

be used for comparison of other tRNA structures. For example, on a distance plot of type II tRNAs which have a variable loop with greater number of residues characterised by a classical helix and a loop, a distinct domain would occur between the domains 3 and 4. Such a comparative study would enable elucidation of possible differences in tertiary interactions and polynucleotide backbone folding which may be important in understanding their functional role.

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## DETECTION OF CONTAMINANTS ON SOME SOLID SURFACES BY USING AUGER ELECTRON SPECTROMETER

R. D. LUKTUKU, C. P. GOPALARAMAN AND V. K. ROHATGI\*

*Technical Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India*

### ABSTRACT

Most surface contaminants are elements of low atomic number and hence an indigenously developed Auger electron spectrometer (AES) is used for the detection of surface contaminants. Surface contaminants on stainless steel, reduced lead-glass and aluminium coated phosphor screen are investigated and reported in this paper. Carbon is a major contaminant on the stainless steel surface besides traces of S, Cl, Ar, N and O. Contaminants detected on the reduced lead-glass are Sb, As, Se and In and on the aluminium coated phosphor screen are P and S. The Auger sensitivity factors for pure elements are known but these values for most compounds and for most elements in different chemical environments are not available in the literature. A quantitative estimation of the contaminants is not therefore given.

### INTRODUCTION

THE surface properties are highly dependent on the presence of surface contaminants. The contamination of surfaces can either be due to the effect of environment or processes like diffusion, segregation or migration of the impurities from the bulk of the material itself. We have used an indigenously developed Auger Electron Spectrometer<sup>1,2</sup> (AES) for studying the surfaces of stainless steel, reduced lead glass and aluminium coated phosphor screen. These industrially important samples are chosen for the detection of surface contaminants, with a view to confirming the use of AES in the detection of trace impurities at solid surfaces. The objective of the

work is limited to the detection and identification of contaminants.

### EXPERIMENTAL

Auger spectra are recorded as derivative of the electron energy distribution. Low beam currents are used to avoid the electron beam artifacts at the surfaces investigated. A phase-sensitive synchronous detection (lock-in-amplifier) scheme is employed to record the Auger electron signals. The sensitivity and the time-constant of the lock-in amplifier and the peak-to-peak modulating voltages are optimised to obtain unambiguous derivative Auger peaks. In certain cases, resolution is sacrificed for intensity.

The experiments are performed in a stainless steel UHV system with an operating pressure maintained

\* Plasma Physics Section

usually at  $5 \times 10^{-8}$  torr or better. Residual gas analyses of the UHV chamber have been made under various operating conditions, using a quadrupole mass analyser (M/s. VG make, Q7A). The samples are analyzed without sputter etching the surfaces. However, prior to analysis, the samples were cleaned with acetone.

### RESULTS AND DISCUSSION

Table I summarizes the results of our investigations in the detection and identification of contaminants. Results on stainless steel are discussed here. Auger Spectrum of stainless steel as recorded, is shown in Fig. 1.

Contaminants observed on the surface of the stainless steel sample are S, Cl, C, Ar, N and O. It is inferred that the presence of chlorine on stainless steel surface is not contributed by the UHV system environment as the residual gas analysis of the system does not indicate the presence of chlorine in the vacuum environment. Phosphorus could not be detected, perhaps due to its concentration being below the detection limit (30 ppm) of the instrument. Ar, N and O peaks observed, are due to the deliberate exposure of the sample to a higher pressure ( $1 \times 10^{-6}$  torr) than the operating pressure, for more than 20 hr, the objective being the study of contaminants arising from the environment as well. However, on subsequent

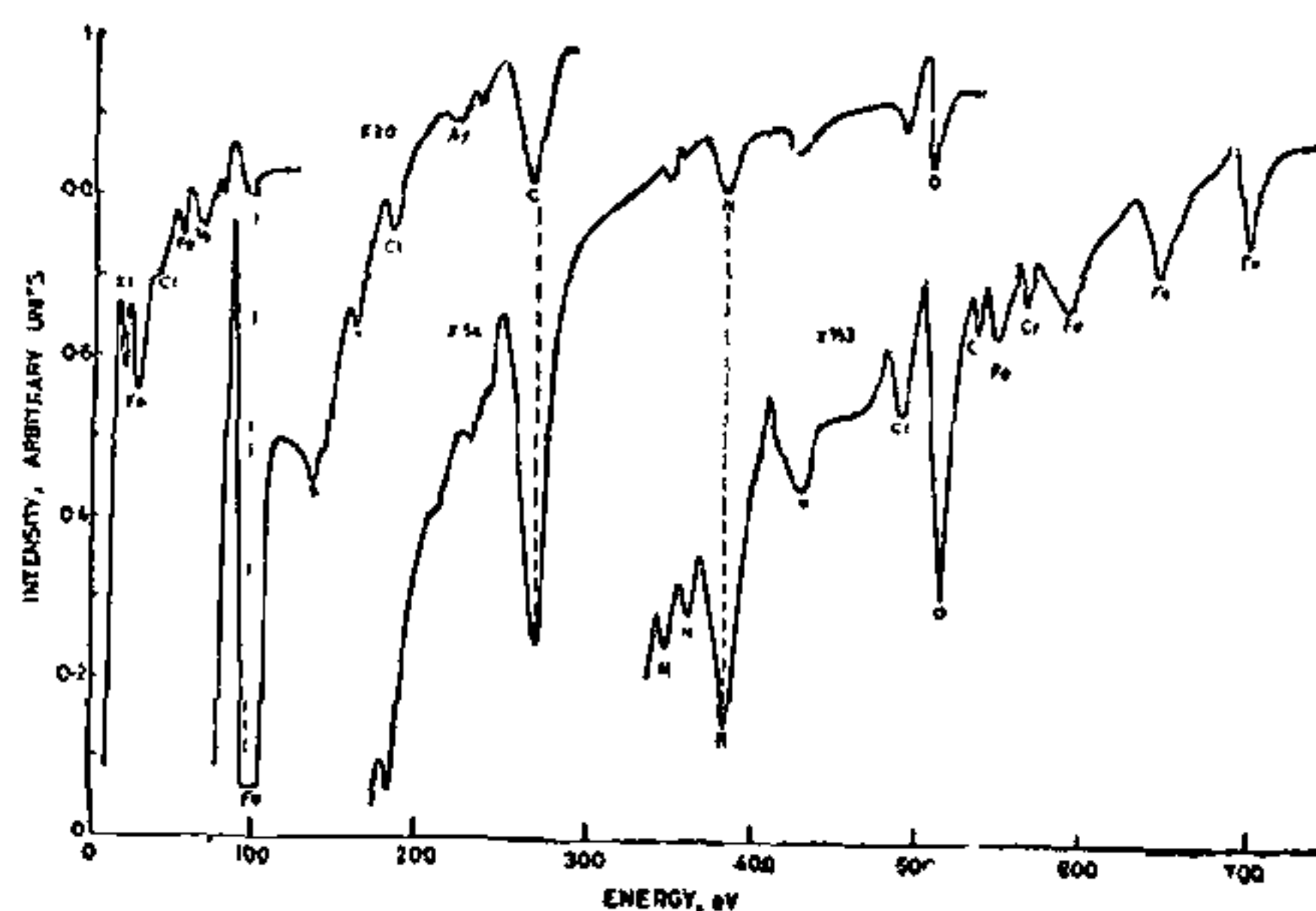


FIG. 1. Derivative auger spectra of stainless steel.

electron bombardment during analysis, these gases desorbed and their amplitudes are reduced with time.

Carbon is the major surface contaminant. The large concentration of carbon is mainly from the surface conditions due to its pre-history (handling) but not from the vacuum environment. This is confirmed by the fact that the other two samples studied in this system under similar conditions did not show the presence of carbon. It is possible that a small fraction of carbon could be due to its segregation from the bulk.

It would be ideal to work out approximate percentages of the contaminants from the peak-to-peak heights of the Auger peaks. Although the Auger yields from pure elements are available in literature, these values for most elements in different chemical environments or for most compounds are not known. Hence a quantitative estimate of the percentage concentrations of impurities is not made here.

With reference to the reduced lead-glass (Table I) besides Pb and Si, Se, Sb, As and In are detected. However, no trace of Na was found on the surface. One possible reason for the absence of Na, in the recorded spectrum is the low sensitivity factor for Na. It is also possible that due to the electrostatic field set-up by the impinging electrons, within the sample to a certain depth proportional to the primary energy, sodium ions are attracted to this region<sup>3</sup>, thereby changing the surface composition of Na.

To confirm the presence of the contaminants, the same sample was subjected to the energy dispersive X-ray fluorescence test. This test confirmed the presence of Se and In.

Aluminium coated phosphor screen was used to optimise the electron beam size and is incidentally analyzed for the surface contaminants,

TABLE I  
Experimental results

| Sample                             | Elements/compounds detected                           | Source  |
|------------------------------------|---|---|
| Stainless steel                    | Fe, Cr, Ni<br>C, S, N, O, Cl, Ar                      | Substrate<br>Impurities in the bulk adsorbed species, cleaning solution, rinse water associated with the processing of stainless steel. |
| * Reduced lead-glass               | Si, SiO <sub>2</sub> , Pb, K, Ca<br>In, O, Se, Sb, As | Substrate<br>Contaminants from possible modifiers.  |
| * Aluminium coated phosphor screen | Al, Al oxide, O, Zn, S<br>P                           | Al coating.<br>Segregated from the phosphor material.<br>Impurity in the aluminium used for coating.                                    |

The spectra are not reproduced in this article.



The spectra are obtained by using the maximum permissible modulating potential to enhance the Auger peak intensities at the expense of resolution without sacrificing the unambiguous identification of various Auger peaks.

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## POTASSIUM N-iodo BENZENE SULPHONAMIDE AS AN OXIDIMETRIC TITRANT IN AQUEOUS MEDIUM

RANGASWAMY AND H. S. YATHIRAJAN

*Department of Chemistry, University of Mysore, Manasagangothri, Mysore 570 006, India*

#### ABSTRACT

Potassium N-iodo benzene sulphonamide (iodamine-B) is proposed as an oxidimetric titrant in aqueous medium. The general conditions for its use and the procedures for estimating hydrazine, ascorbic acid, hydroquinone, semicarbazide, thiourea, arsenic(III), antimony(III) and thallium(I) by a direct titration and thiosulphate, xanthate, dithiocarbamate and thiosemicarbazide by a back-titration procedure are described.

#### INTRODUCTION

RECENTLY, organic haloamines have received considerable attention as redox titrants. Dichloramine-B<sup>1</sup>, dibromamine-B<sup>2</sup>, bromamine-B<sup>3</sup> and iodamine-T<sup>4</sup>, have been reported. A recent addition to the group is iodamine-B, the iodine analogue of chloramine-B. Direct determinations of typical reductants such as hydrazine, ascorbic acid, hydroquinone, semicarbazide, thiourea, arsenic(III), antimony(III) and thallium(I) and back-titration methods for estimating thiosulphate, xanthate, dithiocarbamate and thiosemicarbazide with iodamine-B have been developed and are reported in the present communication.

#### EXPERIMENTAL

##### Reagents

##### Preparation of iodamine-B

The compound can be obtained<sup>5</sup> in pure form as pale yellow crystalline powder which is the potassium salt of N-iodobenzene sulphonamide,  $C_6H_4SO_2NIK$ .

##### Stock solutions of iodamine-B

Solutions of iodamine-B in water are unstable (turbidity appears after two days). By the addition of alkali, the solution can be made stable for about one month. An approximately 0.1N (0.05M) solution was made by dissolving 16.5 g of dry iodamine-B in 1 litre of 0.1 M potassium hydroxide and kept in an amber coloured bottle. The solution was standardised by the iodometric method suggested for chloramine-T<sup>6</sup>.

##### Reductants

Solutions of A.R. quality hydrazinesulphate, ascorbic acid, hydroquinone, semicarbazidehydrochloride, thiourea, antimony(III), thiosulphate, dithiocarbamate, xanthate and thiosemicarbazide in water were prepared. Arsenic(III) solutions were prepared by dissolving known amounts of arsenious oxide in 1M sodium hydroxide, neutralising with 1N sulphuric acid and making up to a known volume with water. Thallous carbonate was dissolved in the minimum amount of acetic acid and made up to a known volume with water. The strengths of all these solutions were checked by standard methods.