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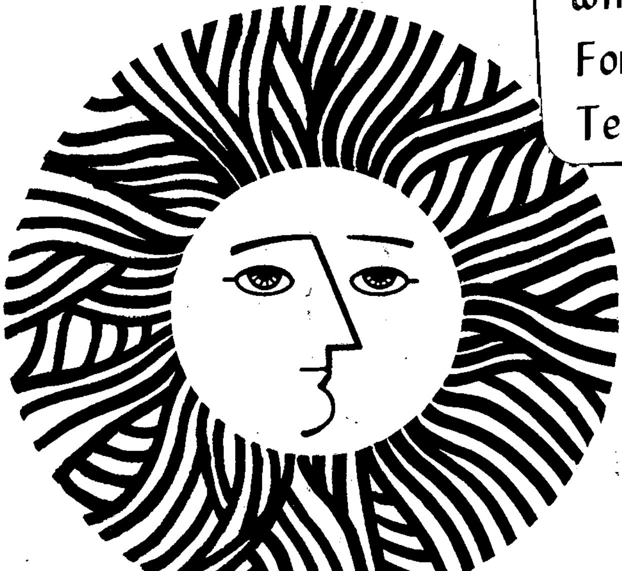
September 1981



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DISCLAIMER

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ABSTRACT

Triplet splitting in the (7,2) band of the B $^3\Sigma_u^-$ - X $^3\Sigma_g^-$ system of S_2 has been detected by absorption of a Cr(I) atomic emission line at 3018 Å, using a technique called Tunable Atomic Line Molecular Spectroscopy. The Cr line was split into Zeeman components, and a differential absorption measurement was obtained as a function of magnetic field strength. Five transitions between triplet split levels of S_2 were calculated to be nearly resonant with the chromium emission line, and used to interpret the absorption spectrum observed by magnetic scanning. A resolution greater than 6 x 10^4 was demonstrated.

This research was supported by the Assistant Secretary for Environment, Office of Environmental Compliance and Overview, Environmental Safety and Engineering Division of the U.S. Department of Energy under Contract No. W-7405-ENG-48.

I. INTRODUCTION

The electronic spectrum of S_2 has been the subject of numerous investigations[1]. Particular attention has been devoted to the extensive B $^3\Sigma_u^-$ - X $^3\Sigma_g^-$ system. The coupling of rotation and electronic motions for both $^3\Sigma$ states, plus the interaction between spins, have the effect that each energy level given by the quantum number K (total angular momentum apart from spin) is split into three levels, F_1, F_2, and F_3, with J = K + 1, K, and K - 1 respectively (J is the total angular momentum quantum number)[2]. As a result of this triplet splitting, each band in the B-X system consists of 10 branches: six main branches for which $\Delta K = \Delta J$, and four satellite branches for which $\Delta K \neq \Delta J$.[3]

The main branches are denoted by R_i (K) and P_i (K), where i indexes the F_i levels involved. These branches are easily observed in both absorption and in emission using high resolution spectrographs[4-7]. The four satellite branches, $^TR_{31}$, $^NP_{13}$, $^RP_{31}$, and $^PR_{13}$, are generally weaker[8], and have been observed in emission for some bands of the B - X system by Barrow and co-workers[9]. Rotational satellites have also been observed by selectively excited fluorescence, using either an argon ion laser line (5145 Å)[10], or atomic emission lines: Zn (3076 Å) and Cd (3261 Å)[11].

We report here the observation of triplet splitting in the (7,2) band of the B $^3\Sigma_u^-$ - X $^3\Sigma_g^-$ system of S_2 by absorption of a Cr (I) atomic emission line at 3018 Å. The Cr emission line is split into Zeeman components by placing the light source in a magnetic field. A direct measurement of the S_2 absorption in the neighborhood of the Cr atomic emission line is obtained by magnetic tuning of the Zeeman components. In this manner, a high resolution spectrum of S_2 can be obtained without the need of a high resolution spectrometer.

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The magnetic scanning of a single Zeeman component of an atomic emission line was first used by Bitter and co-workers[12] for investigating the hyperfine structure and isotope shift of the resonance radiation of mercury. This technique was utilized by Hadeishi and McLaughlin[13] to develop a new type of atomic absorption spectrometer to detect trace mercury, and utilizing the Zeeman effect for background correction. Zeeman effect based atomic absorption spectrometry (ZAA) has been developed further to include the detection of numerous other atomic species[14,15].

Zeeman scanning of molecular line profiles in flames was first demonstrated by Hollander and Broida[16]. Recently, ZAA spectrometry has been extended to the detection of small molecules which exhibit sharp rotational electronic structure[17]. This new technique, called Tunable Atomic Line Molecular Spectroscopy (TALMS), has been used to detect NO, NO_2, SO_2, and HCHO[17,18]. We are interested in applying this technique to the detection and measurement of species present in combustion environments such as flames.

II. EXPERIMENTAL

In a magnetic field, an atomic energy level of total angular momentum quantum number J is split into 2J + 1 states which are identified by a magnetic quantum number M_J, where $M_J = J, J-1, \ldots -J$.[19] Transitions for which $\Delta M_J = 0$ result in the π components of the Zeeman splitting pattern, and are not shifted (or shifted only slightly) from the zero-field value ν_o of the transition. Transitions for which $\Delta M_J = \pm 1$ are called the $\sigma\pm$ components, and are shifted symmetrically to lower and higher energies about ν_o. The π and $\sigma\pm$ components also differ in their polarization. With transverse observation (perpendicular to the direction of the magnetic field), the π components are linearly polarized parallel to the magnetic field, while the $\sigma\pm$ components are linearly polarized perpendicular to the field. For longitudinal observation (parallel to the magnetic field), π components are not observed, and $\sigma\pm$ components are circularly polarized: the high frequency $\sigma+$ component is circularly polarized in a counterclockwise direction, while the low frequency $\sigma-$ component is circularly polarized in the opposite direction.

The splitting and polarization of spectral lines form the basis for detection of molecules by Tunable Atomic Line Molecular Spectroscopy[17,18]. By varying the strength of the magnetic field, one of the Zeeman components of the atomic emission line is tuned into coincidence with a sharp transition in the electronic absorption spectrum of the molecule to be detected, in this case S_2, while the other component is shifted off resonance. The matching Zeeman component indicates the extent of absorption by S_2 plus background absorption due to other species present, while the unmatched component indicates background absorption only. A differential measurement of the absorptions of the matched and unmatched Zeeman components provides a quantitative measurement of the molecule of interest.

A diagram of the experimental apparatus is shown in Figure 1. The light source is a modification of a magnetically confined arc lamp described by Hadeishi and Anderson[20]. The cathode is made by spot welding several turns of nichrome tape (Tophet C, 16% Cr, 60% Ni, and 24% Fe) to the tip of a stainless steel screw, and the anode is constructed from a modified automobile spark plug. Argon is flowed through the light source at a pressure of less than 2 torr. A d.c. discharge of 50-150 mA to the cathode excites the emission spectrum of Cr, Ni, and Fe simultaneously. The atomic emission passes through a hole drilled in one pole of the electromagnet

(Varian model V-4004) for longitudinal observation. The light is focused through a 6 cm quartz cell adapted with a finger containing sulfur (sublimed sulfur, Mallincrodt). The finger is healed in an oil bath to T_f = 200°C. At this temperature, the vapor pressure of sulfur is about 2 torr, and is composed principally of the species S_8, S_6, S_4, and S_2[21,22]. The cell is heated to T_c = 750°C to insure that all the sulfur vapor is dissociated into S_2[22].

The variable phase retardation plate consists of a block of fused quartz mounted at a 45° angle in a magnetic clamp.[23] The clamp is actuated by a pair of drive coils, and the stress on the quartz block produces a retardation of the phase of the light passing through it. By driving the clamp with an a.c. current, a variable retardation effect is produced, which, in combination with the fixed linear polarizer in front of the monochromator (McPherson model 218, grating blazed at 2000 Å, 1200 grooves/mm), allows for the alternate transmission of the matched and unmatched Zeeman components to the photomultiplier tube (Hamamatsu Type YA7122). The signal is processed electronically and displayed on a strip chart recorder.

III. RESULTS AND DISCUSSION

The emission spectrum obtained by exciting the nichrome tape could be readily assigned to Ni, Cr, and Fe using tables of line spectra of the elements[24]. Several lines in the neighborhood of 3000 Å were found to result in differential absorption signals: Cr at 3000.9, 3005.1, and 3017.6 Å, and Fe at 3008.1 Å. The Cr (I) line at 3017.57 Å was selected because of its higher relative intensity, and was assigned to the transition $a\,{}^5D_3 \leftarrow y\,{}^5F^0_4$ at 33,129.6 cm^{-1}.[25]

The upper and lower energy levels of this atomic line are split into 9 and 7 new states respectively by the external magnetic field.[19] The $\Delta M_J = 0, \pm 1$ selection rule results in a total of 21 transitions in three groups of seven closely spaced components, corresponding to σ-, π, and σ+. With the optical axis parallel to the direction of the magnetic field, only the σ- and σ+ groups of components appear. The separation between these two groups is 0.088 cm^{-1}/kgauss, while the separation between the individual components of σ- and σ+ is 0.0066 cm^{-1}/kgauss. Furthermore, at a discharge plasma temperature of 500 K [18], the Doppler width (FWHM) of the individual components is 0.074 cm^{-1}. Therefore, the seven closely spaced components that comprise each of σ- and σ+ appear as single broad and asymmetric lines, and are not resolved into individual components except at high field strengths.

The dependence of the differential absorption signal due to S_2 on the strength of the external magnetic field is shown in Figure 2. As the field is increased the signal decreases and passes through zero at about 8 kgauss. Increasing the field strength further results in differential absorption signals of opposite sign to those obtained at lower fields. This change in polarity implies that at lower field strengths, one of the circularly polarized Zeeman components of the Cr (I) line, σ+ or σ-, is in resonance with a molecular absorption of S_2, while at higher fields, this component falls off resonance while the other σ component tunes into a <u>different</u> absorption of S_2.

The σ components were assigned to the positive and negative differential absorption signals as follows. First, the absorption cell is replaced by a second linear polarizer oriented at 90° to the other polarizer. The variable phase retardation plate is replaced with a block of fused quartz mounted in a small vise at 45° relative to the crossed polarizers. Stress is applied until the intensity of the transmitted light is half the intensity transmitted when both polarizers are parallel and no stress is applied to the block. The second linear polarizer is then replaced by the absorption cell, and a 5 kgauss field is applied to the chromium emission line. At this field strength, a positive differential absorption signal is observed, and the transmitted intensities I and I_o are recorded as the sulfur is heated to constant temperature. The measurement of I/I_o is repeated with the quartz plate positioned at 90° to the initial orientation, and the entire experiment is repeated at 13 kgauss, where a negative signal is observed. In this manner, it was determined that the positive absorption signals correspond to preferential overlap of the $\sigma-$ component with an absorption of S_2, while the negative signals result from the overlap of the $\sigma+$ component with different absorptions of S_2 at higher frequency.

Interpretation of the magnetic field dependence requires a detailed examination of the electronic spectrum of S_2 in the neighborhood of 3018 Å. The frequency of the Cr (I) atomic line at 33,129.6 cm^{-1} could overlap with a low K line in the v', v" = 7,2 band, or with a high K ($K\sim60$) line in the (4,0) band. Rotational analyses of either band have not been published[26].

The energy level diagram for K" = 13 in the (7,2) band of S_2 is shown in Figure 3. The transitions which are allowed between the triplet split levels are also shown. Frequencies for the six main branches and four satellite branches were calculated for K" = 1 to 60 using the expressions for F_1, F_2, and F_3 given in Reference 2. These expressions are functions of two spin splitting constants, γ and λ, which are functions of the vibrational quantum number v, and are different for each of the electronic states. Spectroscopic constants for both the $B\ ^3\Sigma_u^-$ and $X\ ^3\Sigma_g^-$ states were taken from Huber and Hertzberg[27], and γ and λ for the ground state from Barrow and Yee[28]. The spin splitting constants for $v' = 7$ in the excited B state, however, are not well known. Meyer and Crosley[11] reported a value of $\lambda = -4.7$ for the $v' = 0$, 2, and 4 states, and concluded that $v' = 7$ is also inverted ($\lambda < 0$) from published data on bands belonging to $v' = 7$. Barrow and du Parcq[1] reported $\gamma = 0.05$ for $v' = 0$, 1, and 4. These two values, $\lambda = -4.7$ and $\gamma = 0.05$, were assumed for $v' = 7$ and used to calculate the frequencies of the ten branches in the (7,2) band of S_2[29].

Five transitions were calculated to be within 1.5 cm^{-1} of ν_o = 33,129.6 cm^{-1}, the frequency of the Cr (I) atomic line in the absence of an external magnetic field. These transitions are shown at the top of Figure 4, and are R_2 (13) at 33,129.4 cm^{-1}, P_3 (13) at 33,129.7 cm^{-1}, R_1 (21) at 33,130.0 cm^{-1}, $^RP_{31}$ (21) at 33,130.2 cm^{-1}, and $^TR_{31}$ (27) at 33,130.6 cm^{-1}. Since the total pressure in the absorption cell is about 2 torr, these absorptions are predominantly Doppler broadened, and have a Doppler breadth (FWHM) of 0.095 cm^{-1} at 750°C. The relative intensities of these five absorptions were calculated using the results of Tatum and Watson[8].

Figure 4 also shows the Zeeman components of the chromium atomic line at several magnetic field strengths. At 2.5 kgauss, σ- and σ+ overlap R_2 (13) and P_3 (13) respectively. The differential absorption signal at this field strength is positive (see Figure 2), and is due to the more favorable overlap of σ-. As the field increases, σ- and σ+ spread apart, and eventually overlap equally the different absorptions of S_2; at 8 kgauss, the differential absorption signal is zero. Increasing the field to 12.5

kgauss moves σ- out of resonance with R_2 (13), while σ+ overlaps first R_1 (21) and then $^{R}P_{31}$ (21). The absorption signal is negative since the roles of σ- and σ+ as monitors of S_2 and of background are reversed. The σ+ component has a width of about 0.22 cm^{-1} at this field strength, and is too broad to resolve the two S_2 absorptions. As the field is increased further to 21 kgauss, the low frequency σ- component remains off resonance with any of the rotational branches of S_2 (the next branch is calculated to be $^{N}P_{13}$ (9) at 33,127.9 cm^{-1}), while σ+ begins to overlap $^{T}R_{31}$ (27). This overlap results again in a negative differential absorption signal.

IV. CONCLUSIONS

Since a complete experimental rotational analysis of the (7,2) band of S_2 is not available, unambiguous assignment of the differential absorption signals shown in Figure 2 to discrete absorptions of S_2 is difficult.[30] Nevertheless, the magnetic scanning technique described here is capable of resolving individual branches of the (7,2) band which arise due to triplet splitting in the B $^3\Sigma_u^-$ - X $^3\Sigma_g^-$ system. A resolution better than 6 x 10^4 is demonstrated.

Clearly, the Cr emission line at 3018 Å is suitable for the detection of S_2 in spite of its complex Zeeman splitting pattern. However, for high resolution spectroscopy and lineshape determination, a better choice would be the Mg (II) line at 2928.63 Å, which was used by Durand[31] to excite the fluorescence spectrum of S_2. This line overlaps the R_1 (39) transition at 34,135.50 cm^{-1} in the (8,1) band of S_2,[32] and can be assigned to the atomic transition $^2P_{1/2}$ - $^2S_{1/2}$ [33]. In a direction parallel to the magnetic field, the Zeeman splitting pattern for this line consists simply of single σ- and σ+ components.

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FIGURE CAPTIONS

Figure 1 Schematic diagram of the experimental apparatus: EM electromagnet; S light source; L quartz lens; C absorption cell; VPRP variable phase retardation plate; P polarizer; M monochromator; D detector.

Figure 2 Differential absorption signal due to S_2 as a function of the strength of the external magnetic field. The shift of $\sigma\pm$ components is $\Delta V_s = 0.0438\ \mathrm{cm}^{-1}/\mathrm{kgauss}$.

Figure 3 Triplet splitting of the rotational levels of S_2. The $v'' = 2$ level is normal while the $v' = 7$ level is inverted. The six main branches ($\Delta K = \Delta J$) and four satellite branches ($\Delta K \neq \Delta J$) are shown for $K'' = 13$.

Figure 4 Calculated absorption spectrum of S_2 in the neighborhood of 3018 Å. Also shown in the overlap of the $\sigma\pm$ components of the Cr emission line with the S_2 absorptions at four magnetic field strengths.

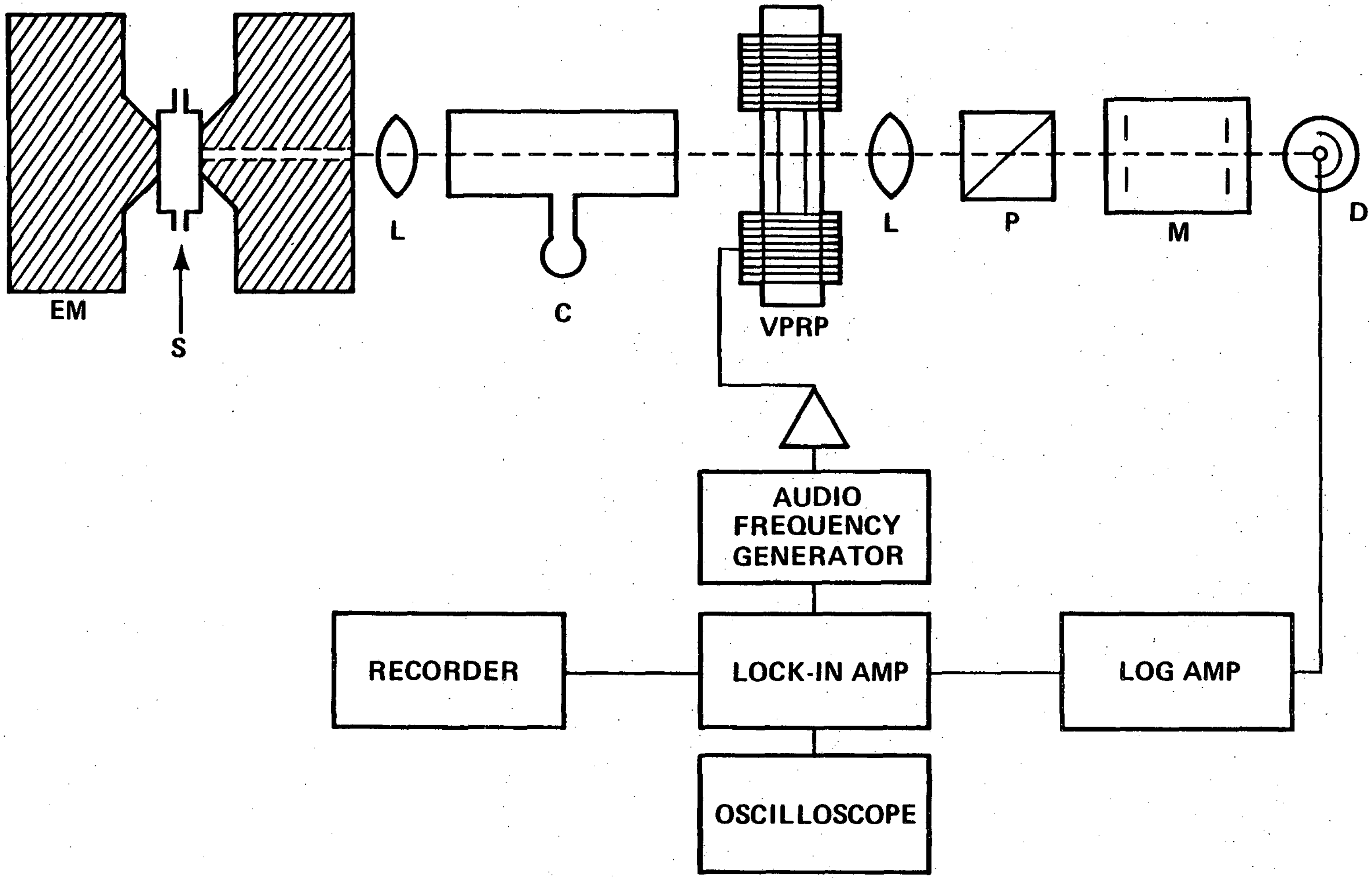

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Figure 1

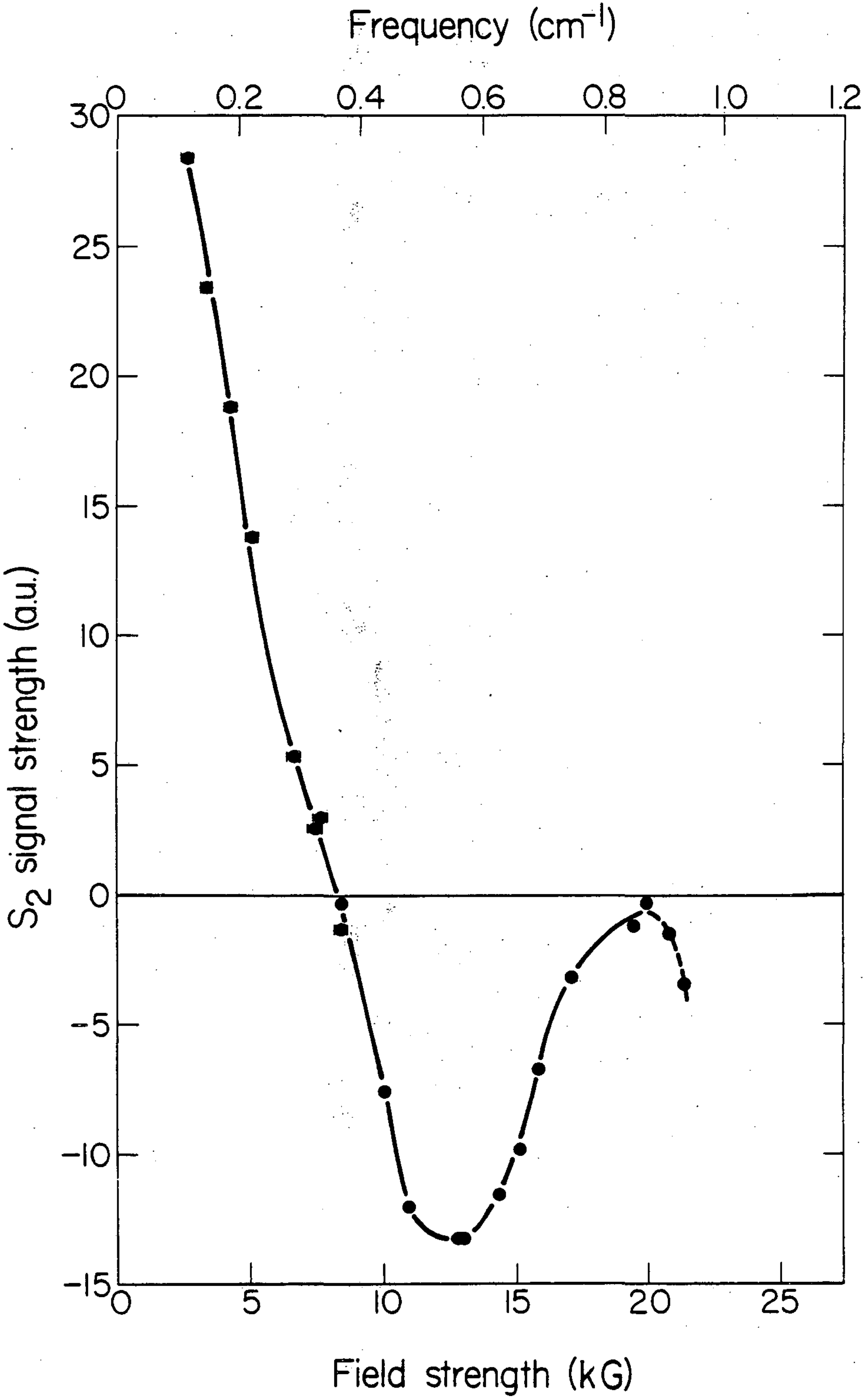

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Figure 2

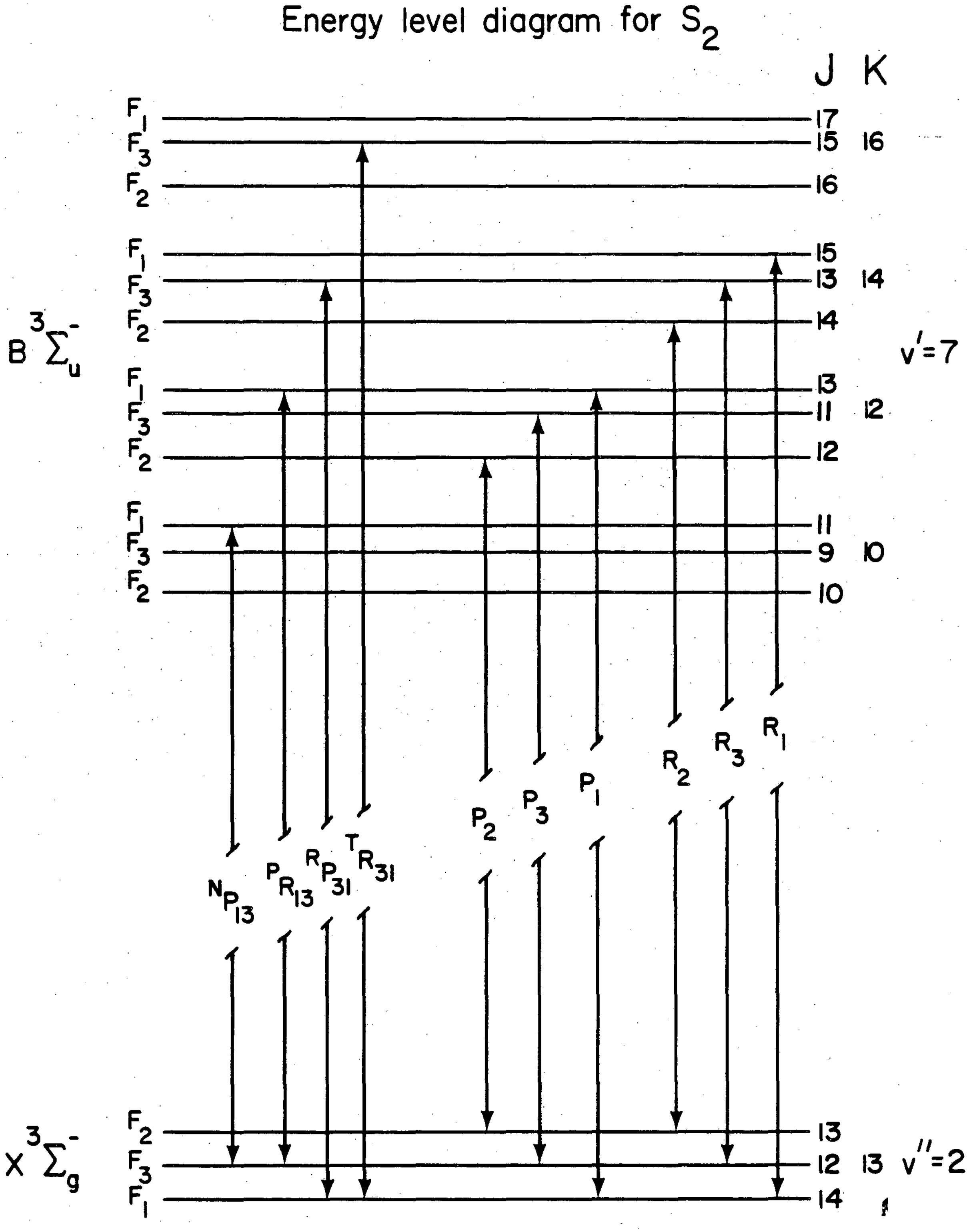

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Figure 3

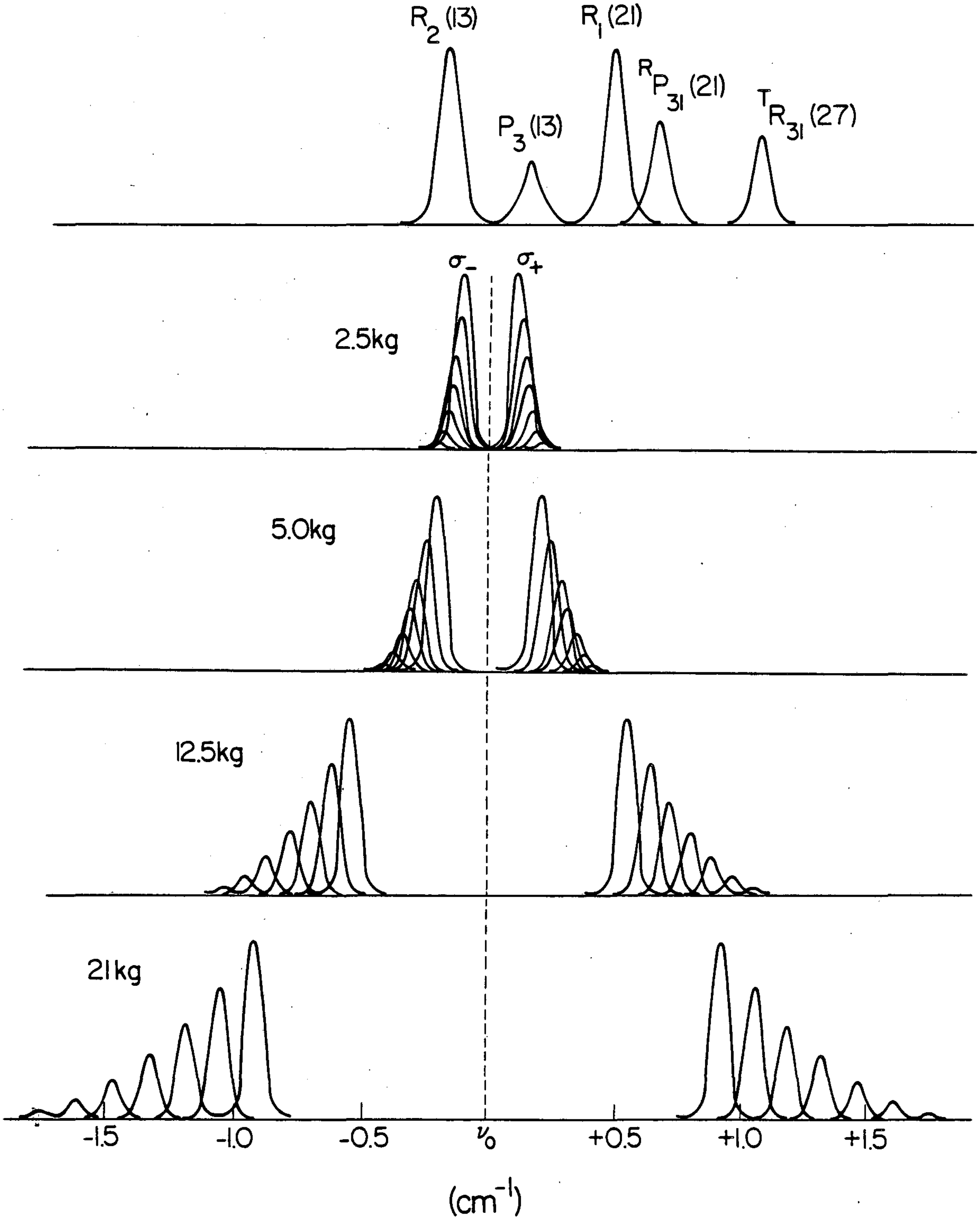

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Figure 4

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