LETTERS TO THE EDITOR

MEASUREMENTS OF L-SHELL PHOTOELECTRIC CROSS SECTIONS IN HEAVY ELEMENTS AT 60 keV

In order to fill the void in the existing data[1] on inner shell photoelectric cross sections, measurements of L shell cross sections at 60 keV in elements W, Hg, Tl, Pb, Bi and U have been made. The method used is similar to the methods used earlier [2,3] and consists of measuring the intensity of emitted L-shell fluorescent X-rays with a calibrated proportional counter spectrometer when a target is irradiated with known flux of 60 keV photons obtained from Am^{241}. As discussed in an earlier communication[2], the other low energy photons, *i.e.*, conversion L X-rays in N_p and 26 keV gamma rays emitted from the source were filtered by a suitable graded absorber. As no other experimental data are available[1], the results have been compared with the recent theoretical calculations of Scofield[4] in Table I. The experimental result

TABLE I

Comparison of the present measurements of L-shell photoelectric cross sections at 60 keV *with theoretical calculations of Scofield*

Element	L-shell photoelectric cross sections at 60 keV	
	Present measurements (b/atom)	Theoretical values (b/atom)
W	770±60	760
Hg	1100±80	1050
Tl	1150±80	1110
Pb	1250±90	1170
Bi	1280±90	1230
U	2040±150	1900

are found to agree with theory within errors which are somewhat smaller than those in the earlier measurements[2] at 145 and 279 keV. In the present measurements, in contrast to the earlier measurements, the incident photons are unable to excite the K-shell electrons of the target element and thus there is no contribution to the L shell fluorescent radiation due to the filling of K-shell vacancies by the jump of L-shell electrons. The term

$$\sigma_K f_{KL} \frac{\overline{W}_{KL}}{\overline{W}_L}$$

in equations 1 and 3 of ref. 2 is, therefore, not needed for the evaluation of the present cross sections. Thus the overall error in the final results is reduced.

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ANNEALING CORRECTION TO THE FISSION TRACK AGES OF PHLOGOPITES

THERMAL events occurring in the geological history of a region affect the fission track ages of minerals. Thus a correction to these ages must be applied which in the case of phlogopites (Borra Mines, Visakhapatnam District) has been estimated to be 7%.

In order to apply correction for thermally lowered fission track ages, annealing experiments have been performed by a number of workers[1,2]. The radiation damage in minerals tends to heal during short time high temperature event or a long time anneal at a slightly elevated temperature.

In our present study the phlogopite samples were irradiated with a thermal neutron dose of $1\cdot2\times10^{16}$ (nvt) in CIRUS reactor at B.A.R.C., Trombay. These irradiated samples of known induced track density were heated in a muffle furnace at different constant temperatures for one hour and the reduction in track density and track length with temperature have been observed. The results are summarized in Table I.

It is evident from Table I that the induced fission tracks in phlogopite do not disappear completely for one hour heating even at 800° C. Thus for complete removal of tracks, intense heating is required. Figure 1 shows that the reduction in track density at all temperatures lags behind the reduction in track length·

TABLE I

Annealing data of phlogopites

Heating time 1 hr

Temperature °C	Percentage reduction track length	Percentage reduction track density
200	3·3	Negligible
300	10·3	8·2
400	17·0	10·7
500	27·3	20·6
600	32·8	30·7
700	50·3	49·0
800	57·4	55·0

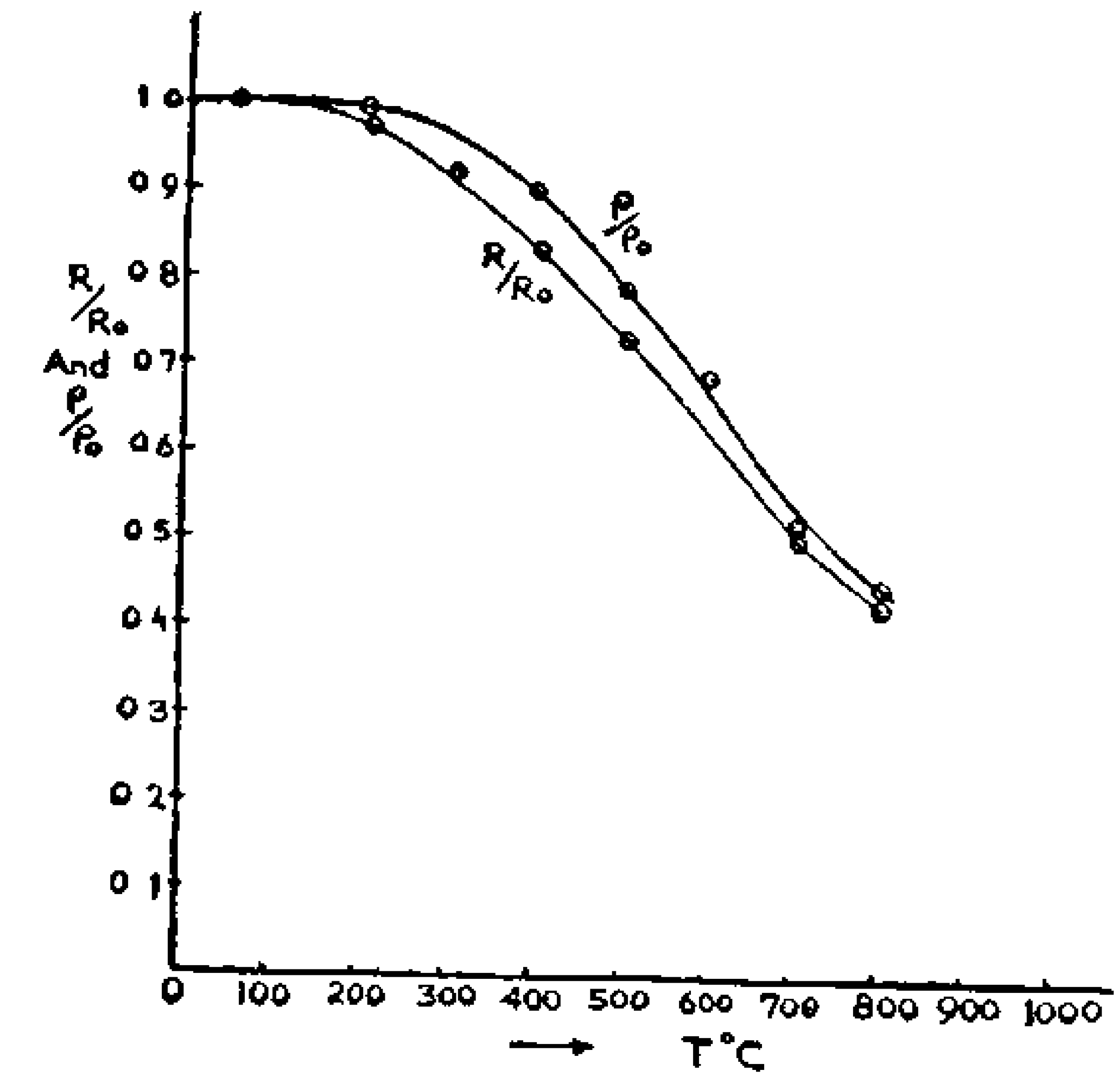
The fission track ages of phlogopites and muscovites have been determined. The experimental details are the same as reported elsewhere[1,3]. The fission track ages are calculated by using the simplified version of the formula[4]

$$T = 6{\cdot}57 \times 10^9 \ln [1 + 9{\cdot}25 \times 10^{-18} \rho_s/\rho_i \times \phi]$$

where ρ_s and ρ_i are the fossil and induced track densities respectively and ϕ is the total thermal neutron dose with which the samples have been irradiated. The fission track ages are given in Table II. The mean age for phlogopites is 491 ± 16 m.y. and that for muscovites is 621 ± 142 m.y. The errors in the results are statistical counting errors. These ages correspond to the last orogenic metamorphic cycle in the geological history of the region.

A linear relation has been observed between track length reduction and track density reduction (Fig. 2). In phlogopite, the mean range of fossil tracks is less than the mean range of induced tracks by 10%. This reduction in track length corresponds to 7% decrease in fossil track density. Hence the correction to fission track ages of phlogopites is also of the same order.

Our age results show an excellent agreement with fission track ages determined by Kere[5] for Gudur biotites and those of Nand Lal *et al.*[6], for apatites of the same region. Thus from the comparative studies on fission track ages of apatite, phlogopite and biotite we conclude that fairly stable thermal conditions have



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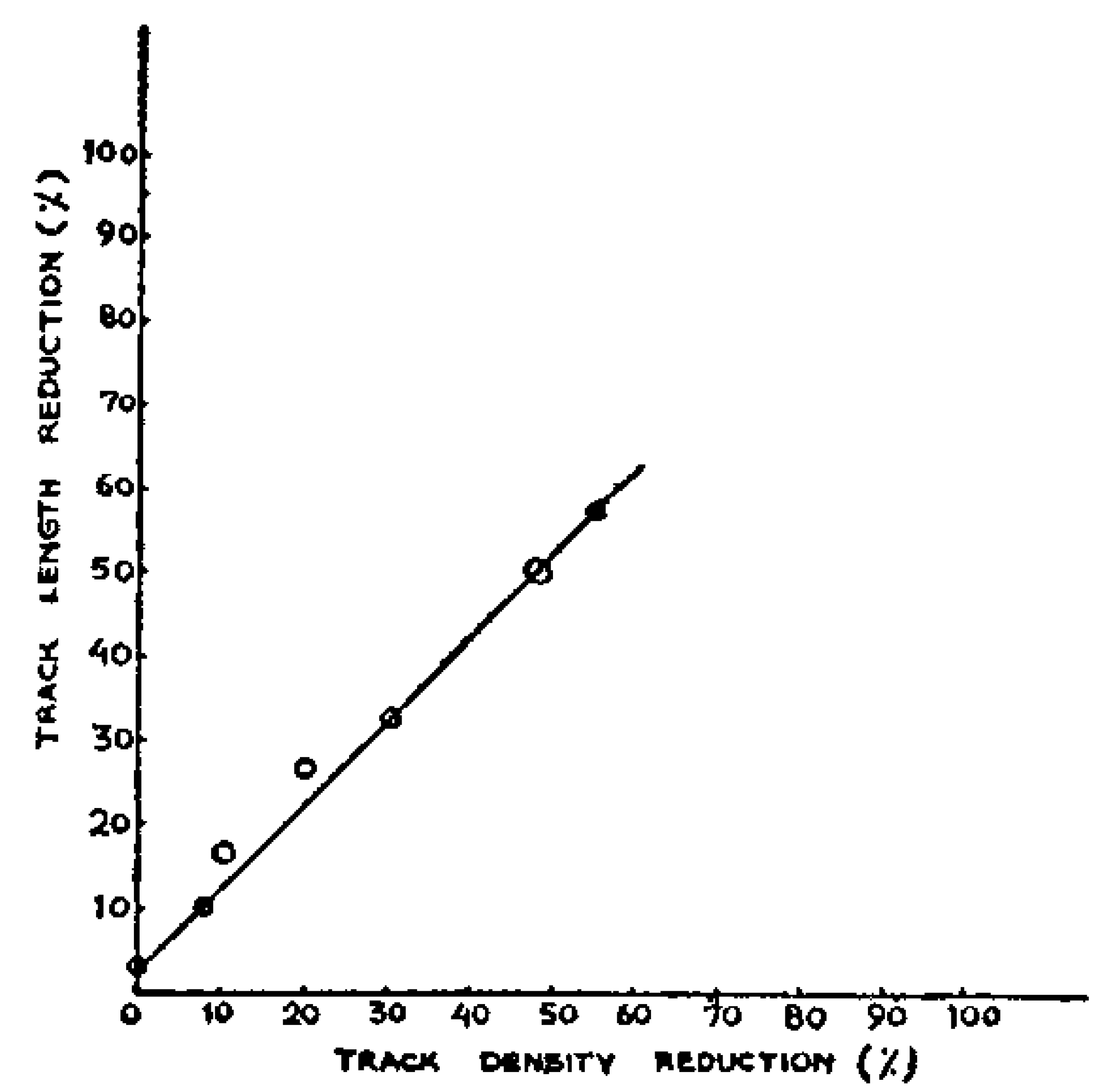

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FIGS. 1–2. Fig. 1. Relation between mean range and track density of induced fission tracks during annealing. Fig. 2. Percentage reduction in track density *versus* percentage reduction in track length.

TABLE II

Fission track ages of some muscovites and phlogopites of Andhra Pradesh

Total neutron dose $\phi = 1{\cdot}2 \times 10^{15}$ (nvt)

Location	Mineral	No. of samples studied	Track density ρ_s/ρ_i	Mean f.t. age (m.y.) T	U. Conc. (atom/atom) $\times 10^{-10}$
Borra Mines	Phlogopite	5	0·70	491±16	79·0
Seetha Rama Mines	Muscovite	5	0·90	621±142	1·2

been prevailing in the East Coast region since the last metamorphic cycle known as Indian Ocean Cycle.

In comparison with our previous studies on the annealing behaviour of muscovite[7] and biotite[8], it has been observed that the correction for loss of tracks by annealing to the fission track ages of phlogopites is slightly less than that for muscovite and biotite.

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A NEW INDICATOR IN THE SEQUENTIAL COMPLEXOMETRIC ANALYSIS OF COPPER AND NICKEL

A NUMBER of reliable analytical procedures for the determination of Cu (II) and Ni (II) are available. There is however no simple volumetric method available for the determination of these metals when present together.

α-mercaptopropionic acid (MPA) forms a violet complex with Cu(II) in the pH range 2·5 and 8[1]. In this paper the application of copper–MPA complex as an indicator in the sequential complexometric estimation of copper and nickel in Cu–Ni alloys is described.

Experimental

Reagent: MPA of analytical grade was used to prepare 1% solution in double distilled water. Stock solutions of copper and nickel (0·1 M) were prepared and analysed by standard methods[2].

Procedure

Aliquots of Cu(II) and Ni(II) solutions containing 3 to 20 mg of the metals were taken in a conical flask. The pH was adjusted between 3 and 4 with phthalate or acetate buffer and two drops of the reagent were added when an intense violet colouration was obtained. The mixture was then titrated against a solution of EDTA of known strength till the colour changed from violet to bluish green. The burette reading was the total reading for Cu(II) and Ni(II).

Again the same aliquots of Cu(II) and Ni(II) solutions were taken in a separate conical flask and the pH of the mixture was adjusted to between 3 and 4 as before. To this was added about two grams of potassium citrate and about two drops of the reagent. The mixture was titrated with the same solution of EDTA till the colour changed from violet to bluish green. The burette reading is for copper only.

Analysis of Copper-Nickel Alloys

In the case of the analysis of copper-nickel alloys known weight of the given alloy was dissolved in nitric acid and the solution prepared in the usual way. The aliquots of the solution can be used for the analysis of copper and nickel as above (Table I).

TABLE I

Analysis of some standard Cu-Ni alloys with MPA as an indicator

Sr. No.	Percentage of metals expected		Percentage of metals found	
	Cu	Ni	Cu	Ni
1.	87·2	11·3	87·0	11·15
2.	85·5	13·5	85·37	13·4
3.	81·0	17·0	81·12	16·85
4.	77·0	21·0	76·82	20·80

It was found that ions like $S_2O_3^{2-}$, NO_2^-, I^-, $Cr_2O_7^{2-}$, MnO_4^- interfere seriously and should be absent, however, ions like Cl^-, Br^-, $B_4O_7^{2-}$ could be tolerated ten times in excess, while ions like SO_3^{2-} and ClO_4^- could be tolerated only five times in excess.

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