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APPLICATION OF THE MÖSSBAUER TECHNIQUE TO THE STUDY OF PROBABLE LUNAR AND PLANETARY SURFACE MATERIAL AND TO THE STUDY OF RETURNED LUNAR SURFACE SAMPLES

Final Report

for

Contract No. NAS9-8083 IITRI V6005-4

Prepared by

C. L. Herzenberg D. L. Riley and R. B. Moler

of

IIT Research Institute 10 West 35th Street Chicago, Illinois 60616

## for

National Aeronautics and Space Administration Manned Spacecraft Center Lunar and Earth Sciences Division Houston, Texas 77058

February 1971

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## FOREWORD

This document is IITRI Report V6005-4, the final report on Contract NAS9-8083, entitled "Application of the Mössbauer Technique to the Study of Probable Lunar and Planatary Surface Material and to the Study of Returned Lunar Surface Samples," covering the period from May 1, 1969 through January 31, 1971.

This report contains only information covering the period from February 1, 1970 through January 31, 1971, for the analysis of Apollo 12 samples. The work carried out prior to February 1, 1970 is included by reference to Report No. IITRI V6005-3 which provides a detailed description of all previous work.

> Respectfully submitted, IIT RESEARCH INSTITUTE RB. Moler-

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C. A. Stone, Director Physics Research Division

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# PRELIMINARY RESULTS FROM MÖSSBAUER INSTRUMENTAL ANALYSIS OF APOLLO 12 LUNAR ROCK AND SOIL SAMPLES

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# ABSTRACT

Nuclear gamma resonance measurements show that iron is generally abundant in Apollo 12 lunar samples, and is predominantly in the ferrous state.

Substantial differences among the spectra of Apollo 12 crystalline rocks indicate considerable diversity in the phase distribution of iron, reflecting significant differences in modal mineralogy. Two different magma types are distinguished on the basis of Mössbauer analyses. Mössbauer analyses indicate that Apollo 12 rocks are generally olivine rich and ilmenite poor, with respect to Apollo 11 crystalline rocks.

Spectra of various Apollo 12 soils are remarkably similar to each other, but different from Apollo 11 soil spectra, suggesting that the Mössbauer spectra provide regional soil signatures. Ilmenite and metallic iron are less abundant, and olivine more abundant in Apollo 12 soils relative to Apollo 11 soils. Possible evidence for a resonance associated with the cryptic component in the fines has been found.

The unusual microbreccia 12013 exhibits resonances indicating the presence of the major lunar minerals, with both olivine and ilmenite less abundant than in any other Apollo 12 sample examined. In the spectra of this particular sample we observe anomalous resonant absorption features, which we tenatively associate with the dark lithology and with a history of intense shock.

" Co-author, but not co-investigator.

This paper presents a preliminary summary of results obtained from Mössbauer effect spectrometric analysis of Apollo 12 samples. The complete report (to be published later) will be a sequel to our earlier major report on Mössbauer effect spectrometry of Apollo 11 samples [Herzenberg and Riley, 1970a].

The studies reported here proceeded by examining the nuclear gamma resonance of the nuclide Fe<sup>57</sup> which is naturally present in lunar material. The general procedures and equipment are similar to those employed in our previous study of Apollo 11 material [Herzenberg and Riley, 1970a, 1970b].

Rock chips, specially mounted thin sections (or probe mounts), and rock powder samples representing four lunar crystalline rocks returned on Apollo 12 have been studied. Powder samples representing one microbreccia (12013) and three soils have also been studied. Rock chips were analyzed using Mössbauer scattering spectrometry [Herzenberg and Moler, 1970; Herzenberg et al., 1971], while measurements on the other specimens were conducted primarily in the transmission mode.

A spectrum from Apollo 12 sample 12002, a medium grained holocrystalline basalt, is shown in Figure 1. This nuclear gamma resonance spectrum was measured in transmission through a thin sample of powder. From the intensity and location of the absorption resonances it may be concluded that this Apollo 12 sample contains an appreciable abundance of iron, and that the

iron is predominantly in the  $Fe^{2+}$  oxidation state. It also follows from the particular resonances present in this spectrum that this Apollo 12 specimen contains as major minerals pyroxene, clivine, and ilmenite, which were all previously identified in Apollo 11 material [Herzenberg and Riley, 1970a, 1970b]. The relative intensities of the resonances indicate however, that the distribution of iron among the mineral phases is quite different than in Apollo 11 material. This implies that the modal mineralogy is also quite different than for Apollo 11 crystalline rocks. On the basis of computer fits to room temperature spectra (using up to 8-lines, or 25 free parameters), and a 6-line, 19 free parameter computer fit to a spectrum measured with the sample at 80°K, as well as additional areal analyses of these and other spectra without the use of computer fitting, we have obtained from the Mössbauer absorption spectra the intercrystalline phase distribution of iron in this rock (Table I). From the data presented in Table I, we have also calculated the weight percentages of minerals present in this sample (Table II). Among the significant differences between Apollo 11 and Apollo 12 material that may be inferred from this spectrum and the tabulated analytic data, are the considerably reduced ilmenite content and the increased olivine content of the Apollo 12 material relative to Apollo 11 material.

Figure 2 shows a spectrum measured for Apolle 12 crystalline rock 12063, a fine grained ilmenite microgabbro. This Apollo 12 rock shows spectra more like those of Apollo 11 material than does any other Apollo 12 sample we have examined. In the classification of Apollo 12 crystalline rocks into two magma types proposed by J. L. Warner [1970] [Wakita et al., 1971], this is a magma type 2 rock. It is of interest to compare this spectrum with spectra of magma type 1 rocks, such as the Apollo 12 crystalline rock 12020 (Figure 3). It is clear that the ilmenite resonances are much reduced in the magma type 1 rocks, reflecting an ilmenite content lower by a factor of 2 (see Tables I and II). The relative ease of distinguishing rocks of the major Apollo 12 magma types on the basis of their Mössbauer spectra suggests this technique may have a future role in studying lunar volcanic stratigraphy.

Figure 4 shows a Mössbauer hyperfine absorption spectrum for a sawcuttings sample for the unusual microbreccia 12013. 12013 is a complex heterogenous rock type exhibiting a unique chemical composition, and age measurements indicate it contains exceptionally old lunar material [LSPET, 1970; Earth and Planetary Science Letters Special Issue, 1970]. The spectrum, while similar to the spectra of other Apollo 12 samples, exhibits marked differences. Contributions from Fe<sup>2+</sup> in pyroxenes, olivine, and ilmenite are present in the spectrum of this microbreccia; however, the results indicate that less olivine and less ilmenite are present in this microbreccia than are present in any other Apollo 12 sample we have examined

(including both crystalline rocks and fines). Additional spectra measured over a greater velocity range show more intense metallic iron resonances than have been observed for Apollo 12 crystalline rocks. In addition to the spectral features which this sample shares with the Apollo 12 crystalline rocks, there are certain unusual features which, while not dominant in the spectrum, are quite apparent when detailed comparisons are made between spectra from this rock and spectra from other samples. These features include a notable excess of resonant absorption in the spectral region occupied by the lower members of the quadrupole doublets for the major paramagnetic minerals, as compared with the region occupied by the corresponding upper resonances. Another feature evident in the spectrum is the excess absorption throughout the region between the paramagnetic doublets, which cannot be accounted for in terms of the resonances observed in the crystalline rocks alone. Minor absorption excesses are observable just outside the major paramagnetic peaks also. The origin of these anomalous features is still under investigation. Our previous studies on intensely shocked terrestrial basalt [Herzenberg and Riley, 1970c, and unpublished results], suggest that some of these features may have originated during a history of intense shock, which is in agreement with other observations of the presence of shock features in this rock [James, 1971].

Mössbauer hyperfine spectra of the three lunar soil samples (12032, 12033, and 12037) are shown in comparison in Figure 5. The similarity in the spectra of the three Apollo 12 soils is evident. The distinctively different character of

these spectra from the spectra characteristic of Apollo 11 soils [Herzenberg and Riley, 1970a, 1970b] may also be noted.

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Preliminary estimates for the phase distributions of iron in these lunar soils, as determined from these and other spectra, are given in Table III. Comparison with corresponding results for Apollo 11 soils [Herzenberg and Riley, 1970a] shows that the Apollo 12 soils have a distinctly different composition. The Apollo 12 soils, for example, are reduced in ilmenite content by a factor of approximately 2.5 relative to the Apollo 11 soils. Furthermore, the Apollo 12 soils contain considerably less magnetic material than do the Apollo 11 soils. Thus, we find that the Apollo 12 soils are reduced in metallic (alpha) iron content by about a factor of two relative to Apollo 11 soils. As was observed in the case with Apollo 11 samples [Housley et al., 1970], the ratio of metallic iron to iron in troilite appears to be systematically greater in the fines than in the crystalline rocks. It is also of interest that all the spectra suggest smaller amounts of glasses that do not show devitrified resonance characteristics, in comparison with Apollo 11 fines [Herzenberg and Riley, 1970a].

As indicated in Table III, the Apollo 12 lunar fines spectra all show a minor anomalous feature not generally present in the crystalline rock spectra, namely, an absorption excess in the low velocity region (the region occupied by the lower members of the paramagnetic doublets). The origin of this feature is still under study, and the possibility that it may be an experimental artifact has not yet been ruled out. We find it of some interest that this feature, which is generally

absent from the spectra of the crystalline rocks, is present in the spectrum of rock 12013 as well as in the spectra of all of the Apollo 12 fines samples studied. This fact leads us to the supposition that this feature might be associated with the so-called "cryptic component," which has been identified as present in the soils in addition to the contribution from crystalline rock materials, and which appears to be similar in elemental composition to the dark lithology in rock 12013 [Schnetzler et al., 1970]. If this association of anomalous excess resonant absorption in the Mössbauer spectra of the lunar soils with the cryptic component in the soils is justified, then a further understanding of the nature and origin of the cryptic component may be forthcoming from a more detailad study of the resonance spectra. Furthermore, as there is evidence that the amount of cryptic component in individual soils can be correlated with the different model ages of these soils [Bottino et al., 1970], this presents the possibility of a correlation of features of the Mössbauer spectra with the ages associated with lunar soils.

It is clear from the Apollo 12 soils spectra and also from Table III that only rather minor differences among these soils exist with respect to the distribution of iron among different mineral phases, a result which seems rather surprising in view of the fact that these samples represent three different soils, gathered in different locations up to 240 meters apart

on the lunar surface. These soils have different characteristics and one of them (12033, a light grey volcanic ash) is visually distinctively different from the other soils. Their Mössbauer spectra, however, are remarkably similar. This evidence suggests the possibility that the phase distribution of iron in lunar soils (as indicated by their Mössbauer spectra) may represent a regional characteristic, nearly constant for the near-surface regolith over regions corresponding in size to distances of perhaps the order of a kilometer or more, but showing considerable variation over distances comparable to the separation of the landing sites, and to the dimensions of the major visual features of the lunar nearside.

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### Table I

## PHASE DISTRIBUTION OF IRON IN APOLLO 12 LUNAR CRYSTALLINE ROCKS\*,\*\*

Mineral Phase	12002	12004	12020	<u>12063</u>
Metallic iron	1.8 <u>+</u> 0.5	0.8 <u>+</u> 0.5 <sup>a</sup>	1.0 <u>+</u> 0.5	1.5 <u>+</u> 0.5 <sup>a</sup>
Troilite	1.0 ± 0.3	$1.7 \pm 0.6^{a}$	1.0 <u>+</u> 0.5	1.3 <u>+</u> 0.6 <sup>a</sup>
Ilmenite	7.2 <u>+</u> 1.0	$6.3 \pm 1.0$	5.9 <u>+</u> 1.0	13.7 <u>+</u> 2.0
Pyroxene	64.8 <u>+</u> 10.0	67.4 <u>+</u> 10.0	74.2 + 8.0	69.5 + 8.0 - 2.0
Olivine	25.2 <u>+</u> 8.0	23.8 <u>+</u> 8.0	17.9 + 3.0 - 7.0	14.0 + 3.0 - 7.0
Total silicate (Fe <sup>2+</sup> in pyroxenes olivines, and glasses)	90.0 <u>+</u> 2.0	91.2 + 3.0 - 1.0	92.1 + 1.0 - 3.0	83.5 + 3.0 - 2.0

\* . Percent of total resonant absorption associated with each mineral phase is tabulated.

Tabulated data is based primarily on results obtained for thin sections (probe mounts), except for the case of 12002, for which only a powder sample was available. \*\*

These entries have been supplemented by data from powder а samples and rock chips.

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# Table II

# WEIGHT PERCENTAGES OF MINERALS IN APOLLO 12 CRYSTALLINE ROCKS FROM MOSSBAUER INSTRUMENTAL ANALYSIS\*

Mineral Phase	12002	12004	<u>12020</u>	<u>12063</u>
Metallic iron	0.29	0.14	0.17	0.25
Troilite	0.25	0.48	0.26	0.34
Ilmenite	3.13	3.07	2.61	6.22
Pyroxene	50.2	58.4	59.8	56.2
Olivine	17.0	19.2	13.4	8.9
Feldspar <sup>a</sup>	29.2	18.8	24.2	28.2

Sample Number

\* Based primarily on thin section spectra.

a Obtained by difference.

# Table III

# PHASE DISTRIBUTION OF IRON IN APOLLO 12 FINES<sup>\*</sup>

Mineral Phase	12037,12	12032,63	<u>12033,93</u>
Metallic iron	2.4 <u>+</u> 1.0	2.7 <u>+</u> 1.0	$2.4 \pm 1.0$
Troilite	1.3 <u>+</u> 0.7	0.8 + 0.6	0.8 <u>+</u> 0.6
Ilmenite	9.1 <u>+</u> 1.7	9.2 <u>+</u> 1.7	8.6 <u>+</u> 1.7
Pyroxene <sup>a</sup>	45.8 <u>+</u> 10.0	47.4 <u>+</u> 10.0	48.6 <u>+</u> 10.0
Glass <sup>a</sup> , <sup>b</sup>	22.8 <u>+</u> 14.0	23.6 <u>+</u> 14.0	24.1 <u>+</u> 14.0
Olivine <sup>a</sup>	12.3 <u>+</u> 6.0	11.6 <u>+</u> 6.0	10.1 <u>+</u> 6.0
Unidentified resonance <sup>C</sup>	6.3 <u>+</u> 1.5	4.7 <u>+</u> 1.5	5.4 <u>+</u> 1.5
Total silicate (Fe <sup>2+</sup> in pyroxenes, olivines,	80.9 <u>+</u> 3.0	82.6 <u>+</u> 3.0	82.8 <u>+</u> 3.0

\* Percent of total resonant absorption associated with each mineral phase is tabulated.

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- a Silicate separations into olivine, pyroxene, and glass contributions are based on room temperature and (for 12037) 80°K measurements, and comparison with both Apollo 11 fines and simulated lunar sample spectra.
- b Entry corresponds to materials vitreous on a molecular scale, and does not include devitrification products.
- c This represents an absorption excess in the low velocity spectral region. See discussion further in text.

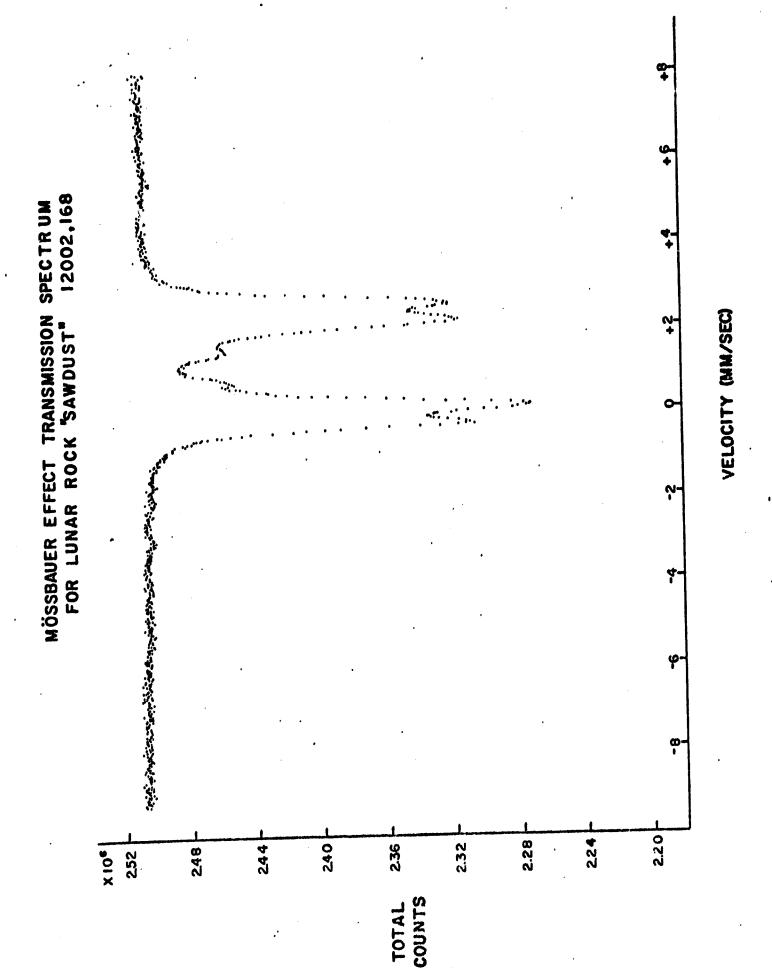
## LEGENDS FOR ILLUSTRATIONS

## Figure

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- 1 Mössbauer hyperfine absorption spectrum for lunar rock powder sample 12002,168, measured at room temperature.
- 2 Mössbauer hyperfine absorption spectrum for lunar rock powder sample 12063,100, measured at room temperature.
- 3 Mössbauer hyperfine absorption spectrum for lunar rock probe mount 12020,56, measured at room temperature.
- 4 Mössbauer hyperfine absorption spectrum for powder (sawcuttings) sample of lunar rock 12013, measured at room temperature.
- 5 Mössbauer hyperfine absorption spectra for three Apollo 12 soils compared: 12032 (upper); 12033 (middle); 12037 (lower).



. Figure 1

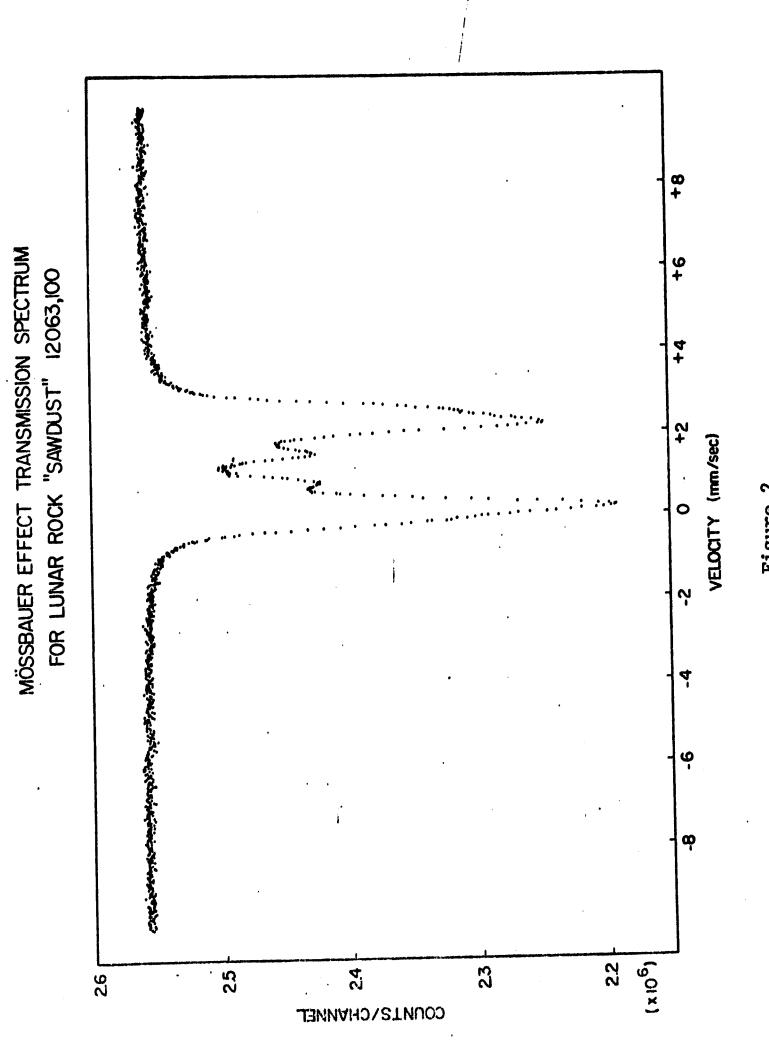
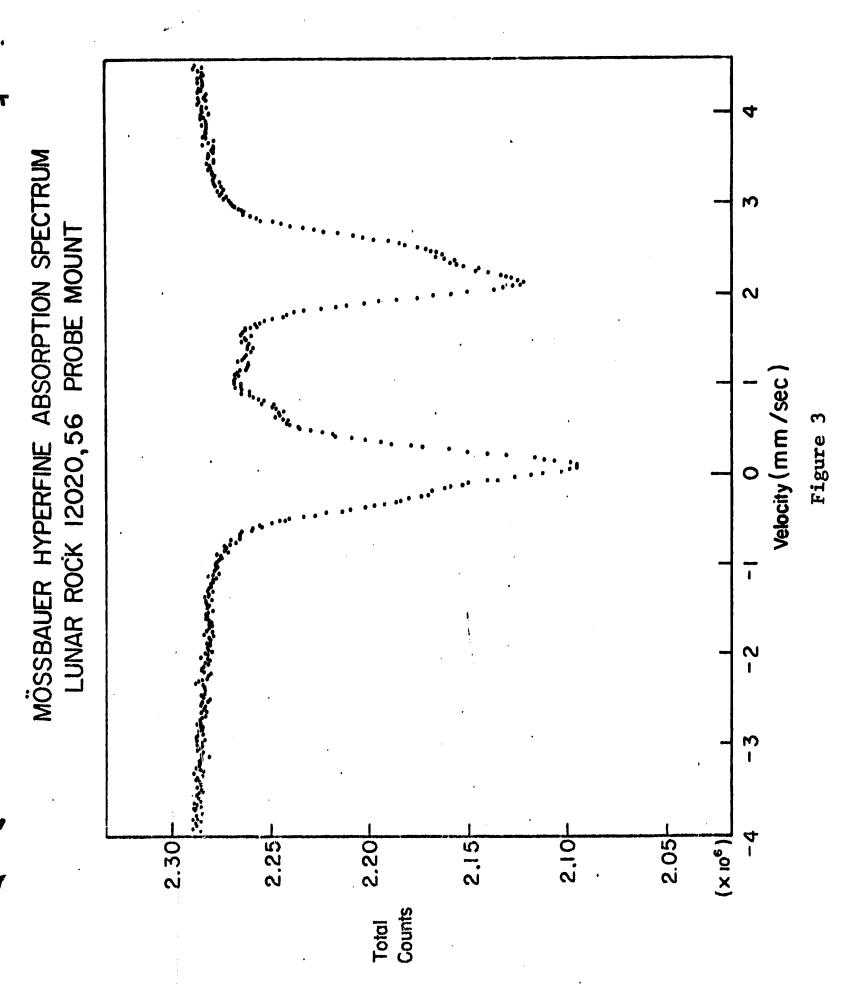


Figure 2



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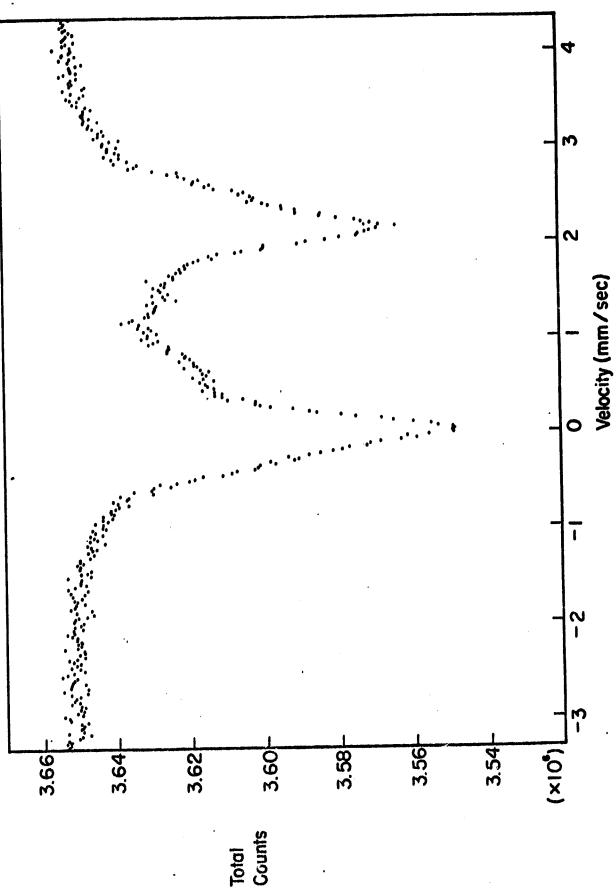


Figure 4

