

APPLICATIONS OF LOW ENERGY X- AND GAMMA RAYS

Edited by

CHARLES A. ZIEGLER

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Preface

THE PAPERS IN THIS VOLUME were presented at the third in a series of symposia on low energy X- and gamma-ray sources and applications held at Boston College, Chestnut Hill, Massachusetts. The symposium, which was sponsored jointly by Boston College, Panametrics, Inc., and the Atomic Energy Commission, was international in character, and broad in scope in the sense that fundamental studies were combined with applications in such diverse fields as geology and medicine as well as a wide variety of industrial applications.

The purpose of the third symposium, as was that of the first and second, was to bring together scientists engaged in the development of X- and gamma-ray technology and to provide an opportunity for an interchange of ideas. This should assist in meeting requirements placed upon us by our ever-expanding society in the fields of medicine, environmental pollution analysis, and industrial quality control.

The general theme of the symposium was biased toward radioisotope-excited X-ray fluorescence analysis (XRF) and the various techniques employed to improve upon the technology. Energy determination using balanced filters and scintillation counters or semiconductor and proportional counters were treated in considerable depth. Other technologies covered were Mössbauer effect spectrometry and electron spectroscopy. It is expected that interest in each of these areas will continue to grow as the spectral capabilities of each are better understood.

It is interesting to note that radioisotope-excited XRF first became practical back in the forties after the Manhattan District Project came into being although the possibility of radioisotope X-ray excitation was demonstrated by Chadwick's experiments in 1912 when he observed that alpha particles could be used to excite fluorescent X-rays. But it has been only within the past 10 years that XRF has begun to show signs of wide acceptance by industry and by scientists working in a variety of disciplines.

Mössbauer effect spectroscopy based on recoilless resonant absorption of gamma rays has, in the 12 years since its discovery by R. L. Mössbauer, likewise made significant advances into the fields of chemistry and metallurgical research. This relatively new analytical

technique and the still newer developments of electron spectroscopy, each in its own right, appear to offer many potential benefits that are yet to be appreciated fully. The sponsorship of government programs with valuable input from industrial and other organizations will, no doubt, bring about a better understanding of these benefits.

JAMES W. HITCH
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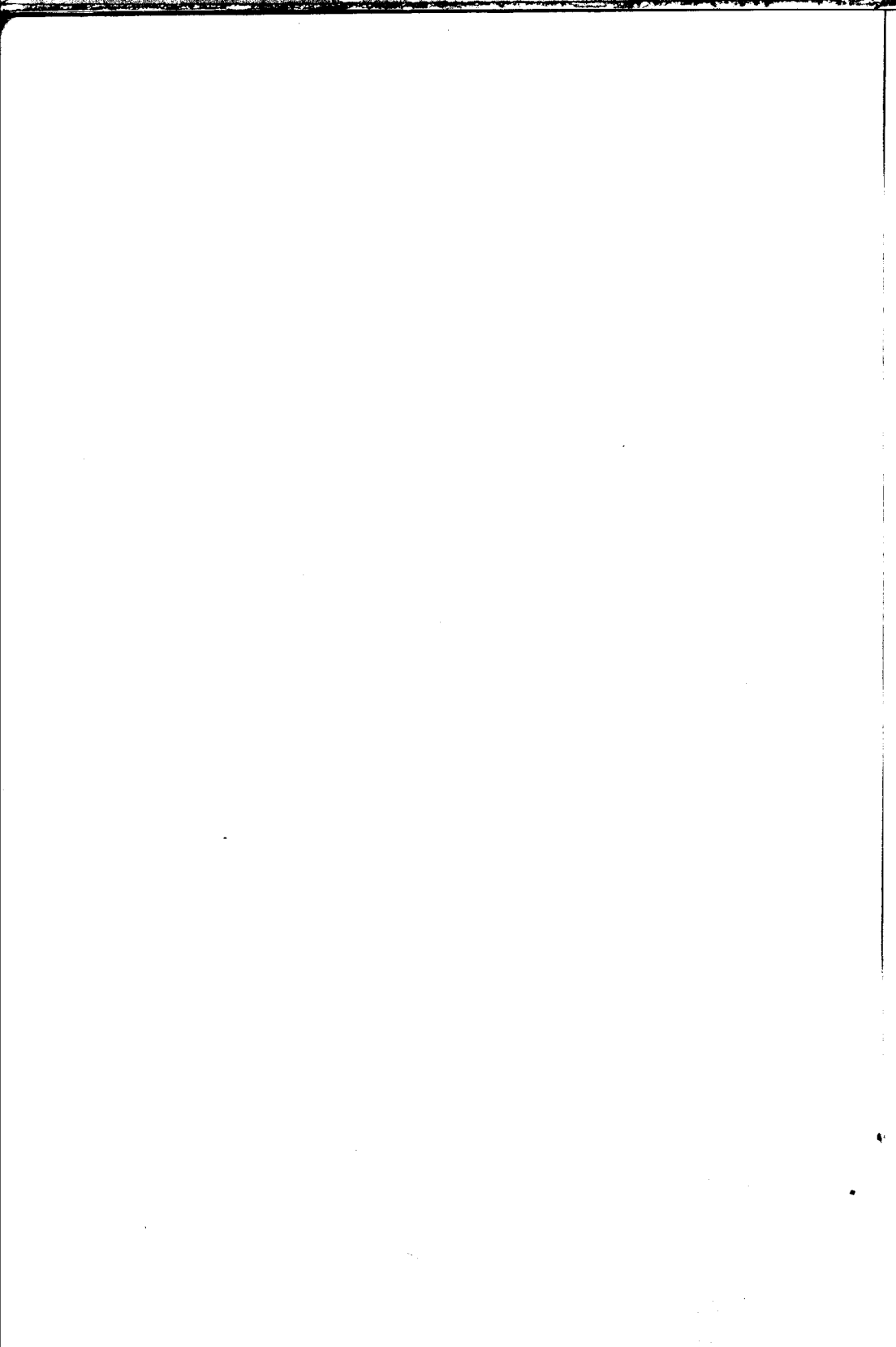
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INDUSTRIAL APPLICATIONS



Progress in Industrial Application of Low Energy Radioisotope X-Ray Techniques

J. R. RHODES

INTRODUCTION

ONE OF THE PROBLEMS of a review such as this is to define its boundaries in time, space and subject matter. This paper reviews applications since 1967,¹ with special reference to instruments and techniques in radioisotope X-ray analysis that have found significant application in industry. Included are field and field-laboratory uses in the mining industry, and developmental equipment which is intended for industrial use.

Radioisotope low energy X-ray fluorescence, absorption and backscatter are used in two main categories of measurement, coating thickness and elemental analysis. A convenient and self-consistent set of formulae and recipes suitable for calculating feasibility of these measurements by the above techniques, and by beta particle backscatter, has recently been published.²

This review is confined to the two most significant areas of industrial application that have developed in the past three years, the use of portable radioisotope X-ray fluorescence analyzers and the installation of a number of on-stream process analyzers.

Later in this volume Cameron³ will be discussing the state of the art in portable, laboratory and on-line methods of coating thickness measurement. Also Chen and Cahill⁴ will describe industrial application of X-ray fluorescence and absorption to the measurement of thickness.

The range of applications of X-ray absorption and backscatter for analysis is not great enough to warrant special review. The main applications are to effectively two-component systems such as coal/ash⁵ and meat/fat,⁶ and to the determination of heavy elements in light matrices such as sulphur or cobalt in hydrocarbons.⁷

APPLICATIONS

Half of all the papers published in the last three years describe portable analyzers and applications of them in various industrial and field en-

vironments. Half of the rest describe applications to industrial process control. These include "on-stream" analyzers where a continuously moving sample of the plant stream is measured, and "off-stream" analyzers which measure discrete samples taken automatically.

It is not known how many individual instruments are in use, but it is probable that the number of portable analyzers in use is much greater than the number of papers written on their use, since they have been commercially available for five years. However, other analyzers are not yet generally available, and the number in use is not likely to greatly exceed the number of papers written.

Table 1 classifies, alphabetically by element, the significant analyses reported in the past few years in the above-mentioned areas. A study of the published feasibility investigations reveals that in about 60% of them the objective was to optimize the accuracy of major component determinations, and in the rest it was to obtain the best possible sensitivity. In the former case, the main sources of error are matrix effects. In applications of portable analyzers, much ingenuity has gone into devising methods of minimizing these errors without compromising the essential simplicity of the equipment and measurement procedures. We expect to see more development along these lines, as matrix effects appear to be one of the main obstacles to much more widespread field use.

Sensitivity is limited by the basic considerations of counting statistics, and the fluorescent to scatter ratio. Using scintillation and proportional counters, detection limits of about 0.005% (and in favorable cases, 0.001%)⁷ can usually be obtained by one of two methods. The first is to use a source whose backscattered radiation is just resolved from the required characteristic X-rays. The second is to excite the required fluorescence with maximum possible efficiency using an energy just above the absorption edge of the element to be determined. Resolution limitations prevent employment of both these methods simultaneously with proportional or scintillation counters. They do not, however, when Si(Li) or Ge(Li) detectors are used, and in this case sensitivities of a few p.p.m. can be obtained even with heterogeneous ore samples.^{8,9} It seems that the development potential of proportional and scintillation counters in this respect has been thoroughly investigated and largely exhausted.¹⁰ This is not the case with Si(Li) and Ge(Li) spectrometers where many studies using obvious combinations of high resolution detectors, monochromatic line sources and thin samples¹¹ (for example) have not yet been performed. We expect to see a rapid increase in the number of investigations in this area.

TABLE 1 Analysis

Element and % conc. range	Material (see Note 1)	Source and activity	X-ray excit.	Filter	Analyzer type (see Note 2)	Application	Detection limit (or 1 σ prec.)*	Remarks
Aluminum 2.4 Al ₂ O ₃	Cement raw mix 13, 49	⁵¹ Hf/Zr, 3 Ci; ²¹⁰ Po, 10 mCi	AlK	Al	On-line P (fused pellets)	Kiln-fed con- trol	0.09% Al ₂ O ₃ *	Also see under Ca, Fe, Si
Barium 1-15	Barytes ores 41, 53	²⁴¹ Am-Sm; ²⁴¹ Am-Ba, 14 mCi	BaK	—	On-stream S (slurry)	Barytes flota- tion pilot plant control	0.01%	Balanced tar- get method
Calcium 40-50	Cement raw mix 22	⁵⁵ Fe, 2 mCi	CaK	—	Portable S	Feasibility study	0.2% CaO*	—
1-5	Silicate rocks 18, 19	⁵⁵ Fe, 10 mCi	CaK	K/Ca	Portable SS	Soil and rock type identity	0.5% CaO*	Also see under K
15-19 CaO	Sinter mix 44, 54	⁵⁵ Fe, 7 mCi	CaK	—	On-line P (pressed pellets)	Blast furnace control	0.15%	Also see under Si; Fe
75-85 CaCO ₃	Cement raw mix 13, 48, 54	⁵⁵ Fe, 2 mCi	CaK	—	On-stream P (slurry)	Kiln-fed con- trol	0.35% CaCO ₃ **	—
40-50 CaO	Cement raw mix 13, 49	⁵¹ Hf/Zr, 3 Ci; ²¹⁰ Po, 10 mCi	CaK	—	On-line P (fused pellets)	Kiln-fed con- trol	0.16% CaCO ₃ *	Also see under Al, Fe; Si
40-50 CaO	Cement raw mix 53, 56	³ H/Ti, 5 Ci	CaK	—	On-stream P (powder)	Grinder control	0.4% CaO*	—
40-45 CaO	Cement raw mix 45	³ H/Ti, 5 Ci	CaK	—	On-stream P (powder)	Kiln-fed con- trol	0.2% CaO*	Also see under Fe
40-45 CaO	Cement raw mix 47, 57	¹⁰⁹ Cd, 1 mCi	CaK	—	Powder sample analyzer P	Feasibility study	0.17% CaO*	Reduced par- ticle size effects

TABLE 1 Analysis—continued

Element and % conc. range	Material (see Note 1)	Source and activity	X-ray excit.	Filter	Analyzer type (see Note 2)	Application	Detection limit (or 1 σ prec.)*	Remarks
Chromium 1-20	Steels 36	^{55}Fe , 7 mCi; $^3\text{H}/\text{Zr}$, 2.5 Ci	CrK	Ti/V	Portable S	Alloy analysis	0.1% Cr*	
0.5-8	Steels 22	^{238}Pu , 20 mCi	CrK	Ti/V	Portable S	Alloy analysis	0.15% Cr*	
0.5-5	Electroplating bath solutions	$^3\text{H}/\text{Zr}$, 4 Ci	CrK	Ti/V	Portable S	Feasibility study	0.15% Cr	
Copper 0.1-15	Copper ore pulps; rockfaces 12, 14, 34	$^3\text{H}/\text{Zr}$, 12 Ci	CuK	Co/Ni	Portable S	Field assay	0.2% Cu*	
0.4-4	Cu ore pulps 55	$^3\text{H}/\text{Zr}$	CuK	Co/Ni	Portable S	Draw control; sub-level caving	0.1% Cu*	
0.03-2.5	Cu ore pulps 22	^{108}Cd , 1 mCi	CuK	Co/Ni	Portable S	Feasibility study	0.03% Cu	
0.1-5	Cu ore pulps 50, 60	$^3\text{H}/\text{Zr}$, 2.5 Ci	CuK	Cr/Mn, Ni/Co	Portable S	Feasibility study	0.2% Cu*	Special method eliminates matrix effects
0.1-10	Core samples 58	^{238}Pu , 30 mCi	CuK	Co/Ni	Portable S	Core analysis	0.05% Cu*	Also see under Ni, Fe and Mn
0.4-1.6	Min nodule pulps 24	^{238}Pu , 20 mCi	CuK	Co/Ni	Portable S	Shipboard assay		
60-95	Cu alloys 36	^{108}Cd 2 mCi	CuK	Co/Ni	Portable S	Alloy sorting	0.45% Cu*	
0.1-26	Various Cu-Pb-Zn ores 39	$^{238}\text{Pu-Ga}$, 30 mCi	CuK	Cu	On-stream (slurry) S	Mill control	0.08-1%*	See also under Pb, Sn and Zn
Gold 0.01-1	Simulated ores 23	^{57}Co , 1 mCi	AuK	Hf/W	Portable S	Feasibility study	0.03% Au	

Iron 20-70	Iron ores (pulpis; cores) 15, 57, 61	²³⁹ Pu, 30 mCi	FeK	Cr/Mn	Portable S	Field assay, mine control	0.5% Fe*	
1-10	Lead-zinc ores							
2-12	Manganese nodule pulps 24	²³⁹ Pu, 20 mCi	FeK	Cr/Mn	Portable S	Shipboard assay	0.24% Fe*	
5-50	Iron ores (crushed rock) 57, 62-64	²³⁹ Pu, 10 mCi	FeK	Cr/Mn	Portable S	Strip mine control	~ 1% Fe*	X-ray back- scatter also considered Also see under Ca; Si
25-45	Sinter mix 44, 54	¹ H/ ² Zr, 4 Ci	FeK	Cr/Mn	On-line (pressed pellets) P	Blas furnace control	0.2% Fe*	
0.5-3	Cement raw mix 13, 49	¹ H/ ² Zr, 3 Ci and ²¹⁰ Po, 10 mCi	FeK	—	On-line (pressed pellets) P	Kiln-fed con- trol	0.05% Fe	Also see under Ca, Fe; Si
0.5-3	Cement raw mix 45	¹ H/ ² Ti, 5 Ci	FeK	Cr/Mn	On-stream (powder) P	Kiln-fed con- trol	Also see under Ca	
0.1-5	Sand (slurry) 13	²³⁹ Pu, 10 mCi	FeK	Cr/Mn	On-stream (slurry) P	Pilot plant	0.003% Fe	
0.1	Lubric. oil 57	¹ H/ ² Zr, 2 Ci	FeK	Fe	Assay G-M	Wear monitoring	0.01% Fe	
Lead 0.05-30	Ore pulps 66	²³⁹ Pu, 30 mCi	PbL	Ga/Ge	Portable S	Field analysis	0.08% Pb	Matrix; particle size studied
1-8	Lead ores 27	¹⁰⁹ Cd	PbL	—	—	—	—	
	Core samples 58	²³⁹ Pu, 30 mCi	PbL	Ga/Ge	Portable S	Core analysis	2% Pb*	
	Lead ores; mine walls 28	⁵⁷ Co	PbK	—	Portable S	Mine control	0.25% Pb	
	Lead ores; core samples 29, 30, 32	⁷⁶ Se	PbK	—	Portable S	Field assay	—	Used nomo- grams
0.05-3	Leaded brass; steels 36	¹⁵³ Gd, 0.6 mCi	PbK	W/Ir	Portable S	Alloy sorting	0.05% Pb	
0.1-32	Ores, zinc, con- centrates, tail- ings 38, 39	²⁴¹ Am, 5 mCi ⁵⁷ Co, 1.2 mCi	—	—	On-stream (slurry) S	Mill control	0.04-0.34% Pb*	Absorption edge analysis. See also under Cu, Sn, Zn
0.5-5	Solutions; slurries 65	¹⁵³ Gd, 1 mCi ¹³⁷ Cs, 1 Ci	PbK	—	On-stream S	Feasibility study	0.4% Pb	Comparison of sources

TABLE 1 Analysis—continued

Element and % conc. range	Material (see Note 1)	Source and activity	X-ray excit.	Filter	Analyzer type (see Note 2)	Application	Detection limit (or 1 σ prec.)*	Remarks
Manganese 20-40	Manganese nodule pulps 24	²³⁸ Pu, 20 mCi	MnK	V/Cr	Portable S	Shipboard assay	0.75% Mn*	Also see under Fe, Cu, Ni
0.2-1.5	Steels 36	³ H/Zr, 214 Ci	MnK	V/Cr	Portable S	Alloy sorting	0.14% Mn*	
0.5-5	Plating bath sol. 23	²³⁸ Pu, 20 mCi	MnK	V/Cr	Portable S	Feasibility study	0.04% Mn	
Molybdenum 0.01-1	Ore pulps; Cu conc. 15, 61	¹⁰⁹ Cd, 2 mCi	MoK	Y/Zr	Portable S	Mine control	0.01% Mo	
1-20								
0.001-0.3	Ore pulps 23	¹⁰⁹ Cd, 1 mCi	MoK	Y/Zr	Portable S	Feasibility study	0.003% Mo	
0.01-5	Steels 36	¹⁰⁹ Cd, 2 mCi	MoK	Y/Zr	Portable S	Alloy sorting	0.04% Mo	
0.1-10	Steels 22	¹⁰⁹ Cd, 1 mCi	MoK	Y/Zr	Portable S	Alloy analysis	0.1% Mo*	
0.002-0.5	Mine samples, Cu conc. slags 67	¹²⁵ I, 2 mCi	MoK	—	Laboratory; on-stream SS	Feasibility study	0.002 to 0.01% Mo	
0.01-7 MoO ₃	Ore slurries 41	¹⁴⁷ Pm/Al-Ag 1 Ci	MoK	Y/Zr	On-stream S	Pilot plant control	0.01% Mo	Also see under Nb; Sn
Nickel 0.8-1.6	Manganese nodule pulps 24	²³⁸ Pu, 20 mCi	NiK	Fe/Co	Portable S	Shipboard assay	0.05% Ni*	Also see under Fe, Cu, Mn
3-30	Cu alloys 36	³ H/Zr, 2.5 Ci	NiK	Fe/Co	Portable S	Alloy sorting	0.3% Ni	
Niobium 0.01-5	Ore slurries 41	¹⁴⁷ Pm/Al-Ag, 1 Ci	NbK	Sr/Y	On-stream S	Pilot plant control	0.01% Nb	Also see under Mo; Sn
0.06-2	Steels 36	¹⁰⁹ Cd, 2 mCi	NbK	Sr/Y	Portable S	Alloy analysis	0.06% Nb	
Potassium 1-5	Silicate rocks 18, 19	⁵⁹ Fe, 10 mCi	KK	Ci/K	Portable SS	Soil; rock type identity	~ 0.5% K*	Also see under Ca

Silicon 12-16 SiO ₂	Sinter mix 44, 57	³ H/Zr, 4 Ci	SIK	—	On-line (pressed pellets) P	Blast furnace control	0.17% SiO ₂	Also see under Fe; Ca
14 SiO ₂	Cement raw mix 13, 49	³ H/Zr, 3 Ci; ²¹⁰ Po, 10 mCi	SIK	—	On-line (fused pellets) P	Kiln-feed con- trol	0.13% SiO ₂	Also see under Al, Ca, Fe
Silver 0.01-0.1	Ag ores 8	¹⁰⁷ Pm/Al, 0.5 Ci	AgK	Mo/Rh	Portable S	Field assay	0.003% Ag	
Sulphur 0.1-5 S	Pulverized coal 22	³ H/Ti, 5 Ci	SK	P/S	Portable S	Feasibility study	0.17% S	Low noise PMT used
0.01-0.5 SO ₂	Stack gases 70	⁵⁵ Fe, 10 mCi	SK	P/S	On-stream P	Feasibility study	0.03% SO ₂	Paper com- parison of nuclear and non-nuclear techniques
Tin 0.05-25	Tin ore pulps 12, 33, 34	¹⁰⁷ Pm/Al, 0.5 Ci	SnK	Ag/Pd	Portable S	Mine control	0.03% Sn	
0.5-5	Tin mineraliza- tion 14, 34, 68	¹⁰⁷ Pm/Al, 0.5 Ci	SnK	Ag/Pd	Portable S	In situ assay of rock faces	0.1% Sn	
0.5-5	Tin mineraliza- tion 28	¹³⁷ Sr/Ba	SnK	—	Portable S	In situ assay of rock faces	0.1% Sn	
0.5-5	Tin ores, cores 39, 58	¹⁰⁷ Pm/Al, 0.5 Ci	SnK	Ag/Pd	Portable S	Core analysis	0.1% Sn	
0.002-0.1	Tin mineraliza- tion 69	¹⁰⁷ Pm/Al, 0.5 Ci	SnK	Ag/Pd	Portable S	Geochemical prospecting	0.005% Sn	Feasibility study
0.01-2	Tin ore slurries 35, 39, 41	²⁴¹ Am-Ba, ²⁴¹ Am-Sn, 14 mCi	SnK	Ag/Pd	On-stream (slurry) S	Process con- trol	0.005% Sn	See also under Mo, Nb
0.1-11	Bronzes; gun metals 36	¹⁰⁷ Pm/Al, 0.5 Ci	SnK	Ag/Pd	Portable S	Alloy sorting	0.2% Sn*	