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Use Of The Instrumental Activation Analysis For The Characterization Of The Terrestrial And Extra-Terrestrial Material*

by

M. P. Menon

Introduction

There has been considerable interest in the past several years in developing modern analytical techniques to determine the chemical composition of the terrestrial rock samples, with better precision and accuracy than the conventional methods, for comparison with that of extra-terrestrial material for possible explanations of the latter's origin. For instance a long controversy still exists as to the possible origin of tektites. Schwarcz¹ has proposed that tektites are probably formed by the rapid fusion of the soil during the impact of extra-terrestrial objects on earth. Urey² believes that tektite may have resulted from the impact of comet on earth, while Vaisavsky³ suggests that tektites are ejected material from the moon. However, the oxygen isotope studies of Taylor *et al.*⁴ and the work of Fleischer and others⁵ reveal a similarity between the tektites and natural glasses. Unlike meteorites, knowledge of the chemical composition of tektites is largely based on the conventional methods of analysis.⁶ Chemical composition is at least one of the important criteria which could give new light about the origin of tektites.

Urey⁷ has recently pointed out the superiority of the activation analysis results from meteorites when compared with those previously reported from conventional methods. He has largely used the activation analysis results to make a new table of atomic abundances of most of the elements present in chondritic meteorites. Although chondritic meteorites were believed to be remarkably constant in chemical composition wide variations have been noticed more recently in the analytical results of Fe, Al, Co and some other trace ele-

*This work was performed in Activation Analysis Research Laboratory at Texas A & M University, College Station, Texas.

¹Schwarcz, H. P. (1962), *Nature*, 194, 8.

²Urey, H. C. (1957), *Nature*, 179, 556.

³Vaisavsky, C. M. (1958), *Geochim. et Cosmochim. Acta*, 14, 291.

⁴Taylor, H. P., Jr., and Epstein, S. (1966), *Science*, 153, 173.

⁵Fleischer, R. L., Price, P. B. and Walker, R. M. (1965), *Geochim. et Cosmochim. Acta*, 29, 161.

⁶Barnes, V. E. (1958), *Geochim. et Cosmochim. Acta*, 14, 267.

⁷Urey, H. C. (1964), *Rev. Geophysics*, 2, No. 1, 1.

ments.^{7,8} These variations can either be due to analytical errors inherent in the measurements or due to the real differences in their concentrations because of the inhomogeneity or possible fractionation of certain elements in the sample material. Since there are marked differences between the published concentrations of both the major and minor elements in chondritic meteorites and the solar abundances obtained by Goldberg *et al.*⁹ from astrophysical studies it appears that the analysis should be performed on a large number of samples with improved techniques to determine whether chondrite really represents the primitive solar matter.

Neutron activation technique has also been proposed to study the chemical composition of the lunar surface material by Hislop and Wainerdi.¹⁰ These authors have performed the analysis of five major elements in several granite and basalt samples using 14 MeV neutrons. Activation analysis using different bombarding particles and measurement techniques will undoubtedly be one of the major methods that could be employed for the characterization of the recovered lunar material in the future.

The purpose of this study was to show the advantages of the instrumental activation analysis, using both fast and thermal neutrons, for the characterization of the major and a few minor constituents of chondritic meteorites and tektites for a possible application to the analysis of lunar material. No attempt has been made to do the analysis of a large number of samples for a meaningful geochemical study. However, such an analysis of a larger number of samples in a short time is quite feasible with the automated activation analysis systems developed by Wainerdi *et al.*¹¹ Since the technique is non-destructive, it will permit inter-laboratory comparison of the results on the same sample. The availability of the high resolution Germanium-Lithium detectors for gamma ray spectrometry makes it possible to improve the analytical results as well as to increase the number of elements detected with purely instrumental techniques.

Experimental Methods

Description of Samples

The tektite and meteorites used for this study were obtained from Wards Natural Science Establishment, Inc., Rochester, New York. The description of the samples quoted by the suppliers is as follows:

Tektite: Bohemia, translucent green

Siderolite: Pallasite Brenham, Kiowa County, Kansas; Olivine Stony-iron (thick block)

⁸Fisher, D. E. and Currie, R. L. (1965), Proc. Symp., Salzburg "Radiochemical Methods of Analysis," Salzburg, Vol. I, 217.

⁹Goldberg, E., Mueller, E. A. and Aller, L. H. (1960), Astrophys. J. Suppl. Ser., 5, 1.

¹⁰Hislop, J. S. and Wainerdi, R. E. (1967), Anal. Chem. 39, 29A.

¹¹Wainerdi, R. E., Fite, L. E., Gibbons, D., Wilkins, W. W., Jimenez, P. and Drew, D. (1965), Proc. Symp. Salzburg, "Radiochemical Methods of Analysis," Vol. II, 149.

Aerolite: Potter, Cheyenne County, Nebraska; Grey brecciated chondrite (small fragments)

Aerolite: Willowdale, Kingman County, Kansas; Grey brecciated chondrite (small fragments)

The above samples were pulverized separately in a clean grinding mill and several 0.5 - 2.5 g size material from each sample were enclosed in polyethylene vials and heat sealed. Particular care has been taken to see that these samples were not contaminated during their preparation.

14 MeV Neutron Activation Analysis

The major elements, namely, oxygen, silicon, aluminum, magnesium and iron were determined using a 150 KV Cockcroft-Walton accelerator and Mark III automated activation analysis system which have been described elsewhere.¹² The useable flux that is available from this machine is $\sim 10^9$ n/cm²/sec although a slightly higher flux for short irradiations may be obtained. Rapid determinations of oxygen in metallic and geological samples using 14 MeV neutrons have been carried out by several investigators in the past.^{10,13,14} This method is based on counting the total gamma ray activity in the energy range of 4.65 - 7.6 MeV resulting from the 7.35 sec ¹⁶N produced by the ¹⁶O(n,p) ¹⁶N reaction. A twenty sec irradiation, 5 sec delay and 20 sec counting times were selected for the oxygen determination in this study. A single channel analyzer, with a dead time of $\sim 2\mu$ sec/count, was used to count the ¹⁶N activity. The standard used for oxygen measurement is reagent grade oxalic acid. Silicon was determined by irradiating the standards (reagent grade silica) and the samples for 1 min and following the decay of the 2.3 min ²⁸Al produced by the ²⁸Si (n,p) ²⁸Al reaction. The activated standards and samples were counted using two 3" x 3" NaI(Tl) detectors and an RIDL 400 channel pulse height analyzer.

Aluminum, iron and magnesium concentrations were measured simultaneously in each sample by irradiating the samples and the respective standards for 5 min and taking several recounts. The nuclear reactions used for the production of the respective activities and the energies of the gamma rays usually chosen for the activity measurement to determine all the above five elements are well known and presented in references 10 and 12. In all irradiations the neutron dose to which each sample and every standard was exposed was measured using a BF₃ neutron detector connected to an RIDL scaler for corrections to be made in the variation of the neutron fluxes. The analyzer data was punched on the paper tape which were subsequently printed on IBM cards.

¹²Cuypers, M. and Cuypers, J. (1966), "Gamma Ray Spectra and Sensitivities for 14 MeV Neutron Activation Analysis," Activation Analysis Research Laboratory, Texas A&M University College Station, Texas.

¹³Pasztor, L. C. and Wood, D. E. (1966), *Talanta*, 13, 389.

¹⁴Vogt, J. R. and Ehmman, W. D. (1965), *Radiochim. Acta*, 4, 24.

Reactor Neutron Activation Analysis

Although by reactor neutron activation analysis many trace elements may be determined in the above samples by a proper choice of irradiation, decay and counting times only the analysis of Fe, Cr and Co was attempted in this study. The samples of different sizes and the appropriate standards were irradiated at a flux of $\sim 10^{10}$ n/cm²/sec for 1 hour and also for 8 hours. These were cooled for several weeks and then counted using a 3" x 3" NaI(Tl) spectrometer as well as a 2 cm³ lithium drifted germanium detector with a resolution of 4.1 KeV for the 1.332 MeV γ -ray of ⁶⁰Co. The data from the Ge(Li) detector was stored in a 3200 channel analyzer and subsequently printed out using a Teletype punch print readout. All the samples and the standards were counted with a registered dead time of 40% or less for the NaI(Tl) spectrometer and 10% or less for the Ge(Li) detector. The influence of the dead time on the shape and the analysis of the solid state detector spectra has been discussed elsewhere.¹⁵

Results and Discussion

The time dependent gamma ray spectra of a typical chondrite sample activated by 14 MeV neutrons are presented in Figure 1. Although silicon was determined separately in all of the samples with an activation time of 1.0 min, it is obvious from these spectra that four of the five major elements can be measured simultaneously in a meteoritic sample using 14 MeV neutron activation technique. Since the activation products from ²⁷Al and ⁵⁶Fe emit gamma rays of identical energies ($\sim .84$ MeV) the individual activities should be resolved by a decay analysis, as is shown in Figure 2, for the measurement of Al and Fe concentrations in the sample. It is quite clear from this figure that the relative abundances of Al and Fe in typical tektite and chondritic meteoritic samples differ significantly.

Figure 3 shows the characteristic spectra of the same tektite and another chondrite activated by reactor neutrons for 8 hours and counted with a NaI(Tl) spectrometer. Although these spectra differ qualitatively in terms of the gamma ray peaks, some of the peaks are not well enough resolved to make a correct estimate of the concentrations of the elements present. However, the use of a high resolution Ge(Li) detector coupled to a 3200 channel analyzer made it possible to resolve most of the gamma ray peaks as shown in Figure 4. Figure 3 and Figure 4 also demonstrate that the analysis of Fe, Co and probably Sc using activation analysis with conventional gamma ray spectrometry may be wrong even though some corrections may have been made for mutual interferences of gamma ray peaks.

The results of the analysis of three chondrites and one tektite using both 14 MeV and reactor neutrons for activation are presented in Table I. In each measurement, with the exception of O, the photopeak

¹⁵Wainerdi, R. E. and Menon, M. P. (1967), Proc. Symp. "Nuclear Activation Techniques in Life Sciences," May 8-13, 1967, Amsterdam (in press).

area of the prominent gamma ray peaks was computed using Covell's method¹⁶ and this activity, after correction for the decay, was compared with that of the standard to estimate the concentrations. The half life of each isotope was checked with a decay curve analysis and corrections were made for the variation in neutron fluxes between the standard and the sample. It is to be pointed out that the analysis, using reactor neutrons, was performed only for a few trace elements but not for all the possible ones using only instrumental techniques. The five major elements, O, Si, Al, Mg and Fe, were determined using 14 MeV neutron activation technique while the rarer elements Cr and Co were measured by the reactor neutron activation analysis. The results of the iron analysis were also checked with the latter procedure. Table I also includes the most recent analytical results for the above elements calculated from the atomic abundances tabulated by Urey⁷ for the L group of chondrites and also for the G-1 granite chosen by Fleischer.¹⁷ The oxygen abundance for the chondrite is not listed in Urey's table, but Wiik's work¹⁸ from which Urey has taken most of the data, indicates that the oxygen abundance is 36.82% by weight for a typical chondrite. Although the present results of the analysis of chondrites are comparable with those reported by Urey, two exceptions may be noticed. In all the three cases, the iron content is very similar, but all the values are slightly lower than the average value for the L group of chondrites chosen by Urey. The magnesium content of at least two chondrites seem to deviate considerably from the average value reported previously. There are indications, however, that fractionation of some of the lithophile elements like Si and Mg takes place in certain type of chondrites.¹⁸ From the results reported by Taylor *et al.*¹⁹ it appears that the concentrations of Si, Al and Mg in tektites vary within the ranges 32.2-37.2%, 5.3-8.2% and 0.8-1.5%, respectively and an inverse relation exists between SiO₂ and other major constituents. Although the present results of analysis of Si and Al fall in this range within the experimental errors, Mg result deviates significantly. Since tektites have not been analyzed so extensively as meteorites using modern analytical techniques, it appears that newer data on tektites of different groups⁵ are needed for a general comparison. The present work on Bohemian Tektite shows that it is nearer to granite than to chondrites in its chemical composition (see Table I and ref. 10).

Various postulates have been advanced about the origin of meteorites and tektites^{1-5,7} and the possible similarity between these and other terrestrial rock samples and lunar surface material in chemical composition. Although none of these hypotheses can be absolutely proved or disproved until an analysis is carried out on the lunar material it is important to have a reliable technique to analyze the lunar material when it becomes available, and good analytical data on the suspected samples for comparison with the composition of this

¹⁶Covell, D. F. (1959), *Anal. Chem.* 31, 1785.

¹⁷Fleischer, M. (1965), *Geochim. et Cosmochim. Acta*, 29, 1263.

¹⁸Wiik, H. B. (1955), *Geochim. et Cosmochim. Acta* 9, 279.

¹⁹Ahrens, L. H. (1964), *Geochim. et Cosmochim. Acta*, 28, 411.

precious material. It appears that the instrumental activation analysis is at least one of the modern techniques which affords tremendous promise for a rapid and non-destructive analysis of most of the important elements and for inter-laboratory comparison of the findings. The automated activation analysis system, known as Mark II, developed by Wainerdi *et al.*¹¹ in this laboratory will be unique in performing a large number of such analyses on geological samples of different origin, including the lunar material, for some of the major and minor elements.

Conclusion

It has been shown that the instrumental activation analysis using both 14 MeV and reactor neutrons and high resolution gamma ray spectrometry yield results comparable with other techniques most of which require the destruction of the sample. All of the available terrestrial and extra-terrestrial samples can be characterized both qualitatively and quantitatively by this technique with less effort and time than some of the other techniques. In view of the existing controversy on the origin of tektites and its possible association with the lunar material³ more information regarding their chemical composition should be obtained using modern analytical techniques.

The present comparative study on the composition of tektite and chondrites together with the previous work reported from this laboratory¹⁰ on granite and basalt using the same technique reveal that the above four geological samples differ significantly in their chemical composition so that a similarity between any of these and the lunar material may be established.

Acknowledgements

The author is grateful to Dr. R. E. Wainerdi, Head of Activation Analysis Research Laboratory of Texas A&M University for his encouragement and also to the other laboratory staff for their assistance for this work.

²⁰Taylor, S. R., Sachs, M. and Cherry, R. D. (1961), *Geochim. et Cosmochim. Acta*, 22, 155.

Table I
Activation Analysis Results of Tektite and Meteorites*

| Sample | O | Si | Al | Mg | Fe | Fe ^a | Cr ^a | Co ^a |
|--------------------------------------|---------------------------|------------------------|-----------------------|-----------------------|------------------------|-----------------|---------------------|---------------------------|
| Tektite, Bohemia | 52.6 ±1.0 ^a | 40.6 ±2.9(3) | 6.7 ±.7(4) | 4.3 ±.4(1) | .83 ±.11(4) | not measured | .014 ±.003(3) | not detected |
| Siderolite (oliuine), Kansas | 33.4 ±1.5(9) | 15.7 ±.8(3) | .32 ±.11(3) | 20.8 ±.6(1) | 19.3 ±1.7(3) | 17.4 ±1.7(2) | .025 ±.004(5) | .025 ±.003(2) |
| Aerolite (Chondrite), Nebraska | 37.8 ±2.0(9) | 22.0 ±2.6(3) | 1.5 ±.1(7) | 14.2 ±.9(1) | 19.7 ±2.0(5) | 17.2 ±2.6(2) | .26 ±.03(5) | .068 ±.004(2) |
| Aerolite (Chondrite), Kansas | 40.5 ±2.0(10) | 19.1 ±2.0(3) | 1.5 ±.2(3) | 9.5 ±.7(1) | 19.0 ±1.5(4) | 20.1 ±2.3(2) | .33 ±.03(3) | .034 ±.002(2) |
| Chondritic Meteorites** | --- | 18.61 | .974-1.153 (1.072) | 14.2-15.48 (14.99) | 20.24-22.61 (21.42) | | .200-.455 (.286) | .039-.074 (.057) |
| G-1, Granite† | 48.5 | 33.87-33.97 (33.92) | 7.43-7.55 (7.48) | .21-.25 (.235) | 1.32-1.37 (1.35) | | --- | .00019-.00025 (.00022) |

* The results are tabulated in Wt. %.

^a These are the results of the reactor neutron activation analysis and the rest from 14 MeV neutron.

^a Integral numbers in the parentheses show the number of measurements made.

** These were calculated from the atomic abundances given in ref. 7 using 39.8% of Si₂ for L group of chondrites.

† The results include the ranges and the average values (in parentheses).

† These were calculated from the analytical results given in ref. 17.

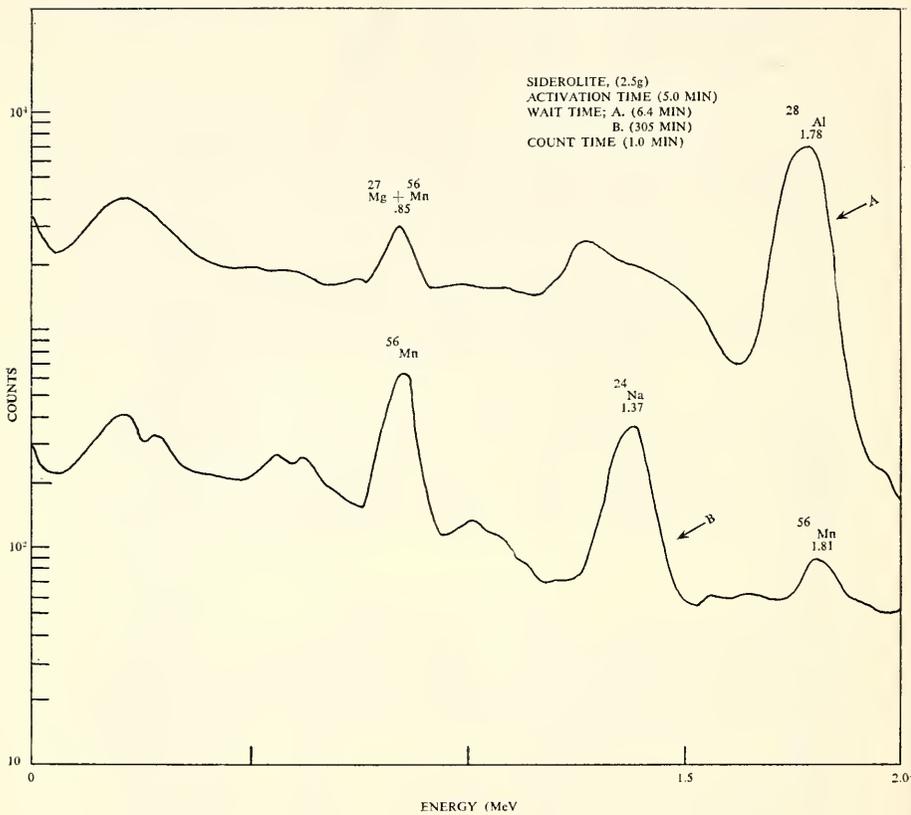


Figure 1. Time dependent spectra of a typical chondrite activated by 14 MeV neutrons.

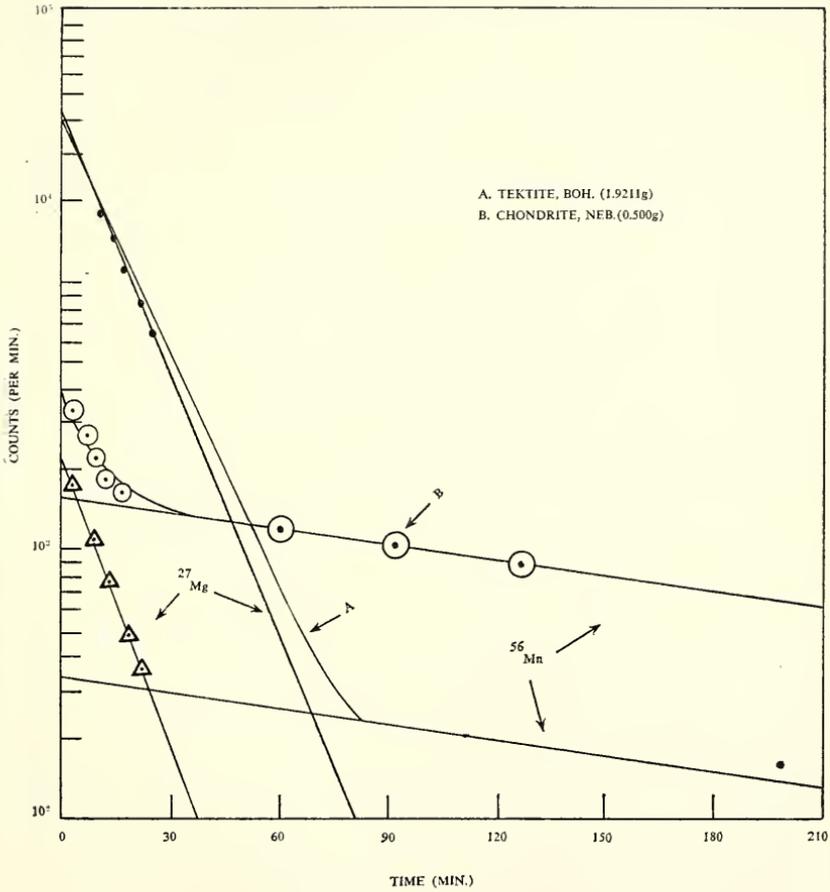


Figure 2. Decay curves of ^{27}Mg and ^{56}Mn in activated tektite and chondrite samples.

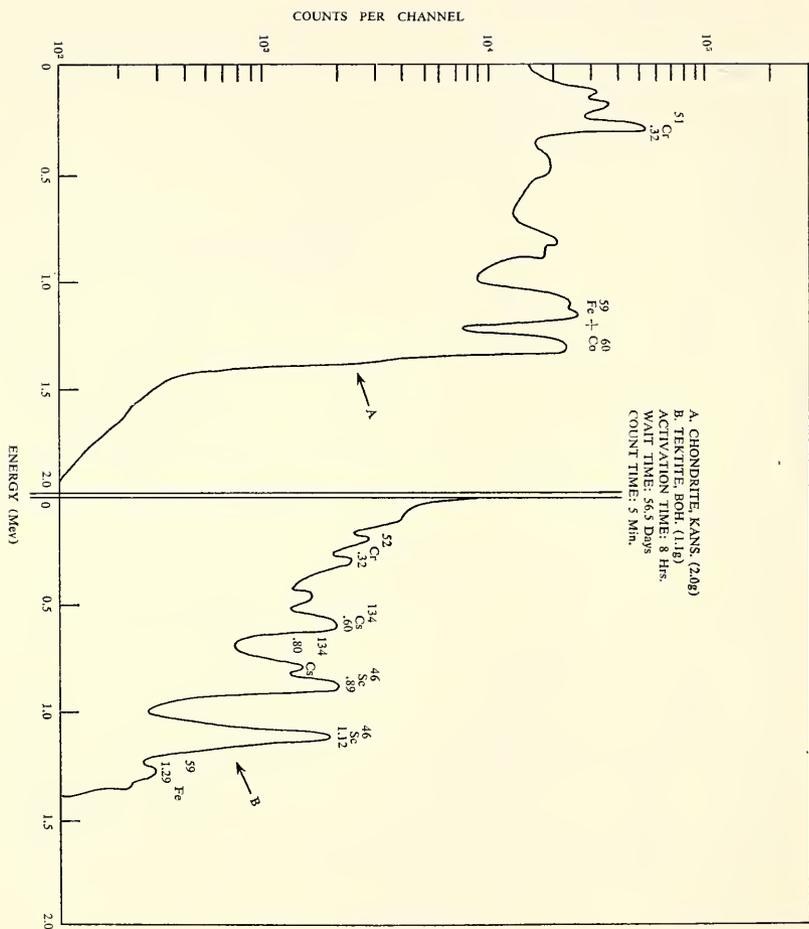


Figure 3. NaI(Tl) spectrometer spectra of the reactor neutron activated tektite and chondrite.

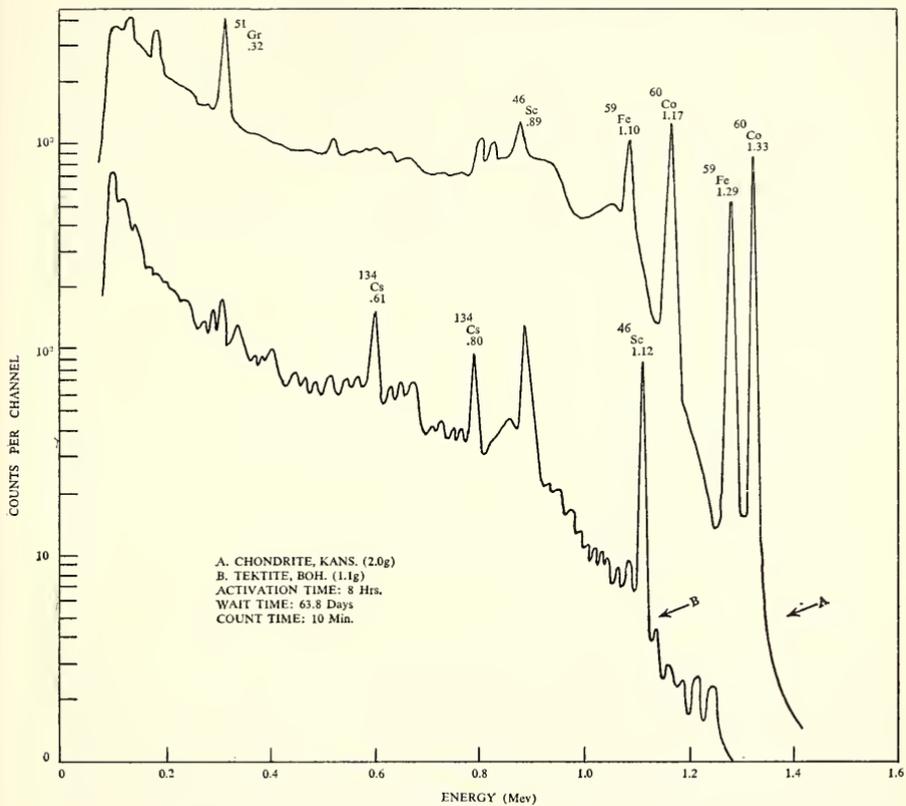


Figure 4. Ge(Li) detector spectra of the reactor neutron activated tektite and chondrite.