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Eechnical Note

AN OUTLINE FOR COOPERATIVE ACTION ON THE DETERMINATION OF X-RAY MASS ATTENUATION COEFFICIENTS IN THE WAVELENGTH RANGE FROM 0.5 TO 100 \mathring{A} (25 to 0.12 keV)

KURT F. J. HEINRICH



U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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Kurt F. J. Heinrich

Spectrochemical Analysis Section Analytical Chemistry Division Institute for Materials Research

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PREFACE

Investigators in x-ray spectrometry, as well as in other related fields, are becoming increasingly aware of the urgent need for a revision of available information concerning x-ray mass attenuation coefficients. It is the purpose of this report to show the extent to which progress in x-ray fluorescence spectrometry and electron probe microanalysis depends upon reliable attenuation coefficients. A list of the limited sources of information presently available is included, and the necessity for further work in this area is indicated.

As a consequence of the unsatisfactory situation, an increasing number of laboratories is presently involved in the measurement of x-ray mass absorption coefficients, and others are planning such activities in the near future. There is, however, a need for a coordinating group which will promote the exchange of information concerning such research, assist in the selection of techniques when requested to do so, suggest areas in which measurement is most urgently needed, and obtain by interpolation procedures the mass absorption coefficients for those elements and wavelength regions for which experimental data are not available.

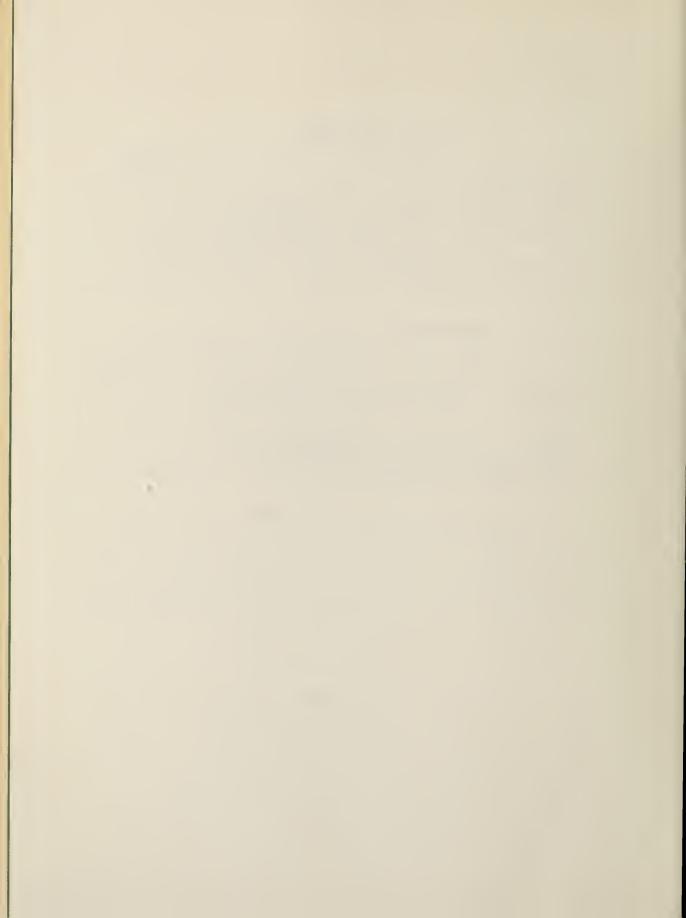
Discussions of these problems with prominent investigators in the field, in this country as well as abroad, have indicated that our group here at the National Bureau of Standards would be appropriate for coordinating this work. It is therefore hoped that those readers who are involved in such measurements or plan to perform them, will assist us in this cooperative task to the benefit of all concerned.

Kurt F. J. Heinrich

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ABSTRACT

Sources of information on x-ray mass attenuation coefficients and the present status of data in this area are reviewed. Many gaps are observed in the existing data and some values derived by interpolation may be of questionable accuracy. Because of the importance of accurate mass attenuation coefficients, the extension and improvement of these measurements is proposed together with a plan for coordination of effort and exchange of information.

1. INTRODUCTION

In the first half of this century, an impressive amount of work was performed on the experimental determination of x-ray attenuation coefficients. The tables published by Allen and re-published in the book X-Rays in Theory and Experiment, by Compton and Allison, are representative of the results achieved in this period. The amount of effort spent in this endeavor can be appreciated if it is taken into account that early workers in this field did not have at their disposal the stable x-ray sources and sensitive x-ray measuring devices which are now available. Practically all of the early work was done by photographic registration of x-rays or by measurements with ionization chambers.

Presently available instrumentation permits the measurement of mass attenuation coefficients with much less experimental effort and greater accuracy. The stability of x-ray sources has been improved considerably; and photon counting techniques using scintillation and proportional. detectors permit satisfactory measurements over a wide range of intensities. As a greater sensitivity is achieved with these detectors, it is now possible to measure attenuation of secondary (fluorescent) radiation. The investigator has at his disposal a very wide range of useful x-ray radiations produced by secondary fluorescent targets external to the vacuum enclosure of the primary x-ray generator. Meanwhile, the amount of work on experimental determination of mass attenuation coefficients has diminished considerably in the last two decades. X-ray physics is not at present as interesting a field for most theoretical physicists as it was forty years ago; those working in the x-ray field are now more concerned about fine structure in absorption and

emission spectra than in absolute values of mass attenuation coefficients. As a result, the present technical capabilities of improving our knowledge of mass attenuation coefficients have remained largely unused.

2. NEEDS FOR MASS ATTENUATION COEFFICIENT TABLES

Under the impact of the instrumental developments mentioned above, x-ray spectrometry has developed into one of the most important methods for instrumental analysis of elementary composition. The two techniques employed most frequently are x-ray fluorescence spectrometry and electron probe microanalysis.

In x-ray fluorescence spectrometry, use is made of the fact that the wavelengths of x-ray lines produced within a specimen irradiated by a polychromatic beam of primary Xrays are characteristic of the elements present in the specimen. As x-ray spectra contain fewer lines than emission spectra in the visible range, the use of such spectra for qualitative analysis is particularly attractive. The intensity of observed lines is also uniquely related to the composition of the emitting specimen. Since x-ray intensity measurements of a precision of 0.1-0.2% of total intensity can be made fairly easily, quantitative x-ray fluorescence emission analysis is often capable of a precision comparable to that of conventional chemical analysis. In many applications this technique is superior in simplicity, economy, and speed, to other analytical procedures. Further advantages are the non-destructive nature of x-ray spectrometry, and the fact that the digital electronic signal output is particularly useful for computer applications and for automated systems.

One major limitation of this technique, however, lies in the fact that the intensity of a particular emission line, although primarily related to the concentration of the element originating it, is also a function to a minor degree of all other elements present in the specimen. The interelement effects thus observed make it necessary, for quantitative analysis, to compare the x-ray intensities obtained from an unknown specimen with those emitted by standards of known composition close to that of the unknown. When the specimens to be analyzed cover a wide range of composition, and particularly with specimens containing more than two components, a great number of well characterized standards is thus necessary to cover the whole compositional range under investigation. Although several attempts have been made in the past to overcome this difficulty by an analysis of the underlying physical events, this was found to be too cumbersome a task. Current practices are in general limited to the use of matching standards, or semiempirical approaches, which, in turn, also are based on the analysis of a great number of specimens.

At a recent discussion session at NBS sponsored by the Spectrochemical Analysis Section of its Analytical Chemistry Division and attended by some of the leading investigators in x-ray fluorescence spectrometry, ways and means of improving the technique of quantitative analysis were discussed. Τt was the consensus of the participants that the current semiempirical techniques, while more useful than a technique of matching standards, still are cumbersome and imperfect. The need for testing such a system by means of known standards renders the results dependent upon the accuracy of the chemical methods used in characterizing the standards. Furthermore, the enhancement effects of fluorescent excitation of one element by fluorescent radiation emitted by another element in the specimen, are not well described by the equation systems currently in use. It was also suggested that in recent years calculation techniques by electronic computers have advanced to a degree that would permit a quantitative re-investigation of the theory of x-ray fluorescence spectrometry on a computational basis not available previously.

The x-ray intensity observed on an irradiated specimen depends to a great extent upon the absorption of the primary radiation penetrating the specimen, and the absorption of the secondary or fluorescent radiation on its way to the specimen surface. What renders the calculation so tedious is the fact that strong monochromatic x-ray sources are not available at present, so that the absorption of the primary polychromatic radiation must be integrated over a wide range of wavelengths. It is also true that the success of such a calculation depends upon the knowledge of the mass attenuation coefficients for the radiation of all wavelengths involved in the process. As x-ray techniques are extended more and more to long wavelengths, the lack of knowledge of attenuation coefficients in the soft range becomes more serious. It was thus concluded at the aforementioned session, that better knowledge of mass attenuation coefficients is a prerequisite for successful theoretical treatment of quantitative x-ray fluorescence analysis.

The interelement effects observable in x-ray fluorescence are also present in <u>electron probe microanalysis</u>. However, empirical correction methods are even more impractical in this technique, in view of the difficulty in obtaining standard reference materials of composition close to that of the sample and homogeneous on a microscopic scale. The theoretical treatment of electron probe analysis, although quite involved, is somewhat simplified by the fact that the exciting electrons are monochromatic, i.e., all electrons striking the specimen possess the same energy within very narrow limits.

The corrections to be applied to the experimentally obtained x-ray intensities in order to calculate the composition of a specimen are the following:

a. The absorption correction, which corrects for differences in the attenuation of characteristic radiation within specimen and standard.

b. The fluorescence corrections, which correct for indirect x-ray excitation (fluorescent radiation) produced by characteristic and continuous radiation of energy higher than that of the line under investigation.

c. The atomic number effect correction, which corrects for differences in the efficiency of production of characteristic x-ray photons, as a function of atomic number of the specimen or standard.

Numerous attempts have been made to establish methods for the corrections outlined above. Practical progress is, however, very slow because of the difficulties in testing experimentally the proposed procedures and in separating correctly the effects of the three corrections. These difficulties are due to the lack of adequate standard materials to be used for critical tests, and to insufficient knowledge of physical parameters, such as the x-ray mass attenuation coefficients, x-ray excitation efficiencies and fluorescence yields, and electron backscattering coefficients.

While the atomic number effect is at present the most controversial, in most cases the absorption correction is of greatest magnitude. The exact value of this correction depends primarily on the mass attenuation coefficients of the specimen for the radiation under investigation. Hence, satisfactory knowledge of the mass attenuation coefficients of all elements within the wavelength region used in electron probe microanalysis is required before performing correct absorption corrections. Only after the absorption effects have been corrected can the other corrections be tested. Incidentally, mass attenuation coefficients enter into the other corrections as well.

Microprobes are now being provided with x-ray spectrometers for the soft x-ray wavelength range from 10 to 100 Å. In this region, attenuation coefficients are virtually unknown. Hence, the need for the determination of such data becomes even more pressing.

There are, obviously, other fields of science in which a better knowledge of mass attenuation coefficients is considered desirable, as for instance in the study of radiation in the upper atmosphere and in space. X-ray spectrometric analysis merely gives most striking demonstration for the need to revise the current attenuation data.

3. SOURCES OF INFORMATION

About 100 publications communicating the results of experimental determination of mass attenuation can be found in the literature. Most of these communications cover a rather narrow range of wavelengths and absorbers, and while there is a considerable overlap in the regions covered, many elements have not been investigated at all. Several attempts have therefore been made to provide the user of mass absorption coefficients with tables in which the available information is covered. In some cases data for those regions which have not been covered experimentally have been derived by interpolation between existing values. Some of the tables most frequently employed by the x-ray spectroscopist will be described in the following sections.

1. S. J. M. Allen, "Mean values of the mass absorption coefficients of the elements." In A. H. Compton, and S. K. Allison, X-Rays in Theory and Experiment, D. Van Nostrand Co., New York, 1935. This compilation reflects the results obtained in early research in the field, and is widely used. Interpolation has been employed, and the tables do not clearly distinguish between experimental data and those obtained by interpolation. Several recent, and presumably reliable, experimental results are at variance with Allen's data; these time-honored tables should by now be considered obsolete.

2. L. S. Birks, Electron Probe Microanalysis, Appendix 3, Interscience Publishers, New York-London, 1963.

3. S. J. M. Allen, "Mass Absorption Coefficients for X and Gamma Rays," Handbook of Chemistry and Physics, 43rd Ed. (1961) 2718-2723. The Chemical Rubber Publishing Co., Cleveland, Ohio.

4. H. A. Liebhafsky, et al., X-Ray Absorption and Emission in Analytical Chemistry, Appendix IV, J. Wiley, New York, 1960.

5. Philips Electronics Instruments, "Table of X-Ray Mass Absorption Coefficients," Norelco Reporter <u>9</u> (1962), May-June (Supplement).

6. K. Sagel, Tabellen zur Rontgen-Emissions- Und Absorptions-Analyse, Springer Verlag, Berlin, Göttingen, Heidelberg, 1959. The preceding tables are basically modifications of Allen's data; particularly for longer wavelength there are serious disagreements among these references; all of them tend to disagree with recent measurements.

7. E. Petrassi, Table of X-Ray Mass Absorption Coefficients, in R. Theisen, Quantitative Electron Microprobe Analysis, p. 32. Springer Verlag, Berlin, Heidelberg, New York, 1965. The section of this table corresponding to soft x-rays and low atomic number elements has been constructed using some of the newest experimental data available. The part concerning heavier elements, however, is inconsistent and disagrees with some of the newer measurements.

8. G. White Grodstein, X-Ray Attenuation Coefficients from 10 kev to 100 Mev. National Bureau of Standards Circular 583 (1957) (35 cents), and Supplement to Circular 583 (1959) (15 cents), available from U. S. Govt. Printing Office, Washington, D. C. This work includes a lucid discussion of the physics of mass absorption coefficients. The wide range of energies considered precluded a detailed documentation of the low energy range of special interest to the x-ray spectroscopist.

9. H. M. Stainer, X-Ray Mass Absorption Coefficients. A Literature Survey. U. S. Bureau of Mines Information Circular 8166 (1963). Available from U. S. Govt. Printing Office, Washington, D. C. This excellent survey contains all references to published data, experimental, calculated, or compiled, up to the date of publication. The published values as well as the corresponding references are included, and the more important sources are characterized as to technique of measurement, the nature of the absorber,

6

and estimate of errors. The wavelengths are indicated in the scale used by each individual author.

10. J. Leroux, "Method for finding mass absorption coefficients by empirical equations and graphs," Advances in X-Ray Analysis, Vol. V (1961), 153-160. Plenum Press, New York. Leroux proposes a self-consistent set of mass absorption coefficients, obtained by interpolation from experimental data, and using the exponential expression $\mu_0 = C. \lambda^n$ in which λ is the wavelength in angstroms, and C and n are determined empirically. Leroux gives the values for C and n for elementary absorbers and the homologous region between absorption edges. The values of C are assumed to vary in a regular fashion as a function of the atomic number of the absorber. An analysis of recently obtained experimental data shows that the equation used by Leroux is a good approximation. Deviation from the exponential equation found in older data seems chiefly due to experimental error. For a given region between homologous absorption edges, the values of C vary with the atomic number of the absorber, being approximately proportional to the third power of the atomic number. Since, however, atomic cross-sections will vary more smoothly than mass absorption coefficients, it is reasonable to expect that the value of C.A.Z -4, in which A is the atomic weight and Z the atomic number, should vary more smoothly than C.Z-3. In practice, these two expressions will be almost equivalent within the accuracy of known mass absorption coefficients. Leroux did not, however, precede his interpolation by a critical selection of experimental data, so that his data still reflect the errors of Allen's tables.

11. K. F. J. Heinrich, "X-Ray Absorption Uncertainty," in T. D. McKinley, K. F. J. Heinrich, and D. B. Wittry, The Electron Microprobe, Proc. of an Electron Probe Analysis Symposium, Wiley & Sons, New York, 1966.

Experimental data through 1963 are tested for selfconsistency and proper technique, and a selected group of data is used as the basis for interpolation, according to the method of Leroux. From the coefficients and exponents obtained by this procedure, the mass attenuation coefficients of all elements except hydrogen and helium are calculated for the K alpha, K beta, L alpha 1, L beta 1, M alpha, and M beta lines of all elements, within the wavelength region from 0.7 to 12 angstroms. Areas in which further work is most urgently needed are pointed out. The data published in the tables included in this work have been obtained by a computer, and the calculation is carried out to a tenth of a unit, regardless of the estimated accuracy. The tables are believed to be more reliable than previously mentioned sources. A typical page from a preprint of the table is reproduced at the end of this report. Experimental data obtained after the publication of the tables, in regions not documented previously, show in general a satisfactory agreement. However, in many areas, the data reproduced in the tables have been derived from very meager and uncertain experimental evidence. The graph attached at the end of the report shows the wavelength and atomic number regions within which data usable for extrapolation were found. It can be seen from this graph that not too much can be expected from an extrapolation starting with such a small amount of experimental data.

Among recent publications not contained in Stainer's bibliography or used in Heinrich's publication, the following should be consulted:

12. G. D. Hughes and J. B. Woodhouse, "X-Ray Mass Absorption Coefficients," Proc. 4th International Congress on X-Ray Optics and Microanalysis, Orsay, September 1965 (to be published).

13. R. W. Carter and R. H. Rohrer, "The Determination of Low Energy X-Ray Mass Absorption Coefficients," Final Report on Research Contract At-(40-1)-2953, Emory University, Atlanta, Georgia, 1964.

14. J. H. Hubbell and M. J. Berger, "Photon Attenuation and Energy Transfer Coefficients. Tabulation and Discussion" National Bureau of Standards, Unpublished report (1965).

15. B. A. Cooke, and E. A. Stewardson, "The Absorption of X-Rays in the Range 7-17A by Be, Mg, Al, Cu, and Ag" Brit. J. Appl. Phys., 15, 1315-1319.

4. PRESENT SITUATION

The urgent need for further data has recently induced several laboratories to perform measurements of mass attenuation coefficients. Some of these laboratories are well qualified to undertake such work, some others, not normally involved in this type of work, should be provided with guide lines in order to avoid the production of data of poor accuracy which could further increase the existing confusion. Facilities for evaluating the uniformity of the absorbers are not available in all of these laboratories. The need is greatest in the region from 10 to 100 angstroms, and it is in this region where the technical requirements are most exacting. Furthermore, some of the data will only be published in internal reports if at all. A considerable fraction of the work under way is repetitious; while confirmation of some data is desirable, it is unfortunate that much of the work concentrates on the shorter wavelength region and on absorbers such as copper, iron, and aluminum, whose mass absorption coefficients are relatively well known in the short wavelength region.

5. NEED FOR COORDINATION

Clearly, coordination of experimental work on mass absorption coefficients would present considerable advantages. A coordinating group for this type of work would be in a position to inform interested persons concerning the regions of wavelength and absorbers which are fairly well established, and indicate regions in which further work is most urgently needed. It could also provide a means for communicating to interested persons the results of investigations performed by others. Discussions with prominent workers in the field show that in general there is very little awareness of work going on in other laboratories, and even of results of concluded work, some of which is published in reports of very limited circulation.

Communication should also be established with respect to techniques of mass absorption coefficient determination. Some work is performed in an incidental manner by laboratories in need of data in a certain region. Precautions as to absorber conditions (especially uniformity of thickness) and to conditions of measurement are not always properly taken. Information on the detector acceptance angle, needed to estimate the effect of smallangle coherent scattering on a given measurement, is often omitted.

Finally, one group must undertake at periodic intervals the task of performing the interpolations for data in regions not covered experimentally, and of communicating the results of these interpolations to the user.

In numerous conversations with persons working in this field we have been assured that such a cooperative task is considered desirable, and that our Spectrochemical Analysis Group, National Bureau of Standards would be an appropriate group for such coordination. We therefore propose the following plan of operation.

6. PLAN FOR OPERATION

As a first step it is proposed that laboratories working in this field give us information concerning the areas which they are presently covering or intend to cover in the near future, and briefly outline the techniques they employ for testing the absorbers and performing their measurements. We propose to prepare an outline of suggested procedures for those laboratories which are interested in such information. Information and inquiries should be directed to Dr. Kurt F. J. Heinrich, Spectrochemical Analysis Section 310.02, National Bureau of Standards, Washington, D. C. 20234.

It is also requested that the laboratories involved give information concerning the results obtained, and give an estimation of the accuracy of the data. We suggest that copies of recent publications in the field, or data originating from other sources be made available. We would also appreciate being informed of other laboratories working in this area.

With the aid of the data that were published since the appearance of Heinrich's tables, and of further information that we expect to receive from these laboratories, a new table of interpolations will be prepared and made available to the users.

We wish to express our thanks to the following participants in the discussions concerning quantitative x-ray fluorescence analysis, which have greatly contributed to our understanding of the problems in this field.

Mr. L. S. Birks, U. S. Naval Research Laboratories, Washington, D. C.

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Mr. J. Criss, U. S. Naval Research Laboratories, Washington, D. C.

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Dr. J. Leroux, Dept. of National Health and Welfare, Ottawa, Ontario, Canada.

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Prof. Hanns Malissa, Techn. Hochschule, Vienna, Austria. Dr. Ugo Fano, NBS, Washington, D. C.

Dr. R. D. Deslattes, NBS, Washington, D. C.

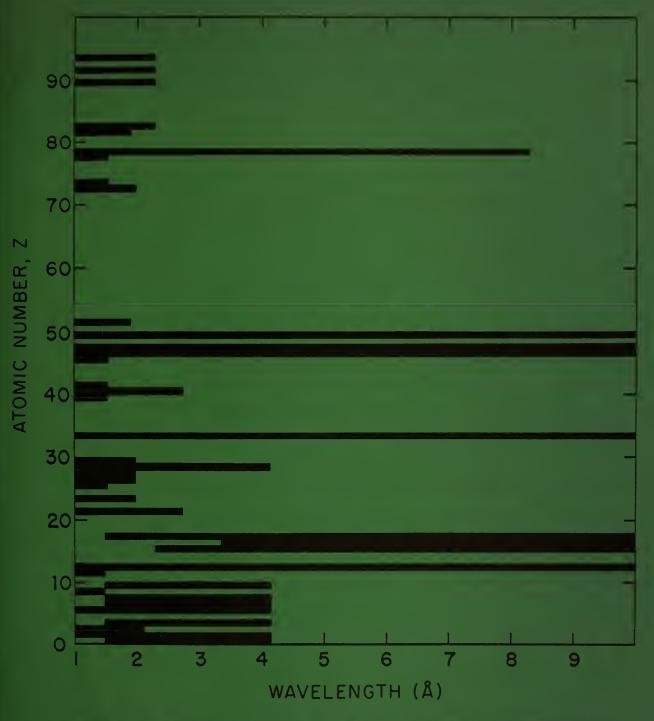
Mr. J. H. Hubbell, NBS, Washington, D. C.

Prof. B. Henke, Pomona College, Pomona, California

MAVELENGTH Absorber 3 Li										
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	1021.0	614.7	385.7	3493.2	2324.7	1593.4		÷.	582.8	431.
S	1332.5	802.2	503.4	327.9	2840.1	1949.3		980.8		530.
_	1695.9	1021.0	640.6	417.4		2370.7	1663.6	1192.8	870.3	645.
6 S	2102.5	1265.8	794.3	517.5	346.9	239.4	1965.5	1411.0		765.
17 CL	2578.4	1552.3	914.0	634.6	425.4	293.6	207.1			898.
AR	3132.2	1885.7	1183.2	770.9	516.8	356.6	251.5	181.2	1390.2	1033.
19 K	3729.2	2245.1	1408.8	917.8	615.3	424.6	299.5	215.8	158.1	1189.
20 CA	4412.8	2655.7	1667.0	1086.0	728.1	502.4	354.4	255.3	187.1	139.
	5162.8	3120.3	1957.9	1275.5	855.1	590.1	416.2	299.9		163.
22 11	6056.7	3646.4	2288.0	1490.6	£*666	689.6	486.4	350.4	256.8	191.
1	6939.3	4177.7	2621.4	1707.8	1144.9	790.1	557.2	401.5	294.3	219.
CR	7943.0	4782.0	3000.5	1954.8	1310.5	904.3	637.8	459.6		250.
25 MN	9041.8	5443.6	3415.6	2225.3	1491.8		726.1	523.1	383.4	285.
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-	11464.5	6902.1	4330.8	2821.5	1891.5	1305.3	920.6	663.3	486.2	362.
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AS	2683.6	1655.2	6782.3	5163.9	3461.8	338		1214.0		662.
	2940.7	1813.7	7370.6	5621.8	3768.7	600	1634.3	1321-6	968.7	721.
	3216.6	1983.9	1272.7	5238.5	4111.4	2837.2		441.	1056.7	786.
36 KR	3511.3	2165.6	1389.4	5685.7	4461.1	3078.5	2171.3	1564.4	1146.6	853.
	3824.8	2359.0	1513.4	1006.3	4111.4	3339.6	2355.4	1697.0	1243.9	926.
S	4150.8	2560.1	1642.4	1092.1	4454.0	3595.7	2536.1		1339.3	997.3
39 Y	4501.9	2776.6	1781.4	1184.5	809.3		2730.6	1967.4	1442.0	
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41 AB	5260.6	2	2081.6	1384.1	945.7	664.2	2702.8	277.	1669.5	1243.
		3499.8	2245.3	1493.0	1020.1	716.5	2035.2	2393.4	1756.2	1309.
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	6564.8	4049.0	2597.6	1727.2	•		594.4	1692.8	÷.	
	7047.6	4346.7	2788.6	1854.2	1266.9	889.9	638.2	467.0		1600.8
6. PD	7543.0	4652.2	2984.6	1984.6	1356.0	52		499.8		ŝ
AG		977.	193.		1450.7	1018.9	30	534.7	397.8	1533.5
	~	5321.3	13	2270.0	1551.0	1089.4	781.2	571.7		1162.

MASS ABSORPTIUN CUEFFICIENTS FOR K ALPHA LINES

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are well accounted (mark bars).

U.S. DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20230

OFFICIAL BUSINESS

POSTAGE AND FEES PAID U.S. DEPARTMENT OF COMMERCE