons trapped in the sample initially. This will in turn depend on the number and kind of crystalline defects in the sample, the amount of high-energy radiation which has been absorbed, and the temperature at which the sample has been maintained.

2) The heating rate. In general, the higher the heating rate the higher the peak height. The area of the peak, however, is constant at various heating rates. The limitation on the heating rate is usually determined by the problem of making the temperature within each crystal of the sample follow sufficiently close to the temperature of the heater. For measurements where the primary consideration is to obtain a maximum signalto-noise ratio, very high heating rates such as 50 degrees per second have been used.

3) The geometrical constant, C. This number includes not only the solid angle of light collection and losses due to multiple internal reflections in small particles, but also the internal absorption due to the color of the sample.

Experimental Considerations

The indicated intensity of thermoluminescence, in addition to these factors, depends on the spectral response of the detector photomultiplier for the particular color of the glow. The glow from minerals of various types covers at least the entire range of the visible spectrum, so that the photomultiplier should be selected whose spectral response most closely matches the spectrum of the samples being studied. For example, thermoluminescence from calcites and dolomites lies in the orange-red region of the spectrum. Measurements on these samples should, therefore, use a photomultiplier with a high sensitivity to red colors.

The intensity of the glow of minerals when heated varies enormously

from the lowest levels of detectability to brilliant glows a million times more intense. As an average for qualitative comparison, a surface brightness of about 10 microlamberts is a typical value for many minerals.

Sources of Error

Sampling

With any natural materials, variations in composition and structure will be reflected as variations in thermoluminescence. This requires that samples be thoroughly blended and quartered if ground materials are used. This is probably the largest single source of variation in the quantitative study of thermoluminescence.

Weighing

In the range of sample weights used in this apparatus, the amount of thermoluminescence produced is directly proportional to the weight of the sample. For quantitative work, all samples are accurately weighed on a torsion-type microbalance which can be read directly to 0.2 mg. At the usual sample weight of 50 mg., this introduces an uncertainty of $\pm 0.4\%$.

Geometrical

This source of variation arises from the fact that light-collection efficiency depends on the mechanical distribution of the sample in the heater. To reduce this source of uncertainty, particle size and sample weights have been selected to insure a sample thickness of one particle layer. As long as the strict proportionality between sample weight and amount of thermoluminescence is maintained, the contribution of geometrical errors is insignificant.

Instrumental

The contributions of the individual portions of the electronics to the variations in measured thermoluminescence are estimated as each section of the equipment is discussed. In general, however, the stability of the electronics is as good as required to make the contribution of the electronics small compared to other sources of error.

HEATING-RATE

General Considerations

The heating-rate section must increase the sample temperature along a prescribed path at the rates and with the repeatability required by the use which is to be made of the experimental data. The factors which must be considered for a constant rate of temperature rise are (1) range of heating rates, (2) reproducibility of average heating rate, (3) allowable variations in instantaneous heating rate, and (4) constancy of temperature rise over operating temperature range.

A brief examination of the fundamental physical principles involved will aid in understanding the requirements for these various factors.

The glow curve is described analytically by equation (1). From this equation it can be demonstrated that the temperature at which a peak of the glow curve appears depends strongly upon the heating rate. As an example, for typical dolomite and calcite samples, a change of 2 degrees per minute in the average heating rate results in a shift of about 5 degrees in the location of these peaks. From equation (1) it can also be shown that variations in the instantaneous heating rate cause pronounced fluctuations in the intensity of the emitted thermoluminescence.



FIG. 3. Distortion of glow curve produced by small fluctuations deliberately introduced in the heating rate. Normal glow curve of same sample with constant heating rate shown for comparison.

As the heating rate, β , increases, traps are emptied at a greater rate and the intensity, I, of the glow increases. The subsequent drop in instantaneous heating rate reduces the rate at which traps are emptied and results in a low intensity. This effect is more pronounced at low heating rate (see Fig. 3).

As the heating rate increases, the thermal gradient in the sample also increases. To limit this temperature differential within the sample to less than 2° C., the heating rate is limited to 100° C. per minute. In this equipment, ten heating rates are provided ranging from 10° C. to 100° C. per minute. The operating temperature range is from 25° C. to approximately 600° C.

For fulfilling these requirements, the general approach to the design of equipment is to use a high-gain electrical-thermal feedback system. High sensitivity and power gain are provided by a vacuum-tube amplifier driving a two-stage magnetic amplifier. The basic plan of the heating channel is shown in Fig. 4.

Temperature-Sensing Element

For optimum usefulness in this system, the temperature-sensing element should have the following output characteristics:

- 1) Produced as an electromotive force.
- 2) High sensitivity to temperature.
- 3) Electromotive force linear with temperature over operating range.
- 4) Rapid thermal response.

The chromel-alumel thermocouple very nearly satisfies all these requirements. It has an average sensitivity of 41.5 microvolts per degree centigrade over the entire operating range; the sensitivity never departs from this value by more than 2 per cent. Excellent thermal response is obtained by imbedding the separate thermocouple leads on opposite sides of a silver block on which the sample is heated and across which there is a negligible temperature gradient.

Program Controller

The function of the program controller unit is to develop a voltage which increases linearly with time and corresponds in magnitude to the thermocouple voltage desired at each instant. This voltage is compared continuously to the actual thermocouple voltage, and this difference or error signal is used to control the instantaneous power to the heating coils.

The thermocouple voltage is also used as the input to the Y axis chart drive of the recorder to produce a deflection proportional to the sample temperature.





The linear increase of voltage is obtained by using a small synchronous motor to drive a precision potentiometer, the output voltage of which is proportional to time. The various heating rates are selected by switching different resistors in series with the heating rate potentiometer to change the voltage across its terminals. This produces a proportional change in the rate at which the control voltage raises. All of the components of this section are selected for long-term stability.

The precision with which a particular heating rate is programed depends upon both the precision with which the potentiometer voltage can be established and any fluctuations in the speed of the synchronous motor. A standard reference cell is provided to permit precise measurement of the voltage supplied to the potentiometer. When operated from normal commercial electric power sources, the synchronous motor will not ordinarily introduce an uncertainty of more than 0.05 per cent in the heating rate.

Error-Sensing Amplifier

The difference voltage or error signal which results when the thermocouple output differs from the program controller voltage is supplied to a high-gain, chopper-stabilized amplifier.* The output circuit of this amplifier has been modified to provide a direct current suitable for control of the magnetic amplifier. The amplifier produces about 100 microamperes per microvolt input which corresponds to about 4 microamperes for 0.001° C. equivalent error signal.

Magnetic Amplifiers

The error-sensing amplifier drives the low-level magnetic amplifier which, in turn, supplies control current for the high-level magnetic amplifier. The low-level unit has a current gain of about 750. The overall gain of the two magnetic amplifiers together is such that a temperature error of 0.001° C. results in a change of approximately 15 watts power to the heater coils. The rated maximum output is 280 watts into a 12-ohm heater coil. The system saturates so that the maximum power output obtained in use is about 350 watts.

Heater

There are four basic requirements to be met by the heater used in this thermoluminescence equipment:

- 1) Even heating of sample.
- 2) Constant brightness of heater cup surface.

* Minneapolis-Honeywell Brown Electronik® Amplifier Type 3563581 modified as shown in Reference Drawing 21-G-95, MH Instruction 6002.

3) Small differences in temperature within the heater.

4) Electrical characteristics of the heater which match available power sources.

In the design of the heater, several types of construction were considered. At one extreme is the massive heater with a long thermal time constant. By supplying sufficient power to this block, the temperature can be raised linearly and minor variations will be smoothed by the thermal inertia. However, power demands are great for such a system. At the other extreme is the heater with the smallest possible thermal time



FIG. 5. Cutaway drawing showing construction details of plug-in heater units. Sample is placed in the well in the center of the heater.

constant. In this case, the temperature responds quickly and consequently makes severe demands on the control system. The heater used lies between these two extremes. This results in a system which responds quickly but also has enough thermal inertia to provide reasonable filtering action. The closer matching of the thermal and electrical time constants renders the amplifier system less sensitive to the choice of the operating point.

Radiation losses have been reduced by a ceramic heat-reflecting shield placed coaxially around the heater block. Without this, power demands become excessive as radiation becomes the significant mode of heat transfer. This radiant shield does not affect the linearity at low temperatures since heat losses here are due mainly to conduction and convection.

Some of the details of construction are shown in Fig. 5. The silver fur-

nace is shown in cutaway section. The two chromel-alumel thermocouple leads are silver-soldered into small holes at the bottom of the furnace. The heater coil is a 13-ohm helix of 26 B&S gage nichrome wire wound on the inside of the radiant shield. The heater box is made of transite. Electrical connections are through silver-plated banana plugs to facilitate removal and substitution of heaters. A close-fitting metal ring is placed around the top of the heater block during operation. This prevents light from the glowing heater coil from reaching the photomultiplier if small cracks develop in the transite top because of continued use.

Performance

The over-all performance of the sample-heating section can be checked by recording the output of the heater thermocouple as a function of time. The graph obtained should be a linearly rising line from which the program linearity heating rate, and reproducibility can readily be determined. Typical operating behavior of this equipment is illustrated in Tables 1 and 2.

THE LIGHT-SIGNAL

General Considerations

The function of the light-signal section is to detect and record the intensity of the light emitted by the heated sample. From repeated glow curves of the same sample, it has been found that better than 5% pre-

Date of Experiment	Heater Number	Observed Heat Rate (°C./min.)	Average Heat Rate (°C./min.)	Maximum Deviation from Lineari ¹ y (°C.)
9- 6-57	110	76.1		±1.8
9- 6-57	110	76.9	}77.5	± 1.5
9-9-57	114	79.9		± 1.2
10- 6-57	114	77.0		±1.8
2-26-58	114	76.7	}78.0	±1.2
2-27-58	113	78.8		± 1.5
2-27-58	113	78.8		± 2.1
2-28-58	110	77.8		±1.8
10-23-58	111	78.4	78.1	±1.7
10-23-58	110	77.6		±1.8
10-23-58	113	78.6		± 1.0
10-23-58	113	77.9		± 1.0

TABLE 1. LONG TERM STABILITY OF AVERAGE HEATING RATE

Nominal Heat Rate (degree/min.)	Actual Heat Rate (°C./min.)	Maximum Deviation from Linearity (°C.)
10	9.8	±0.1
20	19.7	± 1.0
50	49.3	± 1.0
80	78.5	± 1.0
100	96.0	± 1.0

TABLE 2. LINEARITY OF VARIOUS HEATING RATES (The Experimental Values Reported Here Are Typical)

cision can be maintained in the recorded light intensities by proper operator technique. This sets the limit of the precision of the measurements. Any error in measurement introduced by the light electronics is to be less than 1%. Some possible sources of variation are

- 1) Photomultiplier tube gain changes.
- 2) Photomultiplier tube fatigue.
- 3) Variations in phototube dark current.
- 4) Changes in the resistance values in the signal path.
- 5) Instability in the dc amplifier or the X-Y recorder.

Since it is known that the wavelengths of the thermoluminescence of minerals extends at least from the near-ultraviolet through the nearinfrared, it is important to have a detecting element with the broadest possible spectral response. The intensity of the light emitted covers a very wide range and depends upon the characteristics of the sample and on the heating rate.

Block Diagram

The heart of the light-signal channel is the photomultiplier tube which converts the low-level light intensities into electrical current. Thus current is amplified and recorded on the X axis of a strip-chart recorder. Since the dc amplifier is operated at constant gain, a current multiplier is provided to allow the operator to utilize the full width of the strip chart. The Y-deflection is provided by motion of the paper, which is driven by the heater thermocouple voltage. The resulting curve is a plot of light intensity vs. temperature. The temperature scale is independent of the heating rate, which simplifies the intercomparison of the various glow curves. A block diagram of this channel is shown in Fig. 6.

Photomultiplier Application

The very high light sensitivity of photomultiplier tubes has made these measurements possible. These tubes have excellent signal-to-noise

ratio and current multiplication ratios of a million. Since sufficient gain is available in several tube types, the deciding factor in the selection of the 6217 photomultiplier tube was its spectral response. The 6217 tube has an S-10 spectral response which covers the range from about 3000 to 8000 angstroms. This response is essentially flat (90 per cent of maximum) over the wide spectral range from about 3700 Å (violet) to 5700 Å (yellow), and has good response (above 20 per cent of maximum) from 3100 Å (ultraviolet) to 6700 Å (red).

For those samples whose thermoluminescence spectra are the same at all temperatures, such as limestones and dolomites, the measurements are proportional to the light intensity during the entire glow curve. For samples such as fluorites which may have marked changes in color of emitted light with changing temperature, rough spectra may be obtained by using either narrow band filters or sharp cut-off filters placed between



FIG. 6. Block diagram of the light-measuring section of the thermoluminescence equipment showing the interrelation of major operational units.

the sample and the photomultiplier (Fig. 7). An ingenious arrangement of filters to obtain a constant photomultiplier response independent of wavelength of the light has recently been described by Luchner (1957). . In the application of a photomultiplier tube to the detection of lowlevel light signals, the ultimate limitation is thermionic emission. This arises from the fact that surfaces which are good photo emitters are also good thermionic emitters. This dark current can be greatly reduced by cooling the photocathode (Boeschoten, Milatz, and Smit, 1954; Engstrom, 1947) by chilling the phototube to approximately -80° C. with a solution of dry ice and acetone. In order to reduce the temperature gradient between the hot heater and the cold photocathode and to increase the light-collection efficiency, a Lucite light pipe is used (Harris and Bell, 1956). The light-collection geometry and the cooling system are shown in Fig. 7. The outside of the light pipe is mirrored with aluminum and then painted flat black to prevent extraneous light from reaching the photomultiplier. This also prevents changes in the light-transmitting efficiency of the light pipe should water condense on the cooled

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lucite during periods of high relative humidity. This coating reduced the light pipe efficiency approximately 15 per cent from its maximum, but since the addition of the light pipe more than doubled the lightcollection efficiency this loss was considered negligible.

Photomultiplier Gain Variations

The current amplification in a photomultiplier tube depends to a large extent upon the voltage per stage. Even slight changes in the applied voltage will result in large changes in output. For example, a 1% change in the supply voltage results in a 7% change in output current. In this equipment the high-voltage power supply is electronically regulated in addition to being operated from a regulated ac voltage source. This



FIG. 7. Cross section of the furnace section. The photomultiplier tube is insulated thermally from the heater and coupled optically by means of a lucite light pipe.

combination supplies high voltage which varies less than 0.01% for 100% load variation and ac supply voltage change from 105 to 125 volts.

To minimize fatigue effects in the photomultiplier tube, an electrically actuated shutter keeps the photomultiplier tube in darkness except when a sample is inserted. The shutter is open only when the sample is fully inserted. The shutter is operated by solenoids as shown in Fig. 8. The shutter is a three-leaf type and has a $1\frac{3}{4}$ -inch opening. Since the original plastic leaves warped at the high heater temperatures, they were replaced with metal leaves. These leaves were etched from sheet brass so that the edges would be smooth and flat. To reduce the impact on the



FIG. 8. Detail of shutter-operating mechanism. The solenoids are actuated alternately to open and close the shutter.

shutter pin, a resistor is included in the solenoid circuit. Two lamps on the front panel indicate whether the shutter is open or closed. A pushbutton switch is also mounted on the front panel to allow the shutter to be closed when the furnace drawer is closed.

The amplification of the photomultiplier tube does not remain constant, but varies with time. The gain usually decreases with use and may be expected to vary by as much as a factor of two (Engstrom, 1947).

Standardization

To compensate for changes in photomultiplier gain and other component variations in the system, the gain of the light channel is standardized by using a reference light source. With the reference light source exposed to the photomultiplier, the high voltage is adjusted to produce a prescribed recorder deflection at a specified sensitivity setting. The ref-

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erence light source is a phosphor disc which is activated by a small amount of radioactive strontium-90. The brightness of the standard falls off only with the decay of the radioactive material which has a half-life of 25 years. The light source has an initial brightness of approximately 5 microlamberts, which is of the same order as the glow emitted by many samples.* To prevent excitation of the light-source phosphor by room light, it is kept in a light-tight housing, which is mounted on the furnace (Fig. 7). A shutter in the housing which contains the reference light source opens to allow the photomultiplier tube to view the source.

Using a single reference light intensity requires operating over a very wide range of photomultiplier current. An alternative method of adjusting the photomultiplier voltage to the brightness of the sample requires a series of calibrated light intensities for the various operating ranges of photomultiplier voltage. This method may be useful for very bright samples, but it has been found operationally to be simpler to reduce the sample size for samples which are too bright for the usual photomultiplier voltage.

Current Multipliers

The current multiplier is an attenuator which allows the operator to select the signal level which will produce the optimum recorder deflection. Since the range of sensitivities required is large, this attenuator is made in two sections. The Fine Current Multiplier section has a decade range in nine logarithmic steps. The Coarse Current Multiplier section has six decade steps from one to one-hundred thousand. All resistors used in the multipliers are wire-wound to a tolerance of $\pm 0.1\%$.

Direct Current Amplifier

This direct current amplifier used is the chopper-stabilized, nullbalance type with a maximum gain of 10^7 and a minimum measurable current of 10^{-11} ampere.** The accuracy of the amplifier is $\pm 0.5\%$. The maximum output of the amplifier is 10 millivolts for driving the X-input of the recorder.

Output Circuits

An X-input selector switch is included to increase the flexibility of the equipment. One position gives normal linear operation, another permits recording the time integral of the glow curve, and the other positions are left for future developments. The integrating unit produces a curve of

** Leeds and Northrup Company, Type 9836-A.

^{*} Available from United States Radium Corporation, Morristown, N. J.



Fie. 9. Schematic diagram for the light-measuring section of the thermoluminescence equipment.

the total light emitted as a function of time.* It is essentially a threestage operational amplifier integrator. The low drift rate and high inherent stability permit the integration of the electrical signal of glow curves lasting over 6 minutes with less than 1 per cent error.

A complete schematic diagram of the light-signal section is shown in Fig. 9.

SUMMARY AND CONCLUSIONS

From an analysis of the requirements for each of the measuring sections of the equipment and the sources of error, an apparatus has been developed for convenient and reliable recording of thermoluminescence glow curves of rocks and minerals with high sensitivity and reproducibility. This equipment has been in regular operation for more than a year with a continuous record of trustworthy performance.

References

- BERGSTROM, ROBERT E. (1956), Surface correlations of some Pennsylvanian limestones in mid-continent by their thermoluminescence: Bull. Amer. Assoc. of Petroleum Geol. 40, 918-942.
- BOESCHOTEN, F., MILATZ, J. M. W., AND SMIT, C. (1954), The application of (RCA 1P28) photomultiplier tubes to the detection of weak light intensities: *Physica*, **20**, 153.
- DANIELS, FARRINGTON, BOYD, C. A., AND SAUNDERS, D. F. (1953), Thermoluminescence as a research tool: Sci., 117, 343-349.
- ENGSTROM, R. W. (1947), Multiplier photo-tube characteristics: Application to low light levels: J. Opt. Soc. Amer., 37, No. 6, 420-431.
- GARLICK, G. F. J. (1949), Luminescent materials: Oxford University Press, London, Chapter II.
- HALPERIN, A., BRANER, A. A., AND ALEXANDER E. (1957), Thermoluminescence of X-ray colored KCl single crystals: *Phys. Rev.*, 108, 928-931.
- HARRIS, C. C. AND BELL, P. R. (1956), Transmission characteristics of light pipers: Inst. Radio Engr. Trans. Nuclear Sci., NS-3, November 1956.
- HOUTERMANS, E. G., JÄGER, E., SCHÖN, M., AND STAUFFER, H. (1957), Messungen der Thermolumineszenz als Mittel zur Untersuchung der thermischen und der Strahlungsgeschichte von natürlichen Mineralien und Gesteinen: Annalen der Physik, 20, 283– 292.
- LEWIS, D. R. (1956), The thermoluminescence of dolomite and calcite: J. Phys. Chem., 60, 698-701.
- LUCHNER, K. (1957), Über die Thermolumineszinz von natürlichen Flusspat, Zeits. für Physik 149, 435-452.
- PARKS, JAMES M., JR. (1953), Use of thermoluminescence of limestones in subsurface stratigraphy: Bull. Amer. Assoc. of Petroleum Geol., 37, 125-142.
- PITRAT, C. W. (1956), Thermoluminescence of limestones of Mississippian Madison group in Montana and Utah: Bull. Amer. Assoc. of Petroleum Geol., 40, 943-952.
- ROWLAND, R. A., WEISS, E. J., AND LEWIS, D. R. (1959), Apparatus for the oscillatingheating method of X-ray powder diffraction: J. Amer. Ceramic Soc., 42, 133-138.

* The integrator unit was designed by S. Kaufman of the Exploration and Production Division, Shell Development Company, Houston.

- SAUNDERS, DONALD F. (1953), Thermoluminescence and surface correlation of limestones: Bull. Amer. Assoc. of Petroleum Geol., 37, 114-124.
- SAUNDERS, D. F., MOREHEAD, F. F., JR., AND DANIELS, F. (1953), A convenient source of gamma radiation: J. Amer. Chem. Soc., 75, 3096.
- ZELLER, E. J., WRAY, J. L., AND DANIELS F. (1957), Factors in age determination of carbonate sediments by thermoluminescence: Bull. Amer. Assoc. of Petroleum Geol., 41, 121-129.

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