

Table 2 Percentage occurrence of the ratio B/ϕ^{\dagger}

Value of B/ϕ^{\dagger}	Occurrence (%)
1 or < 1	2
1.1-1.5	10
1.6-2	10
2.1-2.5	19
2.6-3	16
3.1-3.5	40
> 3.6	3

spread- F (in association with strong specular reflection) is observed at this low latitude station more often than strong spread- F (in association with weak specular reflection). The results of the present investigation agree with those of Krishnamoorthy and Rao⁵ and Klemperer⁸. An attempt to study this aspect in greater detail using more data is under progress.

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THE FORBIDDEN TRANSITION L_1O_1 IN PRASEODYMIUM-59

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ALTHOUGH studies on the L -emission spectrum of praseodymium-59 have earlier been carried out¹⁻⁸ a careful survey reveals many forbidden transitions left

unobserved. It was therefore thought worthwhile to reinvestigate the L -emission spectrum of praseodymium, with special attention directed towards the forbidden transitions.

The experimental set-up and technique were similar to those adopted by Shrivastava *et al.*⁹. The metallic demountable hot cathode x-ray tube, provided with four-faced rotatable anticathode, was operated at 20–22 kV (fullwave rectified) and 5–7 mA. The praseodymium target was prepared by embedding the specpure praseodymium oxide sample (supplied by M/s Johnson Matthey, London) into closely-spaced horizontal grooves cut on the faces of the massive copper anticathode of the demountable x-ray tube (Beaudouin model B-80). First order reflections from (100) and ($\bar{2}01$) sets of planes belonging to the $\langle 010 \rangle$ zone of muscovite mica and yielding a dispersion of about 12 x/mm^{-1} were employed for recording the spectrum on a 40 cm curved crystal spectrograph of transmission type. Using Agfa Curix M 1 x-ray films and with exposures varying from 15–20 hr several spectrograms were obtained. The wavelengths were determined by linear interpolation from the measurements made directly on the negatives with a Carl-Zeiss comparator having a least count of 0.0001 mm.

All our gamma region spectrograms showed the presence of a weak but distinct line very close to and on the long wavelength side of the dipole line $\gamma_{4,4}$ in the praseodymium spectrum. The intensity of this weak line is about one quarter of that of the γ_8 line, as estimated visually. The wavelength of this weak line, as determined taking $\gamma_{4,4}$ and γ_3 lines of praseodymium as the reference lines, has been found to be 1821.5 xu. The line has been assigned to the forbidden transition L_1O_1 in praseodymium. Our observed value of its wavelength is in good agreement with the value calculated for the transition from the energy level tables¹⁰.

A survey of the wavelength tables¹¹ shows that the possible sources of interference with the newly observed line might be ${}^{67}\text{Ho } L\eta$ ($\lambda = 1822.61 \text{ xu}$) or second order reflections of either ${}^{92}\text{U } L\alpha_1$ ($2\lambda = 1817.518 \text{ xu}$) or ${}^{78}\text{Pt } L_1M_5$ ($2\lambda = 1824.4 \text{ xu}$). As none of our spectrograms showed a stronger line of these elements, the possibility of these interferences can be ruled out completely. The fact that the line is present with the same relative intensity in the photographs taken with both the ($\bar{2}01$) and (100) planes eliminates altogether the possibility of its being second order reflections, since the second order reflections from (100) are known to be very weak compared to that from the ($\bar{2}01$) planes. Further, the interference

due to ^{67}Ho L_{η} line can also be ruled out on the ground that no mention is made of holmium in the list of impurities, supplied by the manufacturers (M/s Johnson Matthey, London) for the spectrographically standardised praseodymium oxide sample. The $(\nu/R)^{1/2}$ value for the newly measured line is found to make a nice fit in Moseley plot for L_1O_1 transition (not reproduced here). The existence of the forbidden transition L_1O_1 in the spectrum of praseodymium has therefore been established. It may be mentioned here that the line corresponding to the transition L_1O_1 has been observed¹¹ in the spectra of all the elements from ^{73}Hf to ^{83}Bi and ^{90}Th . Amongst the rare earths, this transition has been reported¹¹ in ^{60}Nd , ^{65}Tb and ^{67}Ho to ^{71}Lu .

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TITRIMETRIC METHOD FOR THE DETERMINATION OF NICOTINOYL HYDRAZINE WITH VANADIUM(V)

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NICOTINOYL and isonicotinoyl hydrazines belong to the family of aroyl hydrazines that find extensive use in chemotherapy. The $-\text{NH.NH}_2$ group in these compounds is highly susceptible for oxidation and quite a few methods¹⁻¹¹ have been reported for the titrimetric determination of isonicotinoyl hydrazine. Similar methods for the determination of nicotinoyl hydrazine are scarce. Rao and Rao¹² reported titrimetric procedures to determine isonicotinoyl hydrazine using vanadium(V) as the oxidising agent. Vulterin¹³ reported the standardisation of nicotinoyl hydrazine in hydrochloric, sulphuric or phosphoric acid media to potentiometric end point using potassium bromate as an oxidant. In this communication, we report the results of our analytical studies on the determination of nicotinoyl hydrazine with vanadium(V) by titrimetric method. Both electrometric and visual indicator methods are employed for the location of the equivalence point.

Reagents: A 0.1 N solution of vanadium(V) was standardised against a standard solution of ferrous ammonium sulphate. A 0.025 M solution of nicotinoyl hydrazine was prepared by dissolving Fluka's pure sample in deionised water and standardised against a standard solution of potassium bromate². A 0.1% solution of osmium tetroxide (Johnson Matthey's London) in 0.1 N sulphuric acid was stored in an amber glass bottle. AnalaR orthophosphoric acid was used throughout this investigation. A 1% solution of diphenylamine in concentrated sulphuric acid (A. R.) and 0.2% solution of barium diphenylamine sulphonate in deionised water were employed. A Toshniwal digital pH meter (CL 44) in combination with platinum and calomel electrodes was used in the electrometric titration.

The control experiments have shown that the potentials at the platinum electrode during the electrometric titration of nicotinoyl hydrazine with vanadium(V) fluctuate and were stabilized in the presence of traces