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Determination of Alloy Content From Plume Spectral Measurements

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DETERMINATION OF ALLOY CONTENT FROM PLUME SPECTRAL MEASUREMENTS

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Abstract

The mathematical derivation for a method to determine the identities and amounts of alloys present in a flame where numerous alloys may be present is described. This method is applicable if the total number of elemental species from all alloys that may be in the flame is greater than or equal to the total number of alloys. Arranging the atomic spectral line emission equations for the elemental species as a series of simultaneous equations enables solution for identity and amount of the alloy present in a flame. This technique is intended for identification and quantification of alloy content in the plume of a rocket engine. Spectroscopic measurements reveal the atomic species entrained in the plume. Identification of eroding alloys may lead to the identification of the eroding component.

Nomenclature

A	transition probability
Av	Avogadro's number
E	energy
g	statistical weight
h	Planck's constant
k	Boltzmann's constant
mw	molecular weight
Φ	radiant power
T	temperature
ν	frequency

w weight percentage

Z(t) partition function

Background

When a single alloy is present in a flame, the identity of its elemental constituents, and therefore the alloy itself, can readily be determined using spectroscopic techniques. If multiple alloys are present in the flame, the identification process is again relatively simple if the alloys are composed of different elements. However, many alloys contain some of the same elemental species, their differences being various weight fractions of the elements. If numerous alloys are present in a flame, and these alloys contain some of the same elemental constituents, the spectra of the individual elements are summed, and a direct determination of identity and amount of the alloy cannot be made solely with spectroscopic techniques.

The emission spectrum of an alloy is the sum of the line emissions from its elemental constituents. Identifying key spectral lines from the various atomic species enables identification of the elements. Since the radiance of a spectral line is related to the number of atoms in the emitting system, atomic concentration can also be determined. If numerous alloys contain some of the same elements, the atom population for the various elements may be due to contributions of the element from numerous alloys. The resulting spectral radiance that is measured from an atomic species

may be due to contributions of the element from numerous alloys.

Emission spectroscopy of a rocket engine exhaust plume is being investigated to determine if engine degradation can be monitored from measurements of material erosion.¹⁻⁴ Knowledge of material erosion from internal components may provide insight into the engine's current health and remaining life. In the space shuttle main engines (SSME), it has been determined that material does in fact erode from internal engine components. Eroded particles are carried along with the hydrogen and oxygen propellants into the combustion chamber, where they are burned (dissociated to atomic species), thermally excited, and emit their characteristic spectrum. Figure 1 displays the propellant flow path within the SSME. Note that the propellants travel throughout the entire engine, with the ultimate destination being the combustion chamber. Figure 2 displays a typical SSME emission spectrum. Atomic lines can readily be identified in this figure. Alloys from which the SSME is fabricated are listed in Table 1. This table lists the alloys, their elemental constituents, and the weight fraction of these elemental constituents.⁵ As can be seen from this table, there are numerous alloys in the engine, many of which contain some of the same elements. Table 2 is a list of the SSME components in the propellant flow path and the alloy from which they are fabricated. Note that the propellants and impurities in the propellants are not included in the above tables.

This paper presents a technique that enables determination of the identities and amounts of alloys that are present in a flame. The mathematical derivation, and errors associated with this technique are discussed. Note that the primary purpose of this paper is to document the mathematics describing this technique, with minimal consideration given to implementation, calibration, and operation of this capability.

From knowledge of which alloys may be present in the flame, their elemental

constituents, weight fraction of their elemental constituents, spectral characteristics of these elements, and the temperature at the location of the emitting system, a series of simultaneous equations may be developed and solved. The solution to these equations yields the amount and identity of the alloy in the flame. Current state of the art in rocket engine diagnostics using plume spectral measurements enables identification and quantification of the atomic species. Application of the technique presented in this paper to a rocket engine enables identification and quantification of eroding alloys, which provides information that can lead to the identification of the eroding engine component. Note that this technique is not specific to rocket engine diagnostics; it may be used in other applications requiring alloy identification and quantification.

Theory

Radiance from atomic line emissions is a function of several variables. In this paper, the general expression for line radiance as a function of atomic population densities shall be considered. It is assumed that the collection of atoms in the emitting system are in thermal equilibrium, and that there is a low population density of the emitting atoms. It is also assumed that the radiance of the line is sufficient to enable spectroscopic measurements. The radiant power of an atomic line is given by the equation:⁶

$$\Phi = \frac{A_{ji} h \nu_{ji} g_j n e^{-E_j/kT}}{Z(t)} \quad (1)$$

where j_i represents one of the allowed transitions for an atomic species from an upper energy state j to a lower energy state i , and n is the total number of free atoms in the emitting system. Selection of a particular transition defines the frequency ν and therefore the wavelength of the emitted photons.

Numerical values for the transition probability, statistical weight and upper energy state for the selected transition can be found in reference tables.⁷ The partition function can either be found in reference tables⁸ or calculated from the following equation:

$$Z(t) = \sum_{j=0}^{\infty} g_j e^{-E_j/kT} \quad (2)$$

The solution to Eq. (1) yields the radiation at a given wavelength from a collection of atoms at a particular temperature. Utilizing emission spectroscopy techniques, this radiance can be measured. Therefore, at a known temperature for an allowed transition, Eq. (1) can be solved for the total number of free atoms in the emitting system. The number of atoms for the various elemental species can be used to determine the amount of the alloy that is present in the flame. To simplify the writing of Eq. (1), the following definition will be made:

$$R = \frac{A_{ji} h \nu_{ji} g_j e^{-E_j/kT}}{Z(t)} \quad (3)$$

where R represents the radiant power from a single atom. Substituting Eq. (3) into Eq. (1) yields:

$$\Phi = Rn \quad (4)$$

Consider an unknown amount x_a of alloy a . The alloy consists of ℓ elemental constituents, with the weight fractions of these elements equal to w_1, w_2, \dots, w_ℓ . It is assumed that the exact composition and weight fractions are known. The total weight w of alloy a can be expressed as:

$$w_a = x_a (w_1 + w_2 + \dots + w_\ell) \quad (5)$$

This equation can be expanded to calculate the total number of atoms in the alloy based on the total number of atoms of each elemental species by dividing the weight of an element by its

molecular weight and multiplying by Avogadro's number. Therefore, Eq. (5) can be rewritten as:

$$N_a = x_a A_v \left(\frac{w_1}{mw_1} + \frac{w_2}{mw_2} + \dots + \frac{w_\ell}{mw_\ell} \right) \quad (6)$$

where N_a is the total number of atoms in the amount x of alloy a . Writing similar equations for m different alloys yields:

$$N_1 = x_1 A_v \left(\frac{w_{11}}{mw_1} + \frac{w_{12}}{mw_2} + \dots + \frac{w_{1p}}{mw_p} \right) \quad (7-1)$$

$$N_2 = x_2 A_v \left(\frac{w_{21}}{mw_1} + \frac{w_{22}}{mw_2} + \dots + \frac{w_{2p}}{mw_p} \right) \quad (7-2)$$

⋮

$$N_m = x_m A_v \left(\frac{w_{m1}}{mw_1} + \frac{w_{m2}}{mw_2} + \dots + \frac{w_{mp}}{mw_p} \right) \quad (7-m)$$

where N_1 through N_m are the number of atoms in alloys 1 through m ; x_1 through x_m are the amounts of alloys 1 through m ; mw_1 through mw_p are the molecular weights of elemental species 1 through p , and w_{11} through w_{mp} are the weight percentages of the elemental species in the alloys. Note that the number of elemental species in Eqs. (7-1) to (7-m) has changed from ℓ to p . While ℓ represents the total number of elemental species found in a single alloy, p represents the total number of different elemental species that can be found in all alloys 1 through m . With this type of notation, the weight percentage of an element in the alloy is set equal to zero if the particular element is not present in an alloy.

By summing vertically down the columns of the above equations, the total

number of atoms of each elemental species can be calculated:

$$n_1 = \frac{Av}{mw_1} (x_1 w_{11} + x_2 w_{21} + \dots + x_m w_{m1}) \quad (8-1)$$

$$n_2 = \frac{Av}{mw_2} (x_1 w_{12} + x_2 w_{22} + \dots + x_m w_{m2}) \quad (8-2)$$

$$\vdots$$

$$n_p = \frac{Av}{mw_p} (x_1 w_{1p} + x_2 w_{2p} + \dots + x_m w_{mp}) \quad (8-p)$$

where n_1 through n_p are the total number of atoms of the each elemental species contributed from alloys 1 through m . Substituting these values into Eq. (4) and calculating a value for R from Eq. (3) enables radiance calculations of the p elemental species as a function of the amount x of the alloy. With this substitution, the following series of equations can be written:

$$\Phi_1 = \frac{R_1 Av}{mw_1} (x_1 w_{11} + x_2 w_{21} + \dots + x_m w_{m1}) \quad (9-1)$$

$$\Phi_2 = \frac{R_2 Av}{mw_2} (x_1 w_{12} + x_2 w_{22} + \dots + x_m w_{m2}) \quad (9-2)$$

$$\vdots$$

$$\Phi_p = \frac{R_p Av}{mw_p} (x_1 w_{1p} + x_2 w_{2p} + \dots + x_m w_{mp}) \quad (9-p)$$

At this point, consideration must be given to the known and unknown variables in Eqs. (9-1) to (9-p). The radiant power Φ is measured spectroscopically from the flame, and therefore is known. It is assumed that the temperature of the emitting system is known, resulting in known values for R_1 through R_p as defined by Eq. (3). The weight percent-

ages of the elemental species in the various alloys are obtained from metallurgical handbooks, while the elemental molecular weights and Avogadro's number are known. The unknowns are the amounts of the alloys x_1 through x_m . Therefore, examination of Eqs. (9-1) to (9-p) indicates that there are p equations with m unknowns. If p is greater than or equal to m , a sufficient number of equations are available to solve for all unknowns. If m is greater than p , the equations cannot be solved.

Mathematical Implementation

Assuming that there are a sufficient number of equations to solve for the unknowns, (the total number of different elemental species is equal to or greater than the number of alloys) the following matrix expression of Eqs. (9-1) to (9-p) can be written:

$$Av \begin{bmatrix} \frac{R_1 w_{11}}{mw_1} & \frac{R_1 w_{21}}{mw_1} & \dots & \frac{R_1 w_{m1}}{mw_1} \\ \frac{R_2 w_{12}}{mw_2} & \frac{R_2 w_{22}}{mw_2} & \dots & \frac{R_2 w_{m2}}{mw_2} \\ \vdots & \vdots & \ddots & \vdots \\ \frac{R_p w_{1p}}{mw_p} & \frac{R_p w_{2p}}{mw_p} & \dots & \frac{R_p w_{mp}}{mw_p} \end{bmatrix} \begin{bmatrix} x_1 \\ x_2 \\ \vdots \\ x_m \end{bmatrix} = \begin{bmatrix} \Phi_1 \\ \Phi_2 \\ \vdots \\ \Phi_p \end{bmatrix} \quad (10)$$

The above matrices can be expressed as $[R][X] = [\Phi]$, where the R matrix contains the $R_{1+p} w_{1+p} Av / mw_{1+p}$ terms, the X matrix contains the x_1 through x_m terms, and the Φ matrix contains the Φ_1 through Φ_p terms. Values for the R matrix are derived by simple calculations, values for the Φ matrix are measured spectroscopically from the flame, and the X matrix contains the unknown mass and by definition the identity of a particular alloy. If the total number of elemental species is greater than the number of alloys, $(p-m)$ equations can be eliminated. After

elimination of the excess equations, the R matrix is square and can be inverted. The solution to the X matrix is $[X] = [R]^{-1}[\Phi]$. The matrix inversion and subsequent matrix multiplication can be performed on a computer. Given that the $[R]$ matrix can be inverted, a solution will be produced. The accuracy of the solution is limited by the accuracy of the values in the $[R]$ and $[\Phi]$ matrices.

Examination of Table 1 reveals that there are 17 alloys consisting of 20 elemental species in the SSME. Therefore, three elemental species do not need to be spectroscopically monitored for alloy identification and quantification. The selection of which elements to eliminate is based on the relative abundance and emission intensity of the species. Since zirconium, lanthanum, and nitrogen appear in trace quantities in a very limited number of alloys and are relatively weak emitters,⁹ they should be the species that are eliminated.

Discussion

Although the mathematics described above are relatively straightforward, implementation of this technique requires additional considerations. These considerations include the errors that are inherently associated with the technique, and the sensitivity, dynamic range, and calibration of the instrumentation used to measure the emission spectrum. Following is a discussion of the errors and instrumentation issues. While there may be numerous sources of error, the errors do not appear to be insurmountable individually or collectively.

The derivation of this technique is based on spectroscopic, metallurgical, and mathematical considerations. Errors associated with the implementation of this technique to the analysis of a rocket engine exhaust plume are the result of the limitations and assumptions in spectroscopic theory and spectroscopic measurement techniques, uncertainties in

alloy content, and the assumption that all constituents of the alloy wear at a constant rate. Spectroscopically, the assumptions that may lead to error include: (1) the temperature is known and uniform throughout the emitting system, (2) the radiance can be accurately measured regardless of the number of atoms and radiance from the emitting system, and (3) that exact values for the transition probabilities are known. Metallurgically, the assumption that the exact composition and weight percentages of the elements in the alloys are known may lead to error. The assumptions that the various elemental species in the alloy erode evenly and at the same rate is also a potential source of error. Following is a discussion of the various errors that may be caused by the assumptions.

Temperature dependence of the radiance from atomic emissions can be seen in Eq. (1). For the SSME, temperatures in the combustion chamber and Mach disk regions are well established from analytical calculations.^{10,11} The assumption that the emitting atoms are in thermal equilibrium is valid since the temperatures in the chamber or the Mach disk are reasonably uniform. Temperature variations occur near the walls of the chamber, and near the edges of the Mach structure. If spectral emissions from the center of the combustion chamber or center of the Mach structure are measured and used in the Eq. (9), thermal equilibrium and uniform temperature is assured. Note that using a limited field of view introduces the assumption that metallic species entrained in the plume are homogeneously mixed within it; and that observation of a known percentage of the plume accurately reflects the overall composition of the entire plume. Measurements from a limited field of view requires that a scaling factor be incorporated into Eq. (9) to account for the hot gases and therefore spectral emissions that are not being observed.

To gain insight into the temperature effects with respect to the SSME, the

Boltzmann distribution (temperature dependent terms in Eq. (1)) were evaluated at 3000 and 3149 K for chromium and titanium. Numerical values for the energy levels, statistical weights, and partition functions were obtained from Refs. 7 and 8 respectively. It was calculated that for the 5 percent temperature change, the radiant power increased 46.0 percent for chromium, and 83.6 percent for titanium. As seen from Eq. (1), the temperature term appears in an exponential, and the significance of this term depends on the slope of the exponential at the temperature regime of the emitting system. For the SSME temperature regime, temperature is critical for accurate measurements, and a 5 percent temperature deviation would yield unusable results for this technique.

It is assumed that an adequate number of free atoms of the species being spectrally monitored are available within the observed region to enable measurement. A single or very few emitting atoms of a particular species will not be seen in an environment consisting of many free atoms from numerous species due to either the low radiance from the few atoms, poor sensitivity of the detector, or low transition probability of the transition that is being monitored. If an extremely large number of atoms of a particular species are present in the flame, the flame is optically thick rather than optically thin, requiring that the self absorption terms be included in Eq. (1).⁶ Therefore, the population of the metallic species must be sufficient to provide radiance in excess of that of the background chemiluminescence from the combustion process, yet not great enough to cause self absorption. In the SSME, the population of the eroded metallic species from the alloys is much less than the population of the oxygen and hydrogen propellants (unless a major failure is occurring).

Error may result from the transition probabilities and their associated uncertainties. Radiance emitted from an excited atom is directly proportional to

the transition probability of the specific transition that is being monitored. Low transition probability could lead to error due to the low overall radiance from the atoms in the emitting system. The uncertainty associated with the various transition probabilities may also cause errors. Typically, values for the transition probabilities are listed with an associated uncertainty band. These uncertainties range from 1 percent up to 50 percent for the various transitions as seen in Ref. 7. Selection of a transition with a low uncertainty will minimize error; however this may be difficult, since not all species have been adequately studied to generate high accuracy transition probabilities.

Sensitivity, signal to noise ratio, dynamic range, and resolution of the spectrometer may limit the accuracy of this technique. The sensitivity and signal to noise ratio dictate the minimum radiance level that can be measured. The dynamic range limits the maximum radiance level that can be measured, assuming the spectrometer is optimized for low radiance levels. Spectrometer resolution must be adequate to separate closely spaced spectral lines. It was assumed that spectral emissions from all elemental species that may be in the plume can be measured. This assumption is correct if the detector is sensitive enough to measure potentially low radiance levels. From Table 1 it can be seen that some alloys contain trace amounts of certain elements. It may be extremely difficult to measure spectral emissions from these trace amounts unless a large quantity of the alloy is eroding, or the transition that is being monitored has a large transition probability. Past work⁴ has indicated that large amounts of some elements must be present in the plume for observation due to low transition probabilities. The worst case scenario is the low weight percentage species that has low transition probabilities. The lack of sensitivity may be overcome with higher sensitivity detectors, or by using atomic absorption rather than atomic emission spectroscopy.

Spectrometer calibration must also be considered since absolute, rather than relative spectral emissions are being measured. Calibration must take into account spectrometer throughput, efficiency of the detector at all observed wavelengths, and view angle of the plume. It may be possible to calibrate the spectrometer at a few spectral lines using calibrated lamps and use these measurements as the basis for calibration of the spectrometer.

The assumptions that the composition of the alloy is known exactly and that the elemental constituents of the alloy are homogeneously mixed may lead to error. Typically, the composition of an alloy is expressed as the nominal, rather than exact, weight percentages of its elemental constituents. The exact percentages of the elements are not known unless a thorough analysis of the alloy is made. Also, the alloys may contain trace impurities. If an element is a trace impurity in one alloy but a constituent in another alloy, errors may result. For critical applications such as the SSME, it is presumed that the exact composition of the alloys are known.

To enable a mathematical solution, the number of elemental species must be greater than or equal to the number of alloys that are found in the engine. Examination of Table 1 will verify that this is the case for the SSME. Note that an appropriate set of equations must be generated for the temperature of the emitting system.

Concluding Remarks

The mathematical derivation of a technique that enables determination of the identities and amounts of alloys present in a flame where numerous alloys may be present has been described. This technique is particularly useful when the same elemental species are found in many of the different alloys. The intended application of this technique is to enable identification and quantification

of eroding material from internal components of a rocket engine based on its plume spectral emissions. Identification of the eroding alloy may lead to identification of the eroding component, and quantification of erosion may lead to determination of the remaining life of a component.

Requirements for implementation of this technique include knowledge of which alloys may be present in the flame, their elemental constituents, weight fraction of the elemental constituents, spectral characteristics of these elements, temperature at the location of the emitting system, and measured radiance values from the atomic emissions in the emitting system. Arranging the atomic emission equations for all the elemental species in the alloys as a series of simultaneous equations enables solution for the identity and amount of the alloy in the flame. To enable a mathematical solution, the total number of elemental species from all alloys must be greater than or equal to the number of alloys that are found in the engine. An appropriate set of equations must be generated for the temperature of the emitting system, and that if the temperature changes, a new set of equations must be generated. Note that an "alloy" may consist of a single element.

The viability of this technique is limited by the inherent errors and assumptions that went into its derivation. Although there are numerous sources of errors, they do not appear to be insurmountable. Critical sources that may lead to the most significant error are the uncertainty and likely fluctuation of the temperature in the emitting region, and the uncertainty associated with the transition probabilities.

It is assumed that an adequate number of free atoms of the species being spectrally monitored are available within the observed region to enable measurement, and that spectral emissions from all elemental species that may be in the plume can be measured. The population of

the metallic species must be sufficient to provide radiance in excess of that of the background chemiluminescence from the combustion process, yet not great enough to cause self absorption. The sensitivity, signal to noise ratio, dynamic range, and resolution of the spectrometer that is measuring the spectral emissions must be adequate for the widths and peak intensities of the spectral lines being measured.

The composition of the alloys must be known, and the elemental constituents of the alloy must be homogeneously mixed. The assumption that the exact composition of the alloy is known may lead to error, since alloy composition is typically expressed as the nominal weight percentages of its elemental constituents. Also, the alloys may contain trace impurities. If an element is a trace impurity in one alloy but a constituent in another alloy, errors may result. The elemental constituents of the alloy must be homogeneously mixed, or the erosion rate of the elementals will not be constant.

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TABLE 1. - NOMINAL ELEMENTAL COMPOSITION OF SSME ALLOYS IN WEIGHT PERCENTAGES

Elements Material	Fe	Ni	Co	Mn	Cr	Mo	W	C	Hf	Ti	Cu	Al	Nb	Ta	Si	Ag	V	Zr	La	N
AISI 440A	79.5			1.0	17.0	0.8		0.7							1.0					
347 CRES	68.4	10.5		2.0	18.0			0.1							1.0					
Incoloy 903	41.0	38.0	15.0						1.4			1.0	~1.8	~1.8						
Armco 21-6-9	63.8	6.5		9.0	20.3															0.4
AISI 440C	80.2	0.2		0.5	16.9	0.5		1.0			0.1				0.6					
316L CRES	65.5	12.0		2.0	17.0	2.5									1.0					
Haynes 188	3.0	22.0	37.2	1.3	22.0		14.0	0.1							0.3				0.1	
Stellite	9.0	3.0	35.0		28.0		18.0	3.0						0.1			3.9			
MAR - M246+Hf		58.1	10.0		9.0	2.5	10.0	0.1	1.7	1.5		5.5		1.5				0.1		
Waspaloy X	1.0	56.7	13.5	0.5	19.5	4.2				3.0	0.1	1.2			0.2			0.1		
K-monel	1.0	65.5		0.8				0.1		0.6	29.0	3.0								
Rene 41		55.4	11.0		19.0	10.0		0.1		3.0		1.5								
Inconel 718	18.0	52.9		0.2	19.0	3.0				0.8		0.6	5.3		0.2					
Inconel 625	2.5	61.6	1.0	0.2	21.5	9.0				0.2		0.2	~1.8	~1.8	0.2					
304 CRES	68.7	9.2		2.0	19.0			0.1							1.0					
NARloy A											96.0					4.0				
A-286	55.0	26.0			15.0	1.3				2.0		0.4						0.3		

TABLE 2. - SSME COMPONENTS AND MATERIALS (PARTIAL LIST)

Material	Found in SSME part
A-286	Fuel and oxidizer preburner Main fuel, oxidizer and coolant valve
AISI 440A	Fuel, oxidizer and coolant valve body
AISI 440C	HPOTP and HPFTP bearings
Armco 21-6-9	Oxidizer pump discharge flex joint LPFP discharge flex joint Fuel bleed line flex joint
304 CRES	Main injector heat shield
316L CRES	Heat exchanger
347 CRES	Main injector faceplate
Haynes 188	LOX posts Heat exchanger Fuel bleed line flex joint
Incoloy 903	External gimbal ring flex joint Hot gas manifold liner Heat exchanger HPOTP primary turbine seal HPOTP secondary turbine seal HPOTP turbine interstage seal
Inconel 625	Temperature sensors Fuel preburner faceplate Oxidizer preburner faceplate Heat exchanger Hot gas manifold Heat exchanger

TABLE 2. - CONCLUDED.

Material	Found in SSME part
Inconel 718	Oxidizer pump discharge flex joint LPFP discharge flex joint Fuel bleed line flex joint HPOTP intermediate seal HPOTP boost pump labyrinths HPOTP pump impeller HPOTP pump stator Main combustion chamber shell External gimbal ring flex joint Oxidizer tank pressurization duct flex joint Hot gas manifold Heat exchanger
K-monel	HPOTP primary oxidizer seal
MAR-M246+Hf	HPOTP turbine blades HPFTP turbine blades HPOTP turbine tip seals
NARloy A	Preburner baffles
NARloy Z	Main combustion chamber
Rene 41	HPOTP turbine tip seal
Stellite	Valves
Waspaloy X	HPOTP and HPFTP turbine disks
NiCrAlY	HPOTP and HPFT turbine blades



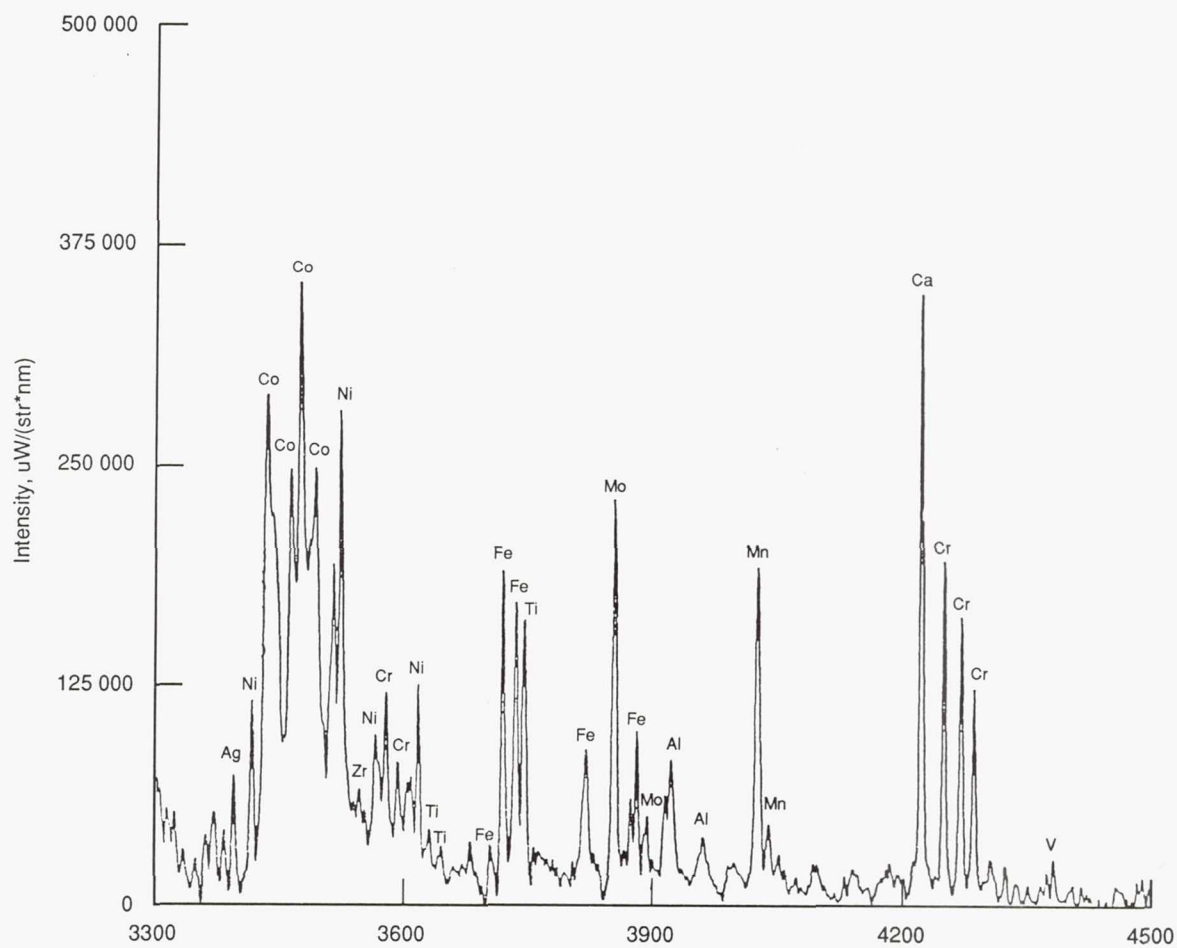


Figure 2.— Typical SSME emission spectrum.



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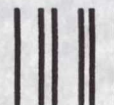
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