

AN ABSTRACT OF THE THESIS OF

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Title: Conformational Analysis: Ethylene Glycol and

Ethylenediamine. An Electron-Diffraction Investigation of the

Molecular Structures, Compositions, Anti-Gauche Energy

Differences and Entropy Differences.

Abstract Approved: **Redacted for Privacy**

Kenneth W. Hedberg

The temperature dependence of the rotameric compositions for ethylene glycol (EG) and ethylenediamine (EDA) have been investigated in the gas phase by electron diffraction. For ethylene glycol at 376 K and 733 K, the gauche conformer strongly dominates even at the higher temperature of the experiment: the amount of the anti form was too small to be determined with certainty. Rotational constants (three isotopic species), dipole-moment components (two isotopic species) and the diffraction data were used simultaneously in the structure analysis. For the ODOD species at the lower

temperature after corrections for the effect of vibrational averaging, the bond lengths (r_a), bond angles and torsion angles ($\angle_\alpha$, τ) and rms amplitudes (l) of vibration with uncertainties (2σ) were found to be $r(\text{C-H}) = 1.135(6)\text{\AA}$, $r(\text{C-O}) = 1.426(2)\text{\AA}$, $r(\text{C-C}) = 1.518(6)\text{\AA}$, $r(\text{O-H}) = 0.992(7)\text{\AA}$, $\angle\text{COH} = 101.1(16)^\circ$, $\angle\text{CCO} = 110.1(3)^\circ$, $\angle\text{CCH} = 111.2(15)^\circ$, $\tau(\text{OCCO}) = 58.4(10)^\circ$, $l(\text{C-O}) = 0.058(4)\text{\AA}$, $l(\text{C-C}) = 0.060(4)\text{\AA}$. The torsion angles $\tau(\text{CCOH})$ had values equal to $20.0(36)^\circ$ and $135.6(34)^\circ$. Values for the other isotopic species are similar.

The EDA molecule was investigated at 343 K, 463 K and 713 K. Three distinct conformers were identified: two of the more stable heavy atom gauche form and a less stable anti form. The amount of the gauche versus the anti form changes slightly as the temperature increases. Assuming that all conformers have the same values for their structural parameters except for the torsion angles, the average bond lengths (r_a), bond angles ($\angle_\alpha$) for some of the more important parameters from the lowest temperature were $r(\text{C-H}) = 1.123(11)\text{\AA}$, $r(\text{N-H}) = 1.024(8)\text{\AA}$, $r(\text{C-N}) = 1.471(4)\text{\AA}$, $r(\text{C-C}) = 1.528(12)\text{\AA}$, $\angle\text{CCN} = 111.4(5)^\circ$. The torsion angles, $\tau(\text{NCCN})$, $\tau(\text{CCNH})$, $\tau(\text{CCNH})$ in the gauche (A) had values equal to $55.3(8)^\circ$, $-74.7(74)^\circ$, $248.0(59)^\circ$, and for the other gauche form (B), these values were equal to $63.6(9)^\circ$, $-38.8(108)^\circ$, and $112.3(57)^\circ$, respectively.

The stability of the gauche form relative to the anti may be attributed in larger part to the internal hydrogen bonding and somewhat to the "gauche effect" in both molecules, rather stronger in EG than in EDA.

Conformational Analysis
Ethylene Glycol and Ethylenediamine:
An Electron Diffraction Investigation of the
Molecular Structures, Compositions, Anti-Gauche Energy
Differences and Entropy Differences

by

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To

my parents,

my teachers,

my students,

in gratitude for what they taught me.

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CONFORMATIONAL ANALYSIS. ETHYLENE GLYCOL AND
ETHYLENEDIAMINE: AN ELECTRON-DIFFRACTION INVESTIGATION OF
THE MOLECULAR STRUCTURES, COMPOSITIONS, ANTI-GAUCHE ENERGY
DIFFERENCES AND ENTROPY DIFFERENCES

INTRODUCTION

The molecular structures of 1,2-disubstituted ethanes with electronegative substituents have been the subject of many experimental and theoretical investigations. Determination of the structures by gas-phase electron diffraction in many cases is a very challenging task, due to internal rotation not only around the carbon-carbon single bonds, but also around the bonds from carbon to the substituent groups. As a result of these rotations, there is the possibility for several conformers to be present simultaneously at equilibrium. The relative amounts of these rotamers are determined by small, subtle differences in electronic structure, by intramolecular steric effects, by the experimental temperature, and by hydrogen bonding.

As far as the influence of electronic structure is concerned, both theoretical and experimental studies have shown that in the presence of adjacent lone pairs and/or highly polar bonds, these molecules have a tendency to adopt that structure which has the maximum number of gauche interactions between the adjacent electron pairs and/or polar bonds. This is called the "gauche effect"¹. An example of the lone pairs effect is seen in the molecule

dimethyl diselenide , $\text{CH}_3\text{-Se-Se-CH}_3$ ², where the gauche (dihedral angle 82°) form shows greater stability than the anti. An example of the polar bond effect is seen in the structure of 1,2-difluoroethane³, where the gauche form is more stable and dominates the rotameric composition, whereas in other 1,2-dihaloethanes the anti forms are more stable⁴⁻⁶.

The second factor which is responsible for the stability of gauche conformers in 1,2-disubstituted ethanes is the intramolecular steric effect, where large substituent groups on carbon atoms tend to make the anti form more stable by minimizing the repulsion between the substituent groups. The intramolecular steric effect appears to be the primary effect in yielding the larger proportion of the anti form in 1,2-dichloro-, -dibromo-, and -diiodoethane.

The temperature effect is a simple consequence of the Boltzmann distribution law, where at high temperature more molecules attain the less stable form, therefore changing the rotameric composition.

The last factor which presumably influences the conformational stability of 1,2-disubstituted ethanes is internal hydrogen bonding. To understand the effect of internal hydrogen bonding, we have to extract the hydrogen bond energy. In order to obtain the hydrogen bond energy, we need the equilibrium composition (mole fraction) of the conformers at several temperatures. For the reaction $\text{A} \rightleftharpoons \text{G}$, the mole fractions are related to the equilibrium constant by

$$\Delta G^\circ = -RT \ln K$$

where K , the equilibrium constant, is the ratio of the mole fraction of one form, say gauche (N_g/N_{tot}) to the mole fraction of the anti form (N_a/N_{tot}) at a given temperature. These quantities can be measured experimentally. ΔG° is related to the enthalpy and entropy changes by the equation

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ.$$

At constant pressure and volume ΔH° is equal to ΔE° , where ΔE° is the energy difference between the gauche and the anti forms ($\Delta E^\circ = E_g^\circ - E_a^\circ$). Likewise, ΔS° is the corresponding entropy difference ($\Delta S^\circ = S_g^\circ - S_a^\circ$) between the two forms. Hence, from the above equations we have:

$$K = \exp(-\Delta G^\circ/RT)$$

or

$$K = 2 \exp(-\Delta E^\circ/RT) \cdot \exp(\Delta S^\circ/R)$$

Here we have introduced a weighting factor (2) which takes into account the fact that we have two equivalent gauche forms, but only one anti form. Rearranging the last equation and rewriting in logarithmic form yields

$$R \ln K = -\Delta E^\circ/T + \Delta S^\circ + R \ln 2.$$

By plotting $R \ln K$ against $1/T$, one can determine the slope and the intercept which give ΔE° and ΔS° , respectively. The change in the internal energy (ΔE°) represents a significant, and very interesting, quantity in conformational analysis. Since ΔE° reflects all the intramolecular interactions, in particular hydrogen bonding for the conformers, our interest was to investigate the connection between this parameter (ΔE°) and its implication for bonding. The basic questions are: What role does the internal hydrogen bonding play in the stabilization of the gauche conformers? To what extent is this stabilization due to the "gauche effect"? Here, the polarity of C-X bonds, where X is an electronegative group, together with the polarity of X-H bond responsible for the internal hydrogen bonding, favors the gauche conformer. In other words, when internal hydrogen bonding can occur so, too, can the gauche effect.

This thesis describes the conformational analysis and determination of the molecular structures of two isoelectronic molecules, ethylene glycol and ethylenediamine. The immediate purpose of the experiments was to determine the structures of the rotamers and the variation (if any) of the rotameric compositions as a function of temperature. From the equilibrium compositions, it was hoped to obtain information about the thermodynamic properties, such as the energy and entropy differences of the conformers. Since the molecules of both compounds have the potential of forming intramolecular hydrogen bonds, the study was also concerned with the importance of hydrogen bonding as a determining factor for the

molecular structure and the conformational equilibria.

The earliest electron diffraction investigation on ethylene glycol at sample temperature showed the presence of mainly the gauche conformer.⁷ Initially, we undertook the study at two different temperatures, 376 K and 733 K, where a larger fraction of the molecules is expected to be in the anti form. As it turned out, the gauche form was highly predominant (greater than 95% even at the higher temperature) which prevented us from determining the energy difference ($E_g^\circ - E_a^\circ$) and the entropy difference ($S_g^\circ - S_a^\circ$). This difficulty arises because of the large uncertainty associated with the rather small percentage of N_a .

The molecular structure of ethylenediamine has also been studied by several workers.⁸⁻¹⁰ The latest electron diffraction work on this molecule was done at temperature range of 50-120°C, where the presence of only one gauche conformer was indicated¹¹. On the other hand, microwave work shows the existence of two gauche conformers,¹² one more stable by 0.3 ± 0.2 Kcal mol⁻¹ than the other. A theoretical study¹³ on ten conformations of ethylenediamine has also confirmed the microwave results for the presence of the two gauche forms, apparently stabilized by internal hydrogen bonding. The anti forms were energetically unfavorable. Our study at three different temperatures, 343 K, 463 K, and 713 K revealed a fairly constant fraction of the anti form (17 % - 23 %). The value of ΔE° was calculated to be about -0.338 kcal/mol, indicating the greater stability of the gauche conformers. Some details concerning the molecular structure of each molecule will follow.

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CONFORMATIONAL ANALYSIS. ETHYLENE GLYCOL.

MOLECULAR STRUCTURE, HYDROGEN BONDING, AND COMPOSITION.

Introduction

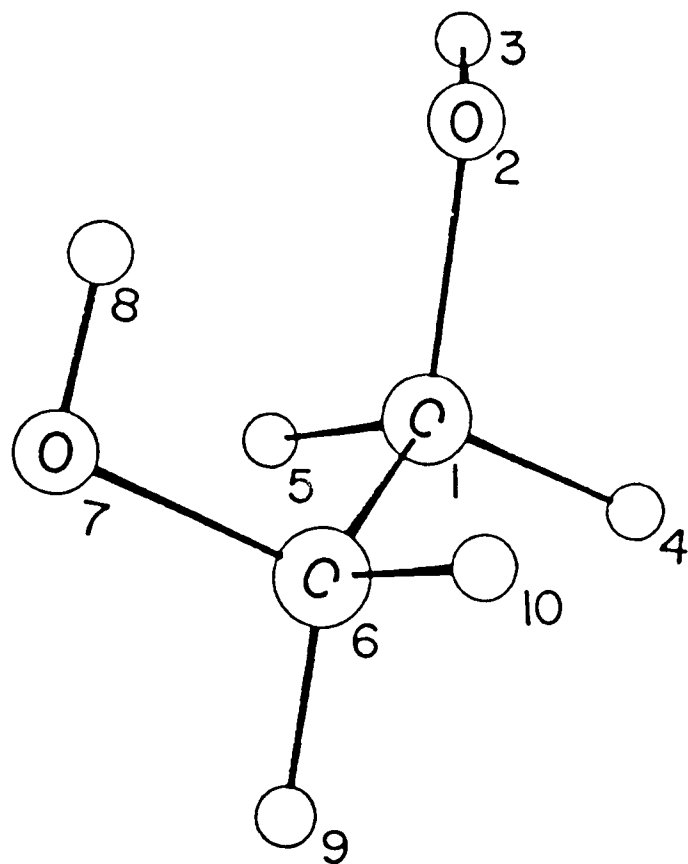
In the gaseous state, 1,2-disubstituted ethanes exist in a mixture of gauche and anti forms which is the result of their internal rotation around C-C bonds. The gauche forms are the more stable when pairs of electronegative substituents, such as any combination of F, OH, and NH_2 , are present. With pairs of less electronegative substituents such as Cl and SH^1 , the anti form is the more stable, and when substituents of both types are present the stabilities of the gauche and anti forms tend not to differ much. An important factor in the stabilization of gauche forms relative to the anti seems to be the formation of internal hydrogen bonds. These bonds have been identified from variation in the intensities of vibrational bands observed as a function of temperature. In some molecules where the potential for intramolecular hydrogen bonding exists, such as 2-fluoroethanol², 2-aminoethanol³, 2-mercaptoethanol⁴, 2-chloroethanol⁵, and ethylenediamine^{6,7}, the gauche conformer predominates (greater than 80%) at room temperature. On the other hand, in 1,2-difluoroethane,⁸ where there is no possibility for intramolecular hydrogen bonding, the gauche form is still the predominant conformer (about 95%). This unusual stability has been attributed to the "gauche effect"⁹. In 1,2-dibromoethane¹⁰, where there is neither the possibility of internal hydrogen bonding nor the gauche effect, the

anti form predominates at room temperature (about 95%).

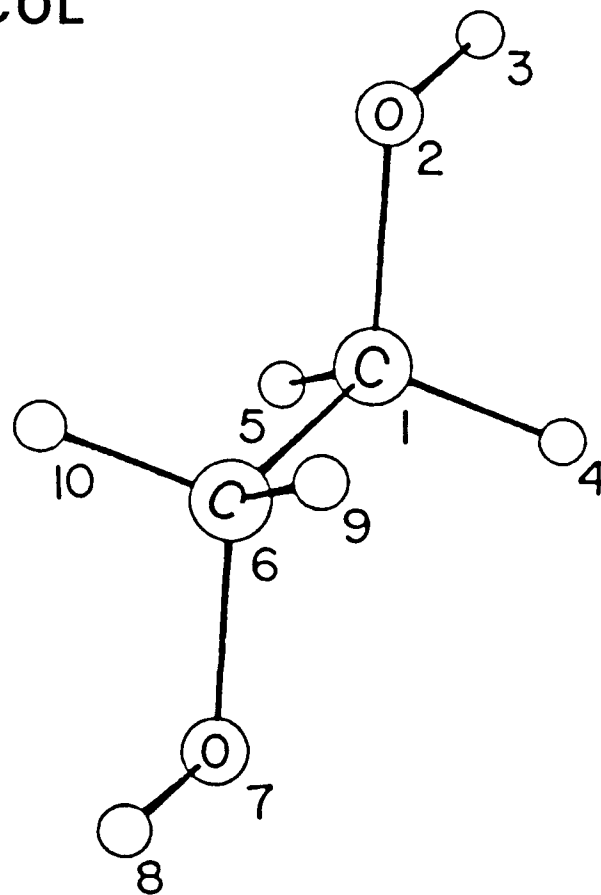
The molecule ethylene glycol ($(\text{CH}_2\text{OH})_2$, EG), diagrammed in Figure 1, forms a very strong intramolecular $\text{O}\cdots\text{H}-\text{O}$ hydrogen bond due to the high electronegativity of the OH groups. Hence the gauche form is predicted to be the more stable form at room temperature, and was shown to be so by an early electron-diffraction investigation by Bastiansen.¹¹ Infrared studies¹² on the rotational isomerism of EG were interpreted in terms of the presence of the gauche isomer only. In another study, Marstokk, et al.¹³ were unable to assign the microwave spectrum of the most abundant isotopic species of EG (OHOH), while Walder, et al.¹⁴ were able to assign the glycol- $\text{O}-\text{d}_2$ (ODOD) species. Caminati et al.¹⁵ measured the microwave spectrum of the two possible O-monodeuterated isotopic species (OHOD, ODOH). Their study revealed that one oxygen is gauche with respect to the other, and one H (or D) participates in a hydrogen bond while the other D (or H) is anti with respect to the C-C bond. An extensive ab initio study by Van Alsenoy¹⁵ also predicted the presence of two gauche forms tGg' and gGg', where G indicates that the oxygen atoms of the grouping OCCO are in the gauche conformation. The lower case letters t, g, and g' refer to the CCOH torsions: t is trans (180°), g is gauche (60°) and g' is gauche (-60°).

Our aim in this study was to investigate the possibility that high temperatures would generate a measurable amount of the anti conformer, and if so, to deduce the energy and entropy differences of anti and gauche forms. Our results indicated only marginally

ETHYLENE GLYCOL



GAUCHE



ANTI

Figure 1. Atom numbering for anti- and gauche-ethylene glycol.

detectable amounts of the anti form of EG are present even at the highest temperature of our experiment (733 K). The fraction of the anti form is estimated to be less than 5% at this temperature. Consequently, neither the standard energy difference nor the entropy difference could be determined.

Experimental Section

The sample of EG was Baker Analyzed Reagent grade which was further purified by fractional distillation under reduced pressure through a column packed with glass helices. A middle fraction was chosen for the experiments. Diffraction photographs were made in the Oregon State apparatus at nozzle-tip temperatures of 733 K, and 376 K. Conditions of the diffraction experiments were as follows: sector shape, r^3 ; plates, Kodak projector slide medium contrast 8x10 in.; ambient apparatus pressure during exposure, 1.8×10^{-6} - 1.8×10^{-5} torr ; exposure time, 60 - 180 sec; beam currents, 0.33 - 0.44 μ A; nozzle-to-plate distance, 74.614 - 74.716 cm (" long camera "), 29.871 - 29.970 cm (" middle camera "), electron wavelengths 0.05814 - 0.05812 Å; accurately determined by voltage measurements calibrated against gaseous CO_2 with $r_a(\text{C}=0) = 1.1646$ Å and $r_a(0 \cdots 0) = 2.3244$ Å. Four long camera plates and three middle camera plates were chosen at both temperatures for analysis. Data over the range $2.00 \leq s \leq 13.75 \text{ Å}^{-1}$ were obtained from the low-temperature and $2.00 \leq s \leq 14.00 \text{ Å}^{-1}$ from the high-temperature long camera plates. For middle distance plates

data over the range $8.00 \leq s \leq 33.75 \text{ \AA}^{-1}$ for the low temperature and $7.00 \leq s \leq 33.00 \text{ \AA}^{-1}$ from high temperature were taken. The data interval was $\Delta s = 0.25 \text{ \AA}^{-1}$. Procedures used for obtaining the total scattered intensities ($s^4 I_t$) and the molecular intensities ($s I_m(s)$) have been described.^{17,18} Tables of experimental data are given in Appendix I. Figure 2 shows the curves of the total scattered intensities and the final backgrounds for the experiment at 376 K. Figure 3 shows the experimental radial distribution of EG at two temperatures. These curves were calculated by Fourier transformation of the function

$$I'_m(s) = Z_c Z_o A_c^{-1} A_o^{-1} s I_m(s) \exp(-0.0025s^2)$$

where $A = s^2 |F|$ and F is the complex electron-scattering amplitude. Data in the unobserved regions $s < 2.00 \text{ \AA}^{-1}$ were taken from the theoretical intensity curve corresponding to the final model. Amplitudes and phases for all calculations were obtained from tables.^{19,20}

Structure Refinements

Detailed answers to the question of torsional problem in EG are not possible by the electron-diffraction method alone. This is particularly so when a light atom such as hydrogen is involved in torsional motions. The problem becomes even worse when the experiment is carried out at higher temperatures, where the vibrational

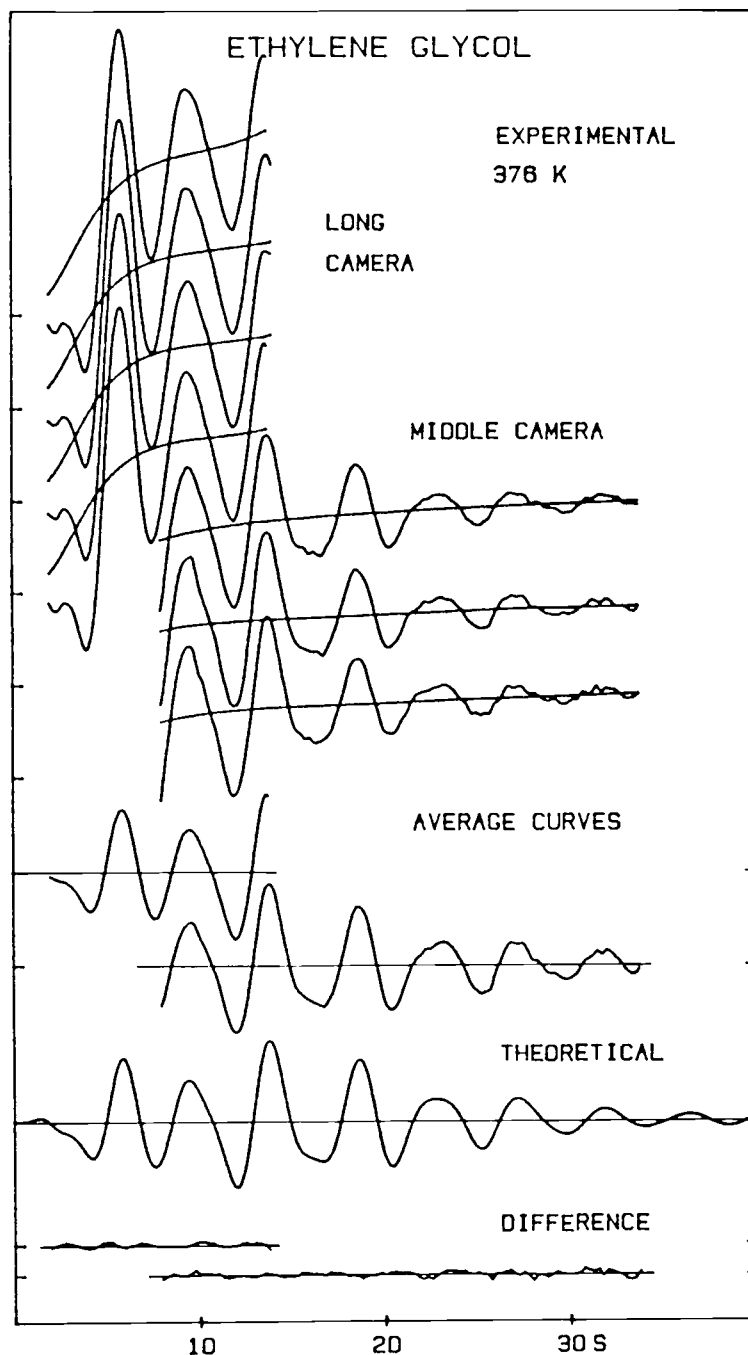


Figure 2. Intensity curves. The experimental curves are s^4I_t shown superimposed on the final backgrounds. The theoretical curve is sI_m for the final model. The difference curves are the experimental minus the theoretical.

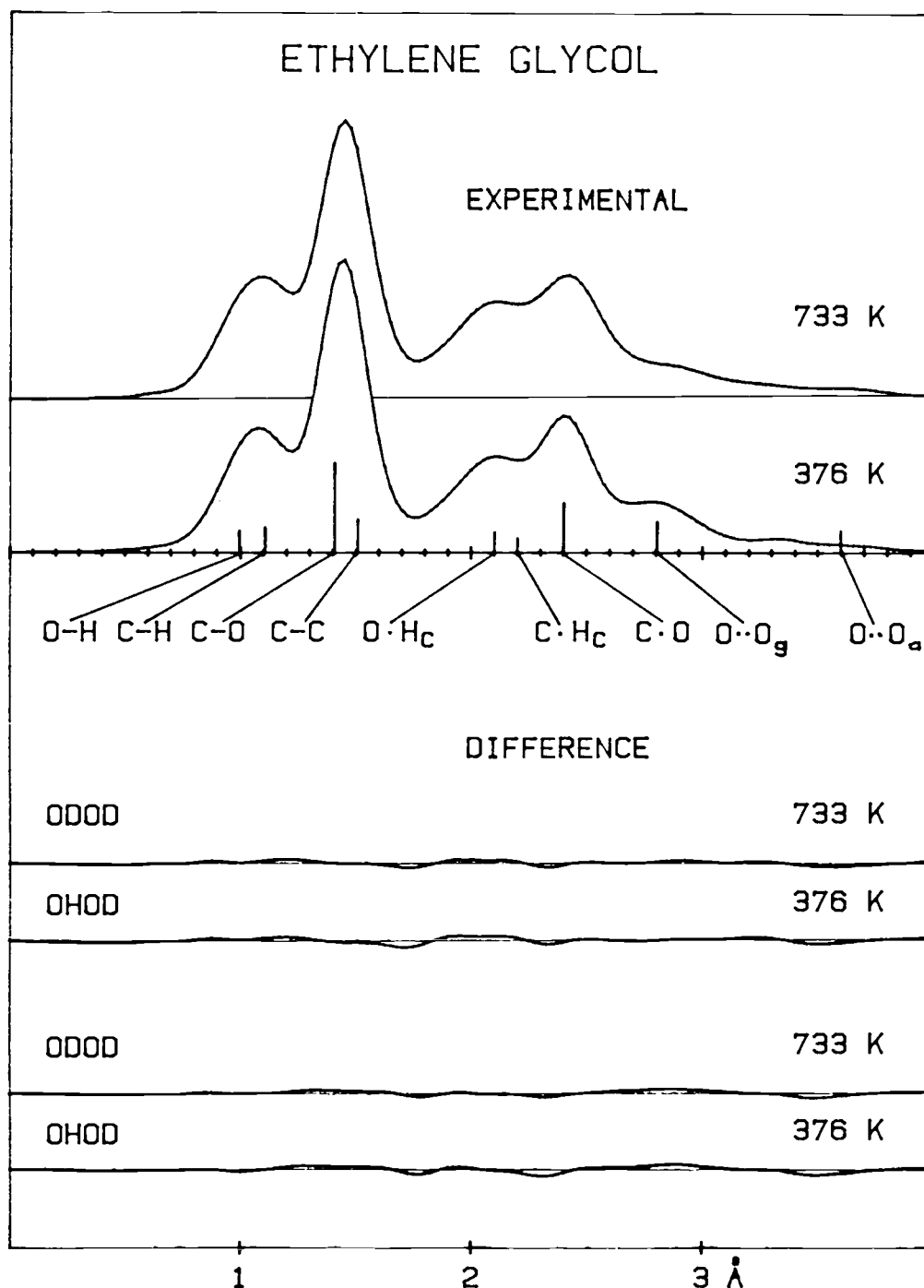


Figure 3. Radial distribution curves. The experimental are calculated from composites of molecular intensities from the two camera distances.

amplitudes become appreciably larger. This imposes an even greater uncertainty upon the average positions of all atoms, most noticeably hydrogen. Consequently, it has become common practice in our structure refinements to include rotational constants as observables when available together with the diffraction data. In doing so, the former serve as important constraints and, because of their precise values, their inclusion generally leads to more reliable results than do electron-diffraction data alone. Another useful, but not so common, type of information is the dipole-moment components. While the rotational constants are very sensitive to the heavy atom torsion angle (OCCO) in EG, dipole-moment components are extremely sensitive to the torsional angles of the two -OH groups. The value of the dipole-moment components in electron-diffraction analysis is still somewhat uncertain, mainly due to the lack of knowledge about the charge distributions. Nevertheless, these components have proven to be helpful in the determination of the orientation of peripheral groups in other molecules, and we decided to include them in our structural refinements of EG.

In order to include dipole-moment components as observables in our least-square refinements, we followed the approach adopted by Nandi, et al.²¹ We assumed the total dipole moment in EG to be the resultant of two $\text{-CH}_2\text{OH}$ group moments²². The group moment was assigned the magnitude found for $\text{CH}_3\text{-OH}$ ²³, and a direction defined by its angle made with the C-O bond. This angle was given the value found in $\text{CH}_3\text{-OH}$ (62°). The group moment was resolved into two "bond" components, one collinear with the O-H bond and one collinear

with the C-O bond comprising a $\text{H}_3\text{C-O}$ fragment. The directions of the components were defined as a consequence of an assignment of the oxygen atom as negative both with respect to the hydrogen atom and the methyl group. During the least square refinements the Cartesian coordinates for each atom in the principal axis system were calculated. The two "bond" components were used to calculate the bond moments using Cartesian coordinates and the results were added separately for each of the three components.

The structure refinement included simultaneous fitting by least squares of the calculated molecular intensity distribution to the experimental ones obtained from the average intensity curves, the calculated B_z rotational constants to the observed B_o corrected to B_z , and the calculated dipole moment components (μ) to those observed from microwave spectroscopy. These corrections are calculated from force field which will be discussed later. The weight matrix was diagonal, with each intensity point weighted equally. During the refinement, tests were made to investigate the effects of the available microwave observables on the overall structural results as obtained by electron diffraction alone. Our objective was to allow the microwave data to dominate the refinement of the C-O torsion angles. For the final refinement, the weights were adjusted in such a way that the sum of the weighted squares of the rotational constants was about 1500 times the sum of the weighted squares of the diffraction intensities while the sum of the weighted squares of the dipole-moment components was about 0.2 times the sum of the squares of the diffraction data. The dipole moment components

were given equal relative weights while the rotational constants were given unit weight, except for the largest one which was given 1/3 as much. The refinement was done separately on each isotopic species of EG. Because of expected high correlation among the parameters $r(\text{O-H})$, $l(\text{O-H})$, $r(\text{C-H})$, and $l(\text{C-H})$, and among $r(\text{C-C})$, $l(\text{C-C})$, $r(\text{C-O})$, $l(\text{C-O})$, it was decided to specify the distance parameters as averages and differences. The parameters chosen for the refinement and their definitions were as follows:

$\langle \text{CH,OH} \rangle = [2r(\text{C-H}) + r(\text{O-H})]/3$; $\Delta(\text{CH,OH}) = r(\text{C-H}) - r(\text{O-H})$; $\langle \text{CC,CO} \rangle = [r(\text{C-C}) + 2r(\text{C-O})]/3$; $\Delta(\text{CC,CO}) = r(\text{C-C}) - r(\text{C-O})$; $\langle \angle \text{COH} \rangle = [\angle \text{C}_1\text{O}_2\text{H} + \angle \text{C}_6\text{O}_7\text{H}]/2$; $\Delta(\text{COH}) = [\angle \text{C}_1\text{O}_2\text{H} - \angle \text{C}_6\text{O}_7\text{H}]$; $\angle \text{CCO}$; $\angle \text{CCH}$; $\tau(\text{OCCO})$; $\tau(\text{CC}_1\text{O}_2\text{H})$, and $\tau(\text{CC}_6\text{O}_7\text{H})$. The zero dihedral angles were defined as that in which oxygen atoms are cis to each other and the hydroxyl protons lie anti with respect to C-C bond. Rotation about a bond is positive when, looking along the bond, the near group (atom) rotates in counter clockwise direction. In the case of C-H and O-H, the simultaneous refinement of the corresponding amplitudes was unsuccessful. Therefore, the calculated amplitudes from the force field were assumed.

Structure Analysis

One of the goals in the reinvestigation of EG was to determine the equilibrium composition at higher temperatures. As shown in Figure 3, the barely resolved peak at 2.80 Å represents the 0··0 distance for the gauche form. Like the other peaks in the range

$r \geq 2.0 \text{ \AA}$, its height is slightly diminished while its width slightly increased at the higher temperature. This is due to the larger amplitude of vibration. On the other hand, the $0\cdots 0$ distance for the anti form is expected to be at about $3.66 \text{ \AA}$. As shown, there is no significant change, if any, in the appearance of the RD curve about this region. Therefore, no significant increase in the amount of anti form is indicated.

Our structure analysis is complicated by the fact that rotational constants have not been measured for the most abundant isotopic species, (OH₂O), of EG. Since the rotational constants and dipole moment components are valuable pieces of information for the investigation of the internal rotation, we decided to use the experimental microwave data from glycol- $0\text{-}d_2$ (ODOD) and the two possible 0 -monodeuterated isotopic species (OHOD, ODOH), where either the H atom or the D atom is involved in hydrogen bonding¹⁵. The implicit assumption that the molecular structure of EG is the same as the deuterated species except for the torsion angles was maintained throughout the structure analysis. The three ground-state rotational constants (B_0) for the two isotopic species were converted to B_z according to

$$B_z = B_0 + \frac{1}{2} \sum \alpha_i d_i^{\text{har}},$$

where α_i is the rotation-vibration interaction coefficient, d the vibrational degeneracy of the normal modes, and the summation sign extends over all of the $3N - 6$ vibrational modes. The experimental

values for the B_o 's were obtained from Walder, et al.¹⁴ for the ODOO species and from Caminati, et al.¹⁵ for the O-monodeuterated species, which with our harmonic vibration corrections gave (in MHz) $A_z = 14394.63 + 9.52$, $B_z = 5276.27 - 6.31$, and $C_z = 4324.53 - 1.85$ for the ODOO species, $A_z = 15126.955 + 6.70$, $B_z = 5311.085 - 6.66$, $C_z = 4412.157 - 2.04$ for OHOD, and $A_z = 14620.287 + 7.78$, $B_z = 5548.481 - 7.41$, $C_z = 4517.854 - 2.27$ for ODOH, respectively. The above corrections were obtained from a force field using known geometry and the observed vibrational frequencies. These, together with the experimentally measured dipole moment components^{14,15} for ODOO and OHOD species, were used in separate refinements. The geometrically consistent set of distances $r_\alpha^\circ = r_z$ that may be used to calculate the B_z is related to the r_a set used to calculate scattered intensities by the following equations

$$\begin{aligned} r_a^T &= r_g^T - l^2/r_\alpha^\circ \\ &= r_\alpha^\circ + 3/2 a_3 [(l^2)^T - (l^2)^\circ] + K^\circ + \delta r^T - (l^2)^T/r_\alpha^\circ, \end{aligned}$$

where a_3 is a Morse function anharmonicity constant estimated to be $2.13 \text{ \AA}^{-1}$ for C-C, $1.98 \text{ \AA}^{-1}$ for C-H, $2.30 \text{ \AA}$ for O-H; the perpendicular amplitude corrections K° , and the centrifugal distortions δr were calculated from the force field discussed below.

Infrared spectra of several isotopes of EG in Ar and Xe-matrices have been studied by Frei, et al.²⁴. For our force field calculation we began with a trial set of force constants taken from their valence force field, our model of the structure, and their observed vibrational frequencies in Ar-matrix for the 23 observed

Table I. Symmetry Coordinates, Force Constants, and Wavenumbers for Ethylene Glycol.

Species	Symmetry Coordinates	Force Constants		Wavenumbers		
		i, j	F _{i, j}	ω_{obs}	ω_{obs}	ω_{calc}
A	S ₁ = r ₂₃	1, 1	7.507	3667.0		0
	S ₂ = r ₇₈	2, 2	7.351	3629.0		0
	S ₃ = $\frac{1}{2}\Delta(r_{14} + r_{15} + r_{69} + r_{610})$	3, 3	4.635	2968.0		1
	S ₄ = $\frac{1}{2}\Delta(r_{14} - r_{15} + r_{69} - r_{610})$	4, 4	4.670	2940.0		0
	S ₅ = $\frac{1}{2}\Delta(r_{14} + r_{15} - r_{69} - r_{610})$	5, 5	4.631	2891.0		0
	S ₆ = $\frac{1}{2}\Delta(r_{14} - r_{15} - r_{69} + r_{610})$	6, 6	4.608	2885.0		0
	S ₇ = r ₁₂	7, 7	4.761	1468.0		-8
	S ₈ = r ₆₇	8, 8	4.955	1459.5		0
	S ₉ = r ₁₆	9, 9	4.355	1415.5		-17
	S ₁₀ = α_{123}	10, 10	0.543	1383.0		5
	S ₁₁ = α_{678}	11, 11	0.629	1350.0		4
	S ₁₂ = $\frac{1}{2}\Delta(\alpha_{415} + \alpha_{9610})$	12, 12	0.607	1271.0		3
	S ₁₃ = $\frac{1}{2}\Delta(\alpha_{415} - \alpha_{9610})$	13, 13	0.492	1246.0		18
	S ₁₄ = $\frac{1}{2}\Delta(\alpha_{614} + \alpha_{615} - \alpha_{214} - \alpha_{215})$	14, 14	0.629	1163.0		-14
	S ₁₅ = $\frac{1}{2}\Delta(\alpha_{1610} + \alpha_{169} - \alpha_{7610} - \alpha_{769})$	15, 15	0.654	1100.0		-8
	S ₁₆ = $\frac{1}{2}\Delta(-\alpha_{614} + \alpha_{615} - \alpha_{214} + \alpha_{215})$	16, 16	1.250	1069.0		-11
	S ₁₇ = $\frac{1}{2}\Delta(-\alpha_{1610} + \alpha_{169} - \alpha_{7610} + \alpha_{769})$	17, 17	1.253	1041.5		18
	S ₁₈ = $\frac{1}{2}\Delta(-\alpha_{614} + \alpha_{615} + \alpha_{214} - \alpha_{215})$	18, 18	0.931	880.0		-5
	S ₁₉ = $\frac{1}{2}\Delta(-\alpha_{1610} + \alpha_{169} + \alpha_{7610} - \alpha_{769})$	19, 19	0.887	865.0		10
	S ₂₀ = α_{216}	20, 20	1.255	514.0		1
	S ₂₁ = α_{167}	21, 21	1.290	361.0		-12
	S ₂₂ = τ_{1678}	22, 22	0.051	309.0		6
	S ₂₃ = τ_{6123}	23, 23	0.036	268.0		0
	S ₂₄ = τ_{2167}	24, 24	0.198	185.0		0
		7, 8	0.50			
		7, 9	0.14			
		8, 14	-0.25			
		9, 14	0.08			
		8, 15	-0.16			
		9, 15	0.15			
		7, 19	0.10			

normal modes. The unobserved frequency was estimated to be 25 cm^{-1} lower than the corresponding Xe-matrix frequency (210 cm^{-1}), as was used in their least squares fit. The overall fit to the observed fundamental frequencies was adjusted to within 18 cm^{-1} or less (less than 1.5%). The calculated force constants are very close to those given by Frei, et al., when the error limits are taken into account. Table I shows this approximate force field together with the symmetry coordinates, force constants, and the overall fit to the observed frequencies. As seen, due to the asymmetric nature of the gauche conformer, symmetry blocking of the "symmetry coordinates" was not possible. Hence, all of the twenty-four normal modes are found in one block. It is worth mentioning that the force field has no special significance in terms of its effect on the calculated bond distances and valence angles. However, its inclusion in our calculation would be necessary in order to convert r_z 's ($r_z = r_\alpha^\circ$) obtained from the rotational constants (B_z 's) to the appropriate interatomic distances used in the electron diffraction intensity data (r_a). The correction terms needed for this transformation are obtained from the force field calculations.

Results and Discussion

The values of the geometrical parameters obtained for the two isotopic species do not differ significantly at different temperatures, except for the torsion angles within each species. In the first place, our experimental results indicate that the amount of

Table II. Structural Results^a for isotopic species of Ethylene Glycol.

	376 K			733 K		
	ODOH	OHOD	ODOD	ODOH	OHOD	ODOD
$\langle \text{CH}, \text{OH} \rangle^b$	1.068(5)	1.066(7)	1.070(5)	1.074(5)	1.075(8)	1.075(6)
CH-OH	0.167(6)	0.164(10)	0.168(7)	0.181(6)	0.180(10)	0.181(7)
$\langle \text{CC}, \text{CO} \rangle$	1.455(2)	1.455(3)	1.456(2)	1.462(2)	1.462(3)	1.463(2)
CC-CO	0.088(6)	0.090(9)	0.092(6)	0.106(6)	0.105(7)	0.109(5)
$\langle \angle \text{COH} \rangle$	103.2(17)	99.2(29)	101.1(16)	96.8(22)	91.5(33)	97.1(19)
$\angle \text{CCO}$	110.6(4)	110.0(4)	110.1(3)	110.4(5)	110.0(6)	109.9(4)
$\angle \text{CCH}$	112.2(7)	112.4(10)	111.2(15)	113.0(7)	113.9(10)	113.6(7)
$\tau_{\text{OC-CO}}$	53.8(22)	57.9(12)	58.4(10)	53.8(54)	55.7(14)	56.2(10)
$\tau_{\text{C}_1-\text{O}_2}$	14.5(153)	18.2(43)	20.0(36)	28.3(311)	19.9(49)	23.0(36)
$\tau_{\text{C}_6-\text{O}_7}$	180.2(112)	122.3(47)	135.6(34)	173.9(307)	123.9(47)	136.5(34)
$\Delta(\text{C})^c$	0.2	5.6	2.6	2.0	3.9	2.6
$\Delta(\text{B})$	0.1	-2.3	-1.1	9.6	-1.7	-1.0
$\Delta(\text{A})^d$	0.1	-0.6	-0.3	-15.0	-0.5	-0.3
$\delta(\text{c})^d$	-	0.04	-0.02	-	0.04	0.02
$\delta(\text{b})$	-	0.31	0.09	-	0.29	0.09
$\delta(\text{a})^e$	-	-0.05	-0.02	-	0.07	0.01
χ_f	0.019(111)	0.104(199)	0.067(120)	[0.280]	0.280]	0.280(270)
R	0.058	0.074	0.065	0.070	0.086	0.074

^a Distances (r_α^o) in angstroms, angles in degrees. ^b See text for definitions. ^c Differences between the observed corrected rotational constants (B_z 's in MHz) and the calculated ones. ^d Differences between the observed components of the dipole moment (μ 's, in Debye) and the calculated. ^e Mole fraction of the anti form. ^f $R = [\sum_i \omega_i^2 \Delta_i^2 / \sum_i \omega_i^2 (s_i I_i(\text{obsd})^2)]^{1/2}$ where $\Delta_i = s_i I_i(\text{obsd}) - s_i I_i(\text{calcd})$.

anti form (if any) is very small, perhaps less than 5%, even at 733 K. As far as the quality of the fit to microwave data is concerned, we have been able to obtain a better fit to the ODOD species than to the OHOD species where the hydrogen atom is involved in hydrogen bonding. As seen from Table II, the C-O-H bond angles in the three species are smaller than the value found in methanol (108.9°). This may be attributed to strong hydrogen bonds which seem to be present. (We assumed the C-O-H bond angles to be equal on each side. An attempt was made to refine the difference between the C-O-H angles, but it was unsuccessful). Also, no distinction was made between O-H bond distances. As Table III shows, the C-C and C-O bond distances are close to those of similar molecules isoelectronic with EG. For instance, $r_s(\text{C-C})$ for 2-aminoethanol³ was found to be about $1.526(16)$ Å and $r_a(\text{C-C})$ to be $1.518(6)$ Å for the ODOD species. However, our O...O distance for the gauche conformer does not agree with the previous electron-diffraction results which reported 2.97 Å¹¹. Our result is closer to the value 2.85 Å obtained by Burgess, et al.²⁵ from empirical theoretical methods. Our CCO angle is close to that in 2-fluoroethanol $112.2(1.9)^\circ$. Also the dihedral angle is within the range of the other dihedral angles such as ethylenediamine⁶ $64.0(4)^\circ$, 2-fluoroethanol $64.6(1.1)^\circ$, and 2-aminoethanol³ $55.4(20)$. Table IV shows the amplitudes and the vibration-rotation corrections (D) for some of the more important distances in EG. Table VI shows D...D distance from the r_s -located positions of the D's in the ODOD species from the microwave work. Here, we should emphasize that our principal coordinate axis system is

Table III. Interatomic Distances^a in Ethylene Glycol for the three isotopic species.

	376 K			733 K		
	ODOH	OHOD	ODOD	ODOH	OHOD	ODOD
C-O	1.426(2)	1.426(2)	1.426(2)	1.430(2)	1.430(2)	1.430(2)
C-H	1.133(6)	1.130(8)	1.135(6)	1.144(6)	1.144(9)	1.145(7)
C-C	1.514(6)	1.516(8)	1.518(6)	1.536(6)	1.535(7)	1.539(6)
O-H	0.991(6)	0.991(9)	0.992(7)	0.988(6)	0.990(10)	0.989(7)
C ₁ ·O ₇	2.418(4)	2.410(6)	2.412(5)	2.432(6)	2.425(7)	2.426(6)
C ₁ ·H ₉	2.203(14)	2.205(20)	2.189(24)	2.238(14)	2.246(19)	2.247(15)
O ₂ ·H ₄	2.072(8)	2.071(12)	2.091(18)	2.072(8)	2.064(11)	2.069(9)
O··O _g	2.790(18)	2.805(9)	2.814(8)	2.796(46)	2.795(10)	2.799(8)
C ₁ ·H ₃	1.903(20)	1.853(35)	1.877(19)	1.819(28)	1.749(44)	1.824(24)
C ₆ ··H ₃	3.198(34)	3.151(27)	3.166(17)	3.116(122)	3.086(38)	3.135(22)
C ₁ ··H ₈	2.230(25)	2.427(49)	2.351(26)	2.125(43)	2.306(53)	2.287(30)
O ₂ ··H ₈	2.300(64)	2.297(53)	2.240(28)	2.168(130)	2.175(53)	2.155(30)
O ₇ ··H ₃	3.567(102)	3.519(36)	3.520(30)	3.417(281)	3.454(46)	3.462(32)
O ₂ ··H ₉	3.401(10)	3.400(15)	3.397(17)	3.435(11)	3.437(14)	3.434(12)
O ₂ ··H ₁₀	2.766(22)	2.732(23)	2.720(26)	2.799(46)	2.783(24)	2.771(18)
O··O _a	3.667(6)	3.656(8)	3.659(6)	3.669(9)	3.666(10)	3.667(8)

^a Distances (r_a) in angstroms. Uncertainties are estimated 2σ .

Table IV. Selected amplitudes, and Vibration - Rotation Correction (D)^a of Ethylene Glycol.

Distance	376 K				733 K			
	OHOD	D×10 ³	ODOD	D×10 ³	OHOD	D×10 ³	ODOD	D×10 ³
C-O	0.058	4	0.058	4	0.061	6	0.061	6
C-C	0.060	3	0.060	3	0.063	6	0.063	6
C-H	0.079	15	0.079	15	0.079	15	0.079	15
O-H	0.070	40	0.070	40	0.070	40	0.070	40
C ₁ ·O ₇	0.081(6)	3	0.084(5)	3	0.110(9)	5	0.110(7)	5
C ₁ ·H ₉	0.105	8	0.156(57)	8	0.126	9	0.126	9
O ₂ ·H ₄	0.083(12)	9	0.087(10)	9	0.083(11)	10	0.083(8)	10
C ₁ ·H ₃	0.113	21	0.113	20	0.126	21	0.126	21
C ₆ ·H ₈	0.110	19	0.110	18	0.121	19	0.121	19
C ₆ ·H ₃	0.120	13	0.120	13	0.142	15	0.142	15
C ₁ ·H ₈	0.191	16	0.191	14	0.242	17	0.242	17
O ₂ ·H ₈	0.227	19	0.227	20	0.297	26	0.297	26
O ₇ ·H ₃	0.233	14	0.233	13	0.311	18	0.311	18
O ₂ ·H ₉	0.117	6	0.124	6	0.119	8	0.181	8
O ₂ ·H ₁₀	0.170	6	0.178	6	0.196	8	0.258	8
O ₂ ·O ₉	0.164	6	0.157	6	0.238	11	0.231	11
O _a ·O _a	0.085	* ^b	0.078	*	0.140	*	0.139	*

^a $D = r_a - r_\alpha + l^2/r_\alpha$. ^b Correction was not calculated. parenthesized quantities are estimated 2σ, those in curly brackets were refined as a group, those without attached uncertainties were calculated from the force field and were not refined.

Table V. Correlation Matrix (x100) for Ethylene Glycol (ODOD) at 376 K.

	$\sigma L \times 100$	r_1	r_2	r_3	r_4	$\angle_5$	$\angle_6$	$\angle_7$	τ_8	τ_9	τ_{10}	l_{11}	l_{12}	l_{13}	l_{14}	l_{15}	l_{16}
1 <CH,OH>	0.189	100															
2 CH-OH	0.265	19	100														
3 <CC,CO>	0.073	15	19	100													
4 CC-CO	0.221	12	12	80	100												
5 $\angle CCO$	11.3	10	-3	-57	-55	100											
6 < $\angle COH$ >	56.5	-53	-12	-36	-23	-38	100										
7 $\angle CCH$	53.1	39	25	47	36	-15	-15	100									
8 $\tau(OCCO)$	35.0	-49	-22	-29	-25	-45	61	-60	100								
9 $\tau(C_1-O_2)$	126.0	3	2	-9	-6	66	-45	37	-51	100							
10 $\tau(C_6-O_7)$	118.9	12	5	13	10	-15	26	-10	9	-24	100						
11 $l(C-O)$	0.086	6	5	-46	-57	39	-1	-24	8	4	-6	100					
12 $l(C_1-O_7)$	0.142	8	7	-3	-2	-6	-4	-20	15	-18	4	25	100				
13 $l(C_1-H_9)$	1.989	-12	-6	-28	-25	-6	12	-76	56	-49	15	24	51	100			
14 $l(O_2-H_4)$	0.313	2	-3	16	12	13	-15	25	-30	27	-2	-3	-22	-44	100		
15 $l(O_2-O_7)$	0.715	8	5	-4	-4	-3	10	24	-7	6	-2	-1	-30	-42	19	100	
16 $l(O_2-H_9)$	1.927	-1	-2	4	6	-11	6	-3	4	-1	4	-2	31	20	-21	-43	100

Table VI. r_s (Å) located positions of D's in ODO species in the principal axis system.^a

	Electron Diffraction		Caminati, et al.			
			calcd.		exptl.	
	D_3	D_8	$H(D_3)$	$H'(D_8)^b$	$H(D_3)$	$H'(D_8)$
x	-0.419(37)	0.023(48)	0.334	0.227	0.310(5)	0.095(16)
y	0.551(26)	1.199(16)	0.629	1.257	0.629(25)	1.286(1)
z	2.078(32)	-0.764(24)	2.122	-0.745	2.128(1)	0.801(2)
$r(D_3 \cdots D_8)$	2.949		2.937		3.009	

^a Uncertainties (ED) are estimated 2σ . ^b $H'(D_8)$ participates in hydrogen bonding.

not identical to that from the microwave data, mainly due to the different assumptions made in the structural analyses. However, in our slightly different principal axis system for our molecule, the $D_8 \cdots D_3$ distance shown in the table agrees to within 0.06 Å (low temperature) from the microwave work by Caminati, et al.¹⁵

As noted above, the gauche form of the molecule is stabilized by an intra-molecular hydrogen bonding interaction of the type $O-H \cdots O$. The hydrogen bond formed is very non-linear, as is the case in similar molecules mentioned above. The value for $\angle O-H \cdots O$ is $117.5^\circ(19)$, which corresponds to $r_\alpha^\circ(O \cdots H) = 2.243(28)$ Å for the ODOD species, considerably less than the sum of the O, H van der Waals radii (2.60 Å). This also suggests a strong interaction between the three atoms involved which presumably confirms the presence of internal hydrogen bonds. As to the presence of the "gauche effect", no quantitative conclusion can be drawn. Perhaps their combined effects will be responsible for the large percentage of the gauche form.

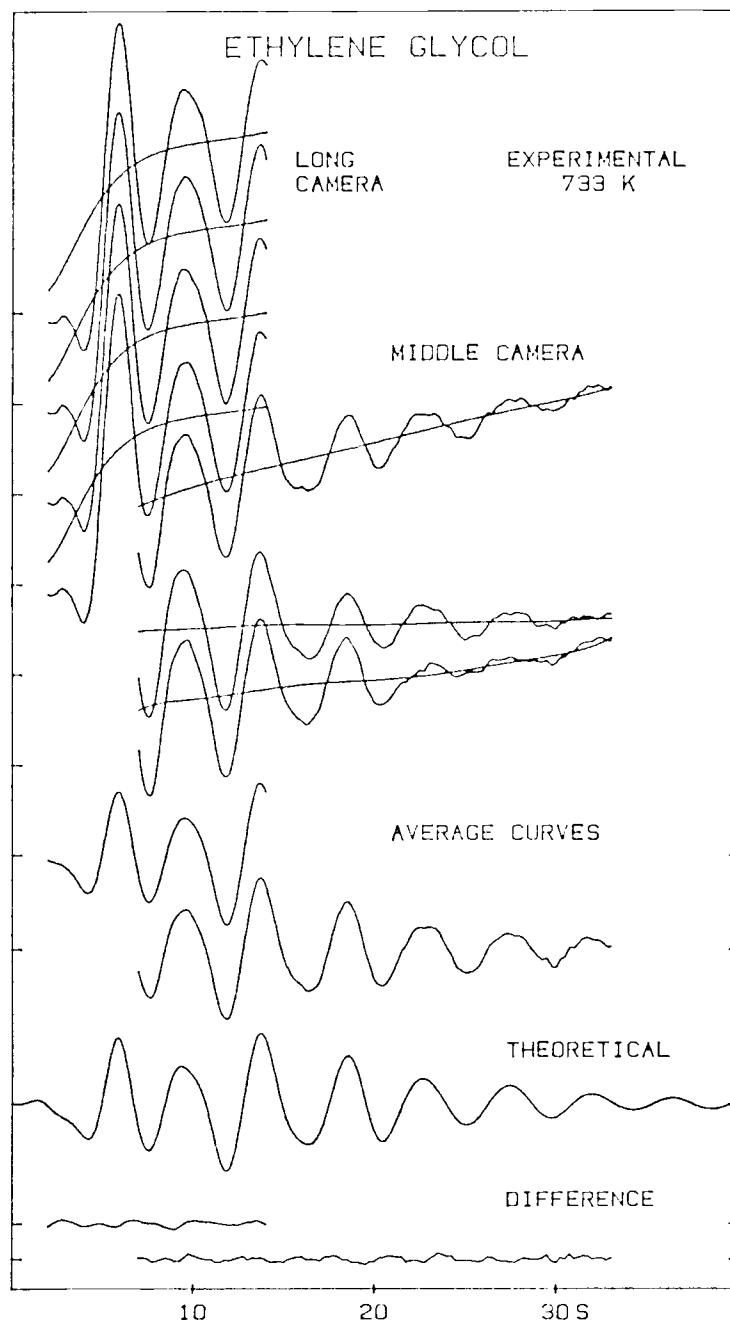


Figure 4. Intensity curves and backgrounds for 733 K experiment.

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CONFORMATIONAL ANALYSIS. ETHYLENEDIAMINE.
MOLECULAR STRUCTURE, COMPOSITION, HYDROGEN BONDING,
ANTI-GAUCHE ENERGY AND ENTROPY DIFFERENCES.

Introduction

During the last two decades, ethylenediamine ($(\text{CH}_2\text{-NH}_2)_2$, EDA), diagrammed in Figure 5, has been studied by several methods. Infra-red spectra of the vapor showed the presence of either cis or the gauche form.¹ An electron diffraction investigation indicated the presence of a dominant gauche conformer in the vapor phase ($50\text{-}120\text{ }^\circ\text{C}$)², and the fraction of any other conformer, if present, was estimated to be less than 5%. No definite conclusions were reached regarding the orientation of the NH_2 groups. A quantum chemical conformational analysis (LCAO-EHMO) revealed that the most stable conformation of the molecule is the gauche conformer³, confirming the previous results obtained by gas electron diffraction. It also showed that the anti form is the next most stable conformation of EDA. An extensive ab initio calculation on 1,2-disubstituted ethanes⁴ showed that EDA has two most stable gauche conformations which are stabilized by intramolecular hydrogen bonding. Microwave study has also confirmed the presence of the two gauche conformations, one being $0.3 \pm 0.2\text{ Kcal mol}^{-1}$ more stable than the other.⁵ An ab initio quantum mechanical calculation at 4-21G level on conformational analysis and molecular structure based upon gradient geometry optimization predicts the presence of the most stable gauche

ETHYLENEDIAMINE

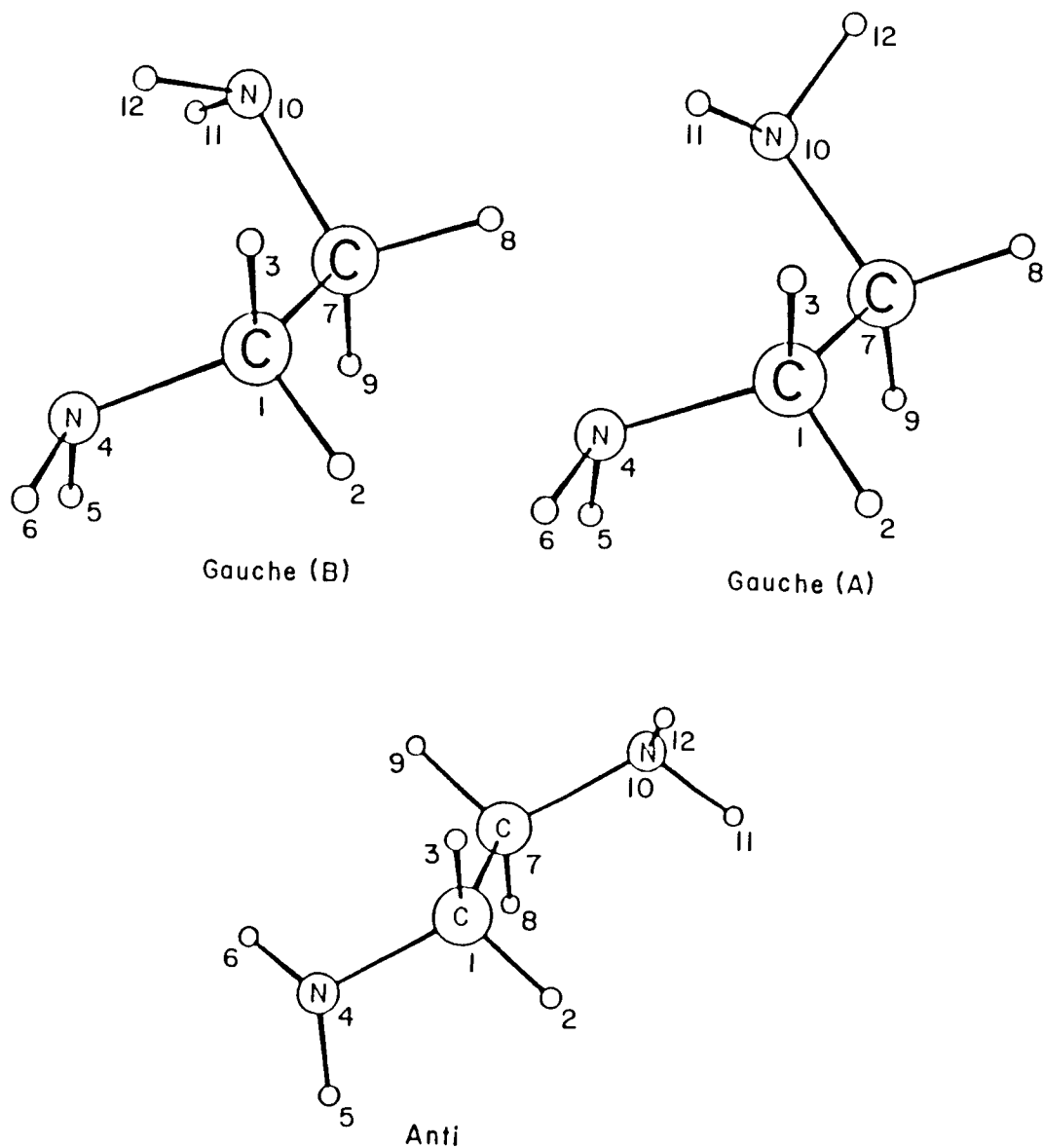


Figure 5. Atom numbering for anti- and gauche-ethylenediamine.

conformations of EDA ($\angle \text{NCCN} = 57.8^\circ$ and 61.9°).⁶

As a part of our studies on the structures of 1,2-disubstituted ethanes, we undertook EDA molecule to investigate the effects of the substituent groups on carbon atoms involving electronegative elements ($-\text{NH}_2$). In particular, we were interested in determining the role of intramolecular hydrogen bonding and possibly the "gauche effect"⁷ in the stabilization of the gauche conformers. Also, from the quantitative data about the rotameric composition of EDA as function of temperature, it was hoped to obtain the energy and entropy differences of the three conformers. Our results indicated that the energy difference between the two gauche forms was so small that we were not able to arrive at a quantitative conclusion.

Experimental Section

The EDA used was a Baker Analyzed Reagent grade of high purity (assay, 98.8) which was purified further under reduced pressure (120 torr) by distillation. At the beginning of the experiment, the mass spectrum of this compound showed thermal decomposition at about 783 K. Therefore, attempts were made to take diffraction photographs at nozzle-tip temperatures of 343 K, 463 K and 713 K. The sample bath was kept at 310 - 320 K.

About 30 diffraction photographs were made in the OSU apparatus with an r^3 sector. Experimental conditions are summarized in Table VII. Analysis of the structure at each temperature was based on at least three plates. The procedures for obtaining the scattered

Table VII. Experimental Conditions

	343 K		463 K		713 K	
	long	intermed	long	intermed	long	intermed
	camera	camera	camera	camera	camera	camera
Nozzle-to-plate distance/mm	744.920	301.090	745.515	301.060	744.580	300.980
Electron wavelength/Å	0.058	0.059	0.058	0.058	0.060	0.060
Exposure time/s	45	120-150	45-90	120-180	60-75	120-150
Electron beam current/ μ A	0.40	0.48-0.51	0.37	0.38-0.53	0.42-0.46	0.55-0.56
No. of plates used	3	3	5	4	3	3
Range of data in $\text{s}/\text{\AA}^{-1}$	2.0-13.0	7.0-32.5	2.0-13.0	7.0-33.5	2.0-13.5	8.0-32.5
Data interval in $\text{s}/\text{\AA}^{-1}$	0.25	0.25	0.25	0.25	0.25	0.25
Ambient apparatus press./torr $\times 10^5$	1.2	1.0	0.5	0.7	0.9	1.0

intensity distribution have been described.⁸ Calculated backgrounds were subtracted from the intensity data from each plate to provide molecular intensities in the form $sI_m(s)$ ⁹ given by

$$sI_m(s) = k \sum_i \sum_j A_i(s) A_j(s) r_{ij}^{-1} \cos(\eta_i - \eta_j) \exp(-l_{ij}^2 s^2 / 2) \sin(r_{ij} - \kappa_{ij} s^2)$$

where $s = (4\pi/\lambda)\sin\theta$ and 2θ = the scattering angle. Figure 6, shows the $s^4 I_t(s)$ curves and the calculated backgrounds at 343 K temperature. Figure 7 shows the corresponding experimental radial distribution curves calculated by the fourier transformation of functions

$$r(Dr) = 2/\pi \left[\Delta s \sum I'(s) \exp(-Bs^2) \sin rs \right]$$

where $I'_m(s) = Z_C Z_N A_C^{-1} A_N^{-1} sI_m(s)$ and B was equal to $0.0025 \text{ \AA}^{-1}$. The electron-scattering amplitudes for our experimental conditions were interpolated from literature values.¹⁰

Structure Analysis

As seen in Figure 7, the radial distribution curves for EDA are very similar over the wide range of temperatures (343 - 713 K). Perhaps the most notable change occurs at about 2.9 Å, where the corresponding peak for N··N distance of the gauche conformer decreases in intensity slightly as the temperature increases. The amount of the anti form does not change significantly as the temperature rises. It is also evident from the relative areas of

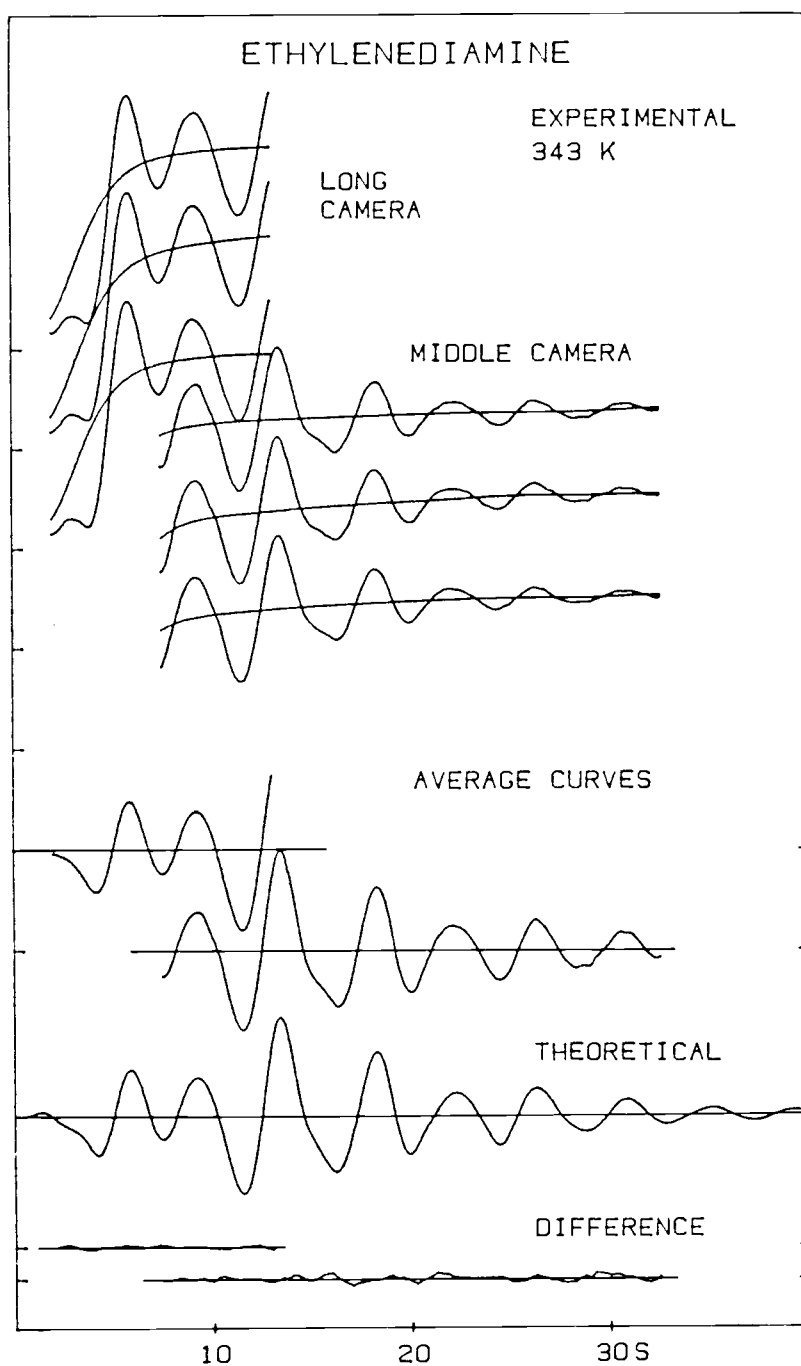


Figure 6. Intensity curves for experiments at 343 K. The experimental curves are for s^4I_t shown superimposed on the final backgrounds. The theoretical intensity curve is sI_m for the final model. The difference curves are experimental minus theoretical sI_m .

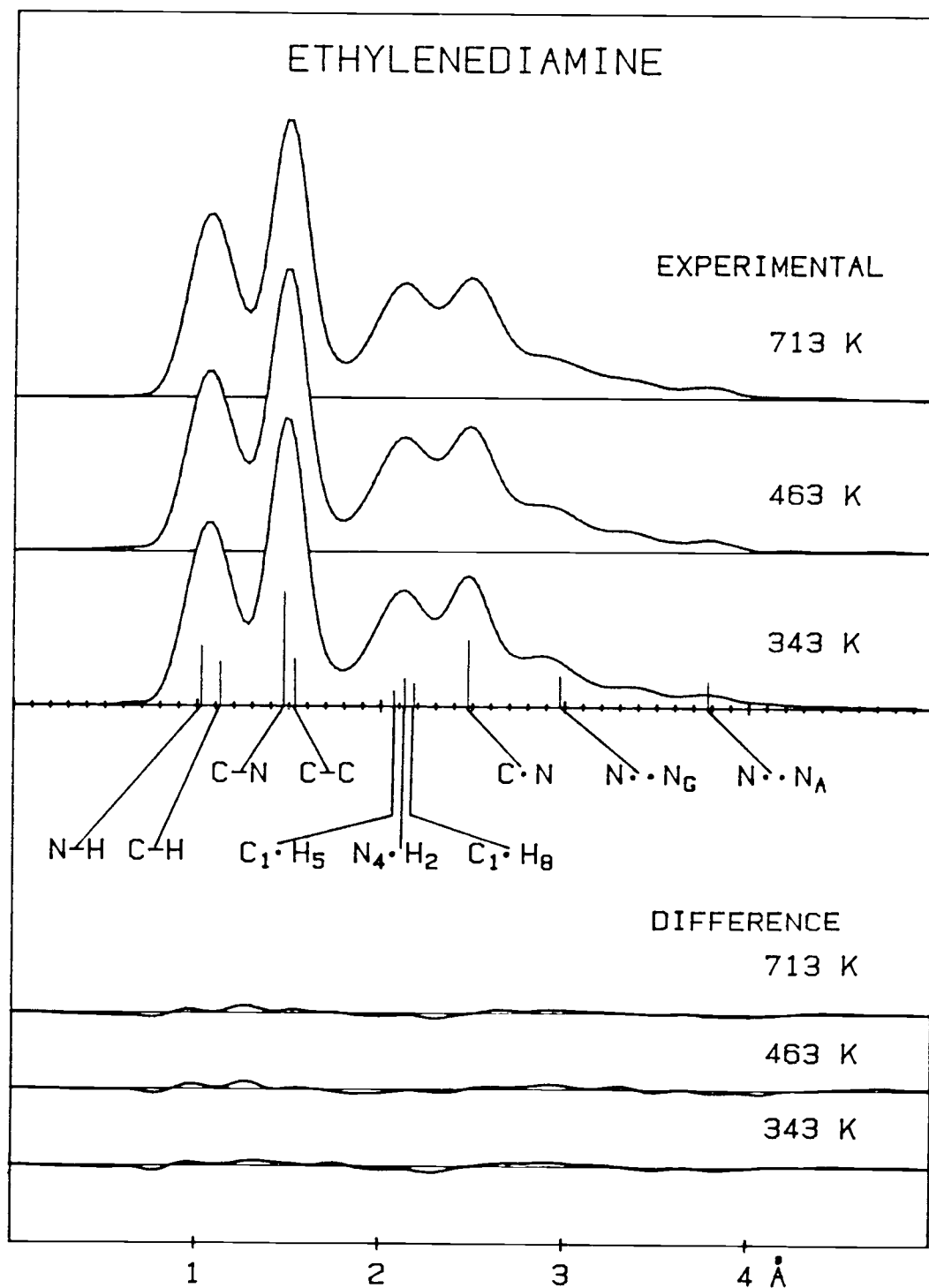


Figure 7. Radial distribution curves. The experimental curves are calculated from composites of the molecular intensities from the two camera distances. Difference curves are experimental minus theoretical for the final model.

the peaks at about 2.9 Å (gauche) and 3.8 Å (anti) that the gauche conformer predominates at all temperatures.

The least square refinements were carried out by fitting the theoretical intensity distribution to the three averaged experimental intensities for each temperature. Since electron diffraction data alone is not sufficient for investigating the torsional motions involving hydrogen atoms in -NH_2 groups, we have included the rotational constants for the gauche forms. This imposes a valuable constraint on our refinements. Because of the effect of vibrational averaging, the r_a distances required by electron diffraction data are not suitable for the observed rotational constants (B_o), the refinements were based upon the r_α° ($r_\alpha^\circ = r_z$) set of distances which were used to calculate the rotational constants B_z related to the observed B_o by

$$B_z = B_o + 1/2 \sum \alpha_s^{\text{har}}$$

The observed values⁵ (in MHz) for gauche A were: $A_o = 14472.017$, $B_o = 5229.206$, $C_o = 4405.511$. The corrected values $A_z = 14472.447$, $B_z = 5222.656$, $C_z = 4403.561$. For the second gauche conformer (B) the observed values were: $A_o = 14355.536$, $B_o = 5125.301$, $C_o = 4356.292$ and the corrected values $A_z = 14355.416$, $B_z = 5118.751$, and $C_z = 4354.692$. The r_a distances for three temperatures were calculated from the r_α° obtained from B_z 's according to

$$r_a^T = r_a^\circ + (3/2)a_3[(l^2)^T - (l^2)^\circ] + \delta r^T + K^\circ - (l^2)^T / r_a^\circ,$$

where a_3 is the Morse function anharmonicity constant, δr is a correction for the effect of centrifugal distortion, K is a correction which accounts for the perpendicular motions to the internuclear axis, l is the overall amplitude of vibration; the superscripts designate temperatures. Since the vibrational spectrum of gaseous EDA in the gauche form has not been reported, an approximate force field was calculated based upon the observed vibrational frequencies¹ for the cis conformer (C_{2v} symmetry). For the unobserved frequencies the calculated values² were given. As with ethylene glycol, because of the asymmetrical nature of the gauche conformers, symmetry blocking of the "symmetry coordinates" was not possible. Hence all symmetry coordinates have been combined. In addition to the rotational constants, we have included the observed components of the dipole moment from microwave data.⁵ The dipole moment components are in effect more sensitive to the torsional motion of the $-NH_2$ groups than the rotational constants. Their inclusion will yet add another constraint to our least square refinements. The combined effect of the rotational constants and the dipole moments will allow us to manage the torsional problems even better. Since no reliable values for the bond moments are available at the present time, we have followed the same approach as in ethylene glycol. We assumed the total dipole moment of EDA molecule to be the resultant of two $-CH_2-NH_2$ group moments. The total group moment was assigned the magnitude (1.296 D) found for

$\text{CH}_3\text{-NH}_2$ ¹² and its direction defined by the angle made with the C-N bond. This angle is given the value found for $\text{CH}_3\text{-NH}_2$ (97.1°). The group moment was then resolved into two "bond" components, one collinear with the resultant obtained from the two N-H bond components, and one collinear with the C-N bond constituting a $\text{CH}_2\text{-NH}_2$ segment. Their directions were chosen in such a way that nitrogen atom was negative both with respect to hydrogen and the methyl group. Using the Cartesian coordinates, the bond moments were obtained and the three dipole moment components were calculated. The rotational constants were weighted about 700 times the diffraction intensities, while the overall weight on dipole moment components were adjusted to 0.02 times the diffraction data.

As has generally been the case, in constructing our model we assumed that all conformers have the same structure except for the torsion angles. This gives us nine parameters for the specification of EDA. A convenient set is:

$r\langle\text{CH},\text{NH}\rangle = [r(\text{C-H}) + r(\text{N-H})]/2$, $\Delta(\text{CH-NH}) = r(\text{C-H}) - r(\text{N-H})$,
 $r\langle\text{CC},\text{CN}\rangle = [r(\text{C-C}) + 2r(\text{C-N})]/3$, $\Delta(\text{CC-CN}) = r(\text{C-C}) - r(\text{C-N})$,
 $\angle\text{CCN}$, three internal torsions of the gauche conformer $\tau(\text{NCCN})$,
 $\tau(\text{C}_1\text{-N}_4)$, $\tau(\text{C}_7\text{-N}_{10})$ and the mole fraction of the gauche form χ . The following angles were assumed: $\angle\text{HCH} = 108^\circ$, $\angle\text{HNH} = 105.9^\circ$ ¹¹, $\angle\text{CCH} = 109.2^\circ$, $\angle\text{CNH} = 112.1^\circ$ ¹¹.

Discussion

Molecular Structure and Bonding. In our structure analysis, the assumption was made that the two gauche conformers are identical except for the torsion angles. Hence, the parameter values given in Table VIII are in fact averages for the two forms at a given temperature. The ratio (χ) of the gauche and the anti forms were about the same within the limits of errors before and after averaging which in turn supports our assumption. Furthermore, the normal vibrational amplitudes obtained before and after averaging are about the same as would be for a single conformer. We conclude that the bond distances and bond angles are nearly the same in the two rotamers. To investigate the orientation of the $-\text{NH}_2$ groups, dipole moment components of methylamine molecule¹² were used to generate the bond moments and subsequently the group (dipole) moments for EDA. The assumption made here was that the total dipole moment for EDA can be generated by combining two separate methylamine molecules through their C-H bonds and eliminating the hydrogens from each side to form C-C bond. Even though this assumption may not seem to be a good one, there is a great deal of experimental evidence that supports it. For instance, the experimentally measured group moments for the gaseous CH_3-NH_2 and $\text{C}_2\text{H}_5-\text{NH}_2$ are 1.23 D and 1.2 D, respectively.¹³ For other isoelectronic molecules like gaseous CH_3-OH , $\text{C}_2\text{H}_5-\text{OH}$ the measured values are 1.69 D. This indicates that the attachment of R-group has little effect to the electronic arrangement of the $-\text{NH}_2$

Table VIII. Results for Structural Parameters of Ethylenediamine^a

	343 K		463 K		713 K	
$\langle \text{CH}, \text{NH} \rangle^b$	1.060(3)		1.063(3)		1.061(3)	
$\Delta(\text{CH}, \text{NH})$	0.107(20)		0.115(14)		0.103(16)	
$\langle \text{CC}, \text{CN} \rangle$	1.489(3)		1.489(3)		1.490(3)	
$\Delta(\text{CC}, \text{CN})$	0.058(18)		0.049(23)		0.061(16)	
$\angle \text{CCN}$	111.4(5)		111.8(5)		111.7(5)	
	A	B	A	B	A	B
$\tau(\text{NCCN})_G^c$	55.3(8)	63.6(9)	56.4(12)	63.5(8)	55.6(11)	63.2(6)
$\tau(\text{C}_1\text{-N}_4)_G$	-74.7(74)	-38.8(108)	-89.6(100)	-62.6(138)	-86.3(90)	-62.7(141)
$\tau(\text{C}_7\text{-N}_{10})_G$	248.0(59)	112.3(57)	239.5(74)	117.3(84)	238.4(67)	119.6(84)
$\angle \text{CNH}$	{112.1}	{112.1}	{112.1}	{112.1}	{112.1}	{112.1}
$\angle \text{CNH}_B$	{101.1}	{105.7}	{101.1}	{105.7}	{101.1}	{105.7}
$\Delta_1(\text{MHz})^d$	8.6	-0.6	14.7	-2.0	17.1	-2.7
Δ_2	-1.8	-3.2	-2.4	1.8	-4.3	1.0
Δ_3	-0.3	-3.7	0.5	1.5	-1.6	0.3
$\delta_1(\text{D})^e$	0.21	<0.01	0.46	-0.31	0.44	-0.25
δ_2	-0.03	0.18	0.19	0.22	0.20	0.23
δ_3	-0.13	-0.46	-0.10	-0.31	-0.20	-0.33
χ^f	0.187(110)	0.181(120)	0.223(118)	0.207(111)	0.222(124)	0.230(112)
R^g	0.053	0.053	0.061	0.062	0.052	0.052

^a Distances (r_α) in angstroms, angles ($\angle_\alpha$) in degrees. Values in parentheses are estimated 2σ . ^b See text for definitions. ^c Torsion angles for gauche form. ^d Difference between observed and calculated rotational constants.

^e Difference between observed and calculated dipole moments. ^f Mole fraction of anti form.

^g $R = [\sum \omega_i \Delta_i^2 / \sum \omega_i (s_i I_i(\text{obsd}))^2]^{1/2}$, where $\Delta_i = s_i I_i(\text{obsd}) - s_i I_i(\text{calcd})$.

Table IX. Distances (r) and Amplitudes (l) in Ethylenediamine^{a,b}

	343 K			463 K			713 K		
	r _a ^c	l ^d _{exptl}	l _{calcd}	r _a	l _{exptl}	l _{calcd}	r _a	l _{exptl}	l _{calcd}
N-H	1.024(8)	0.074	(10)	1.023(7)	0.073	(11)	1.027(4)	0.074	(11)
C-H	1.123(11)	0.078		0.079	1.130(9)		0.078	0.079	
C-N	1.471(4)	0.059	(4)	1.473(6)	0.062	(4)	1.472(4)	0.063	(4)
C-C	1.528(12)	0.067		0.054	1.523(18)		0.071	0.056	
C·N	2.476(4)	0.083(5)	0.073	2.479(6)	0.092(7)	0.079	2.483(5)	0.100(7)	0.093
N ₄ ·H ₂	2.119(9)	0.118	(9)	2.126(8)	0.124	(10)	2.118(5)	0.123	(10)
C ₁ ·H ₈	2.170(12)	0.122		0.113	2.168(16)		0.128	0.116	
Gauche (A)									
C ₁ ·H ₅	2.077(7)	0.111	(9)	2.078(8)	0.116	(10)	2.079(5)	0.115	(10)
C ₇ ·H ₁₁	1.941(6)	0.116		0.107	1.943(7)		0.120	0.108	
N ₄ ··N ₁₀	2.891(11)	0.138(20)	0.135	2.914(16)	0.156(30)	0.154	2.907(13)	0.196(39)	0.188
C ₁ ··H ₁₁	2.282(14)		0.146	2.276(8)		0.155	2.273(6)		0.176
C ₁ ··H ₁₂	3.212(35)		0.124	3.162(43)		0.132	3.152(43)		0.149
N ₄ ··H ₈	3.432(12)	0.100	(39)	3.436(7)	0.124	(63)	3.441(9)	0.116	(53)
N ₄ ··H ₉	2.775(8)	0.147		0.155	2.774(10)		0.192	0.166	
N ₄ ···H ₁₁	2.490(55)		0.234	2.398(48)		0.263	2.403(71)		0.316

Table IX. (cont.)

Gauche (B)												
C ₁ · H ₅	2.077(7)	0.111	} (10)	0.102	2.078(6)	0.118	} (10)	0.104	2.080(8)	0.114	} (10)	0.109
C ₇ · H ₁₂	2.000(7)	0.111		0.102	2.001(6)	0.118		0.104	2.002(7)	0.114		0.109
N ₄ ··· N ₁₀	2.970(10)	0.140(24)		0.133	2.982(15)	0.157(28)		0.152	2.979(15)	0.192(38)		0.186
C ₁ ··· H ₁₁	3.209(36)			0.131	3.182(50)			0.140	3.174(54)			0.159
C ₁ ··· H ₁₂	2.357(8)			0.144	2.357(6)			0.152	2.354(5)			0.172
N ₄ ··· H ₈	3.432(10)	0.094	} (40)	0.107	3.441(7)	0.094	} (41)	0.111	3.436(12)	0.125	} (59)	0.120
N ₄ ··· H ₉	2.709(11)	0.142		0.155	2.714(11)	0.149		0.166	2.714(10)	0.195		0.190
N ₄ ····· H ₁₁	3.415(58)			0.217	3.368(86)			0.243	3.336(88)			0.291
N ₄ ····· H ₁₂	2.482(42)			0.226	2.524(73)			0.254	2.524(78)			0.303
Anti												
N ··· N	3.775(10)	0.067	} (55)	0.069	3.785(11)	0.050	} (41)	0.075	3.784(10)	0.072	} (42)	0.088
C ₁ · H ₅	2.078(7)	0.098		0.100	2.082(7)	0.077		0.102	2.082(8)	0.091		0.107
N ₁₀ ··· H ₂	2.724(6)	0.150		0.152	2.728(8)	0.139		0.164	2.726(7)	0.172		0.188
C ₁ ··· H ₁₁	2.544(8)			0.108	2.551(9)			0.113	2.552(8)			0.125
C ₁ ··· H ₁₂	2.918(7)			0.145	2.921(6)			0.156	2.924(8)			0.179
N ₄ ····· H ₁₁	3.962(14)			0.115	3.974(16)			0.121	3.972(13)			0.134
N ₄ ····· H ₁₂	4.212(10)			0.143	4.221(10)			0.154	4.221(10)			0.177

^a In angstroms. ^b Quantities in parentheses are estimated 2σ. ^c See text for definition. ^d Quantities in braces were refined as a group with differences between group members taken from the calculated values. Corresponding amplitude groups in gauche (A) and (B) were combined to form one parameter. Unspecified amplitudes were assigned the calculated values.

groups. Nonetheless, the calculated components of the total dipole moment were not quite in agreement with the experimental ones. This plus the possibility that both gauche conformers are stabilized by intramolecular hydrogen bonding led us to treat the N-H bond moment slightly different from the N-H bond moment participating in the internal hydrogen bonding. We have added 0.7 D to the bond moment associated with N-H bond involved in hydrogen bonding. This value which incidentally seems to be the best value was obtained after several trial and errors in the least squares. We have also allowed for the CNH_b (H-bonded) angle to be somewhat different from the other CNH angles. In Table VIII we have listed these values. As a result, we were able to reach to a fairly close agreement with the experimentally determined dipole moment components.

One of the interesting features in EDA were the dihedral angles for the two conformers. Our results are in good agreement with the ab initio studies.⁶ The NCCN dihedral angles was calculated to be 57.8° and 61.9° in comparison with our values ($55.3^\circ - 56.4^\circ$) for conformer A and ($63.2^\circ - 63.6^\circ$) for conformer B. No distinction was made between the two CCN angles, within each conformer or among the conformers. Also, our NCCN dihedral angle for conformer B confirms the previous ED study² given for the presence of one gauche conformer. Our bond distances, $r_g(\text{C-N}) = 1.473(5) \text{ \AA}$, and $r_g(\text{C-C}) = 1.531(16) \text{ \AA}$ are in good agreement with the previous ED study² (1.469 ± 0.004), and (1.545 ± 0.008) $\text{\AA}$, respectively.

The values of the quality-of-fit (R) given in Table VIII are essentially constant for the highest and the lowest temperatures,

Table X. Correlation Matrix ($\times 100$) for Gauche (A) and Gauche (B) at 343 K^a

	σ_{LS}^b	$\langle CH, NH \rangle$	$\Delta(CH, NH)$	$\langle CC, CN \rangle$	$\Delta(CC, CN)$	$\angle CCN$	$\tau(NCCN)_B$	$\tau(C_1-N_4)_B$	$\tau(C_7-N_{10})_B$
σ_{LS}		0.10	0.76	0.10	0.70	22.0	31.0	381.0	202.6
$\langle CH, NH \rangle^c$	0.10	100	52	-56	-37	35	-11	-21	0
$\Delta(CH, NH)$	0.66	53	100	-62	-47	47	-16	-24	13
$\langle CC, CN \rangle$	0.07	-44	-56	100	87	-92	46	41	-61
$\Delta(CC, CN)$	0.54	-27	-39	82	100	-87	28	40	-52
$\angle CCN$	12.3	41	54	-86	-86	100	-66	-62	75
$\tau(NCCN)_A$	30.3	35	33	15	-10	-5	100	60	-46
$\tau(C_1-N_4)_A$	262.2	-49	-38	-24	-10	-3	-57	100	-59
$\tau(C_7-N_{10})_A$	209.2	-28	-22	-3	5	-23	-65	36	100

^a Right hand side of the diagonal is the correlation matrix for gauche (B) and the left hand side for gauche (A).

^b Standard deviations ($\times 100$) from least squares. ^c See text for definitions.

for some unknown reason(s) not clear to us, the fit is slightly poorer for the intermediate temperature. Table IX shows the interatomic and the corresponding amplitudes for some of the more important distances.

Hydrogen Bonding

Perhaps one of the most intriguing parts of this study was to investigate the existence of the internal hydrogen bonding. There are several pieces of experimental evidence, which we believe support the presence of the internal hydrogen bond. First, the greater stability of the gauche conformers vs the anti form, even at the highest temperature of the experiment (713 K), is the best indication of the strong attraction between the NH_2 groups. The $\angle \text{CNH}_\beta$ angle is less by 11.0° for conformer A, and by 6.4° for conformer B than its counterpart in methylamine¹¹, bringing the amine proton and nitrogen lone pair into closer proximity. Second, in both gauche conformers (A, B), the hydrogen atoms are pointed toward the nitrogen rather than away from the nitrogen atom, which suggests the possibility of internal hydrogen bonding. Third, the interatomic distance between the nitrogen atom and the hydrogen atom (apparently) involved in hydrogen bonding is 2.500(55) Å in one conformer and 2.491(42) Å in the other, is substantially less than the sum of their van der Waals radii (2.7 Å)¹⁴. However, there is another potentially competing effect, the so-called "gauche effect"⁷, that seems to be operating in 1,2-disubstituted compounds of ethane with

electronegative substituents, which in turn favors the gauche form.¹⁵ To what extent this effect contributes to the internal hydrogen bond (if any) is not clear to us, but it appears the influence should be minimal and the gauche effect can not be solely attributed to the stability of the gauche conformers. It is also interesting to note that the hydrogen bond formed is a very nonlinear one similar to 2-aminoethanol¹⁶ and ethylene glycol with $\angle \text{N-H}\cdots\text{N}$ angle equal to 102.7° for conformer A and 108.8° for conformer B. Because of the close similarity between the two conformers and the weak scattering power of the hydrogen atom, our attempt to determine the percentage of each gauche conformer separately in the mixture was not successful. However, a trial calculation based on molecular mechanics (MM2)¹⁷ without geometry optimization resulted 0.77 Kcal mole⁻¹ as the energy difference between the two gauche conformers, indicating 21% for conformer A and 79% for conformer B.

Energy and Entropy Differences of Conformers.

By plotting of the sample composition as a function of temperature, it is possible to determine the entropy differences between the anti and the gauche conformers. The usual formula

$$N_g/N_a = 2 \exp(-\Delta E^\circ/RT) \cdot \exp(\Delta S^\circ/R)$$

where N_g and N_a are the fractions of gauche and anti molecules in the sample, and the factor 2 is the ratio of their statistical

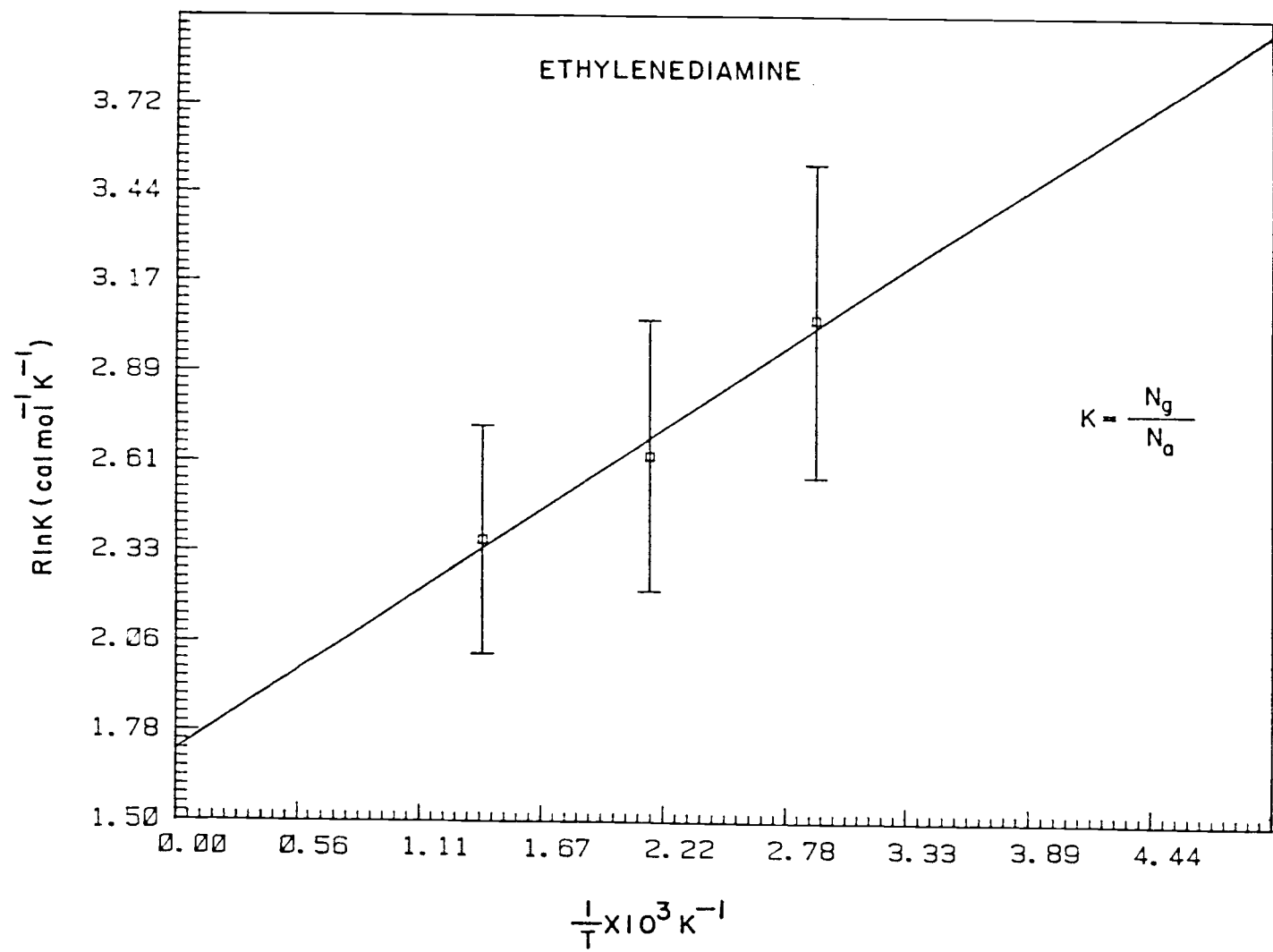


Figure 8. Van't Hoff plot of the anti-gauche compositions.

weights. Figure 8 shows the experimental points plotted in van't Hoff form. The results are: $\Delta E^\circ = E_g^\circ - E_a^\circ = -0.338(\sigma = 0.055)$ Kcal/mol and $\Delta S^\circ = S_g^\circ - S_a^\circ = 0.55(\sigma = 0.12)$ cal mol⁻¹K⁻¹. This energy difference can be attributed mainly to presence of intramolecular hydrogen bonding in the gauche conformers. Our value for ΔE° is lower than the value obtained from the LCAO - EHMO method³ which was calculated to be about 1 Kcal/mol. The larger energy difference obtained in the above work³ can be due to the fact that the inter - molecular interactions, namely, hydrogen bonding were not taken into account.

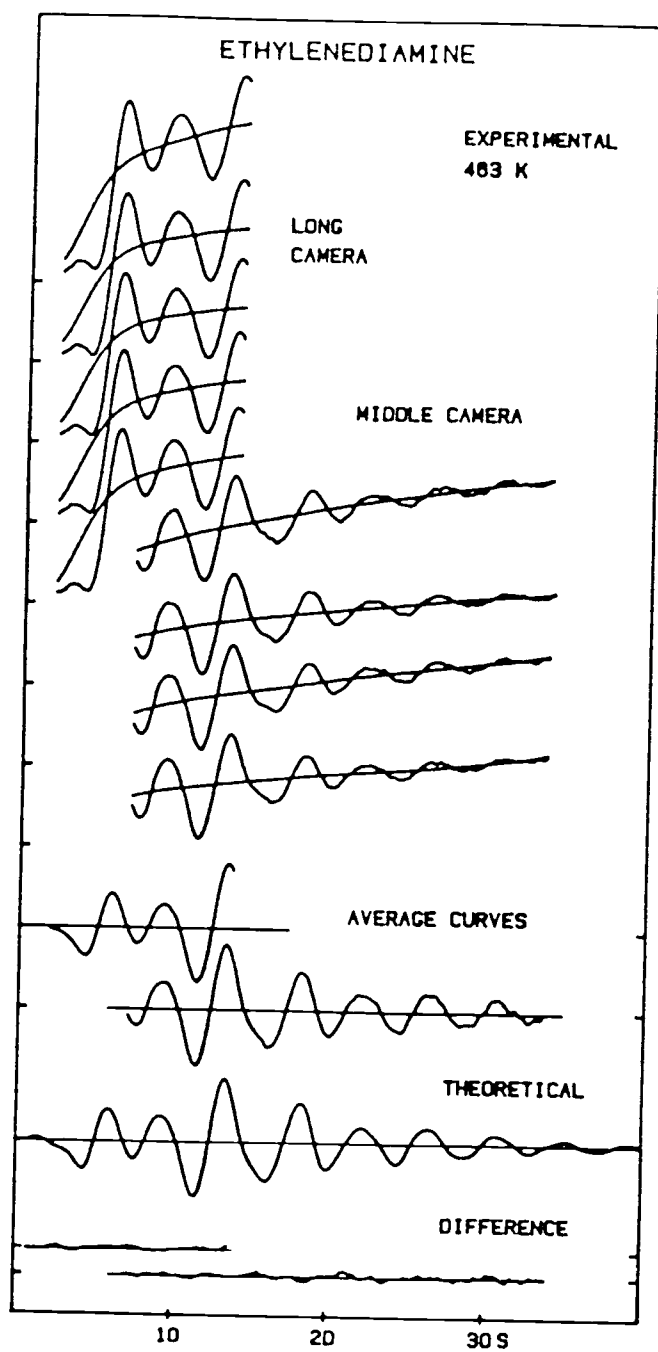


Figure 9. Intensity curves and backgrounds for 463 K experiments.

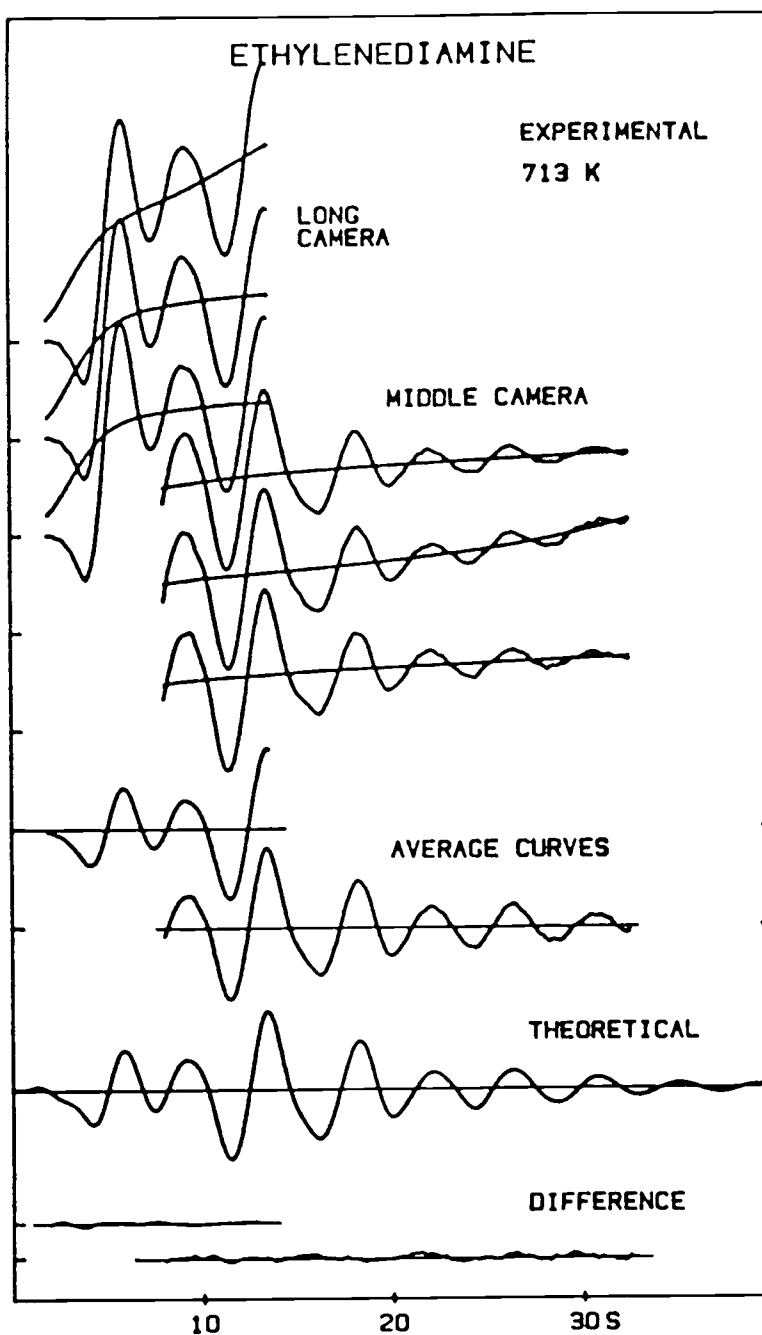


Figure 10. Intensity curves and backgrounds for 713 K experiments.

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Appendix.

Tables of Experimental Data

Table XI. Total Scattered Intensities From Each Plate.

4-83-1 Ethylene Glycol LC 376 K Reduced 10 SEP,85

S	.00	.25	.50	.75
2.	17.87	22.52	28.84	36.63
3.	43.94	51.05	57.32	63.10
4.	69.40	77.52	87.74	99.52
5.	112.11	124.18	134.14	141.02
6.	144.20	144.45	142.44	139.95
7.	137.78	136.77	137.16	139.17
8.	142.83	147.40	151.97	156.41
9.	160.16	162.63	163.72	164.15
10.	164.08	163.40	162.36	161.70
11.	160.40	159.50	158.72	158.78
12.	159.58	162.47	165.93	171.04
13.	176.83	181.99	185.85	188.06

4-79-2 Ethylene Glycol MC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
8.	275.18	283.90	294.09	301.76
9.	308.42	313.11	316.58	317.33
10.	316.57	315.23	314.15	311.68
11.	309.92	306.83	303.84	302.58
12.	302.49	305.38	310.63	318.02
13.	326.87	334.68	340.82	343.36
14.	343.13	340.34	335.87	331.58
15.	328.31	326.50	326.39	326.89
16.	326.29	327.34	327.30	327.87
17.	330.23	332.28	335.66	339.78
18.	343.92	347.40	349.53	349.84
19.	349.09	346.74	343.06	340.60
20.	337.99	336.91	337.55	339.00
21.	341.35	343.96	345.87	347.57
22.	348.52	349.95	350.49	351.59
23.	352.23	352.61	352.72	352.30
24.	352.17	351.54	350.30	350.00
25.	349.73	349.97	351.04	352.08
26.	354.16	355.99	357.11	358.46
27.	358.88	359.05	359.39	359.31
28.	358.33	358.86	358.97	358.53
29.	358.71	358.94	358.88	358.60
30.	359.14	359.50	360.75	361.62
31.	362.95	363.11	363.88	364.72
32.	364.85	365.00	365.15	365.47
33.	364.73	365.06	365.52	365.91

4-83-2 Ethylene Glycol LC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
2.	15.31	19.33	24.68	31.17
3.	37.48	43.46	48.84	53.84
4.	59.14	66.04	74.50	84.63
5.	95.44	105.88	114.42	120.35
6.	123.21	123.40	121.31	118.70
7.	116.55	115.26	115.65	117.02
8.	119.71	123.22	127.07	130.64
9.	133.35	135.61	136.39	136.60
10.	135.92	135.36	134.40	133.43
11.	132.31	130.88	129.66	128.79
12.	128.82	130.14	132.66	136.09
13.	139.88	143.61	146.31	147.64
14.	147.44			

4-79-3 Ethylene Glycol MC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
8.	129.80	133.53	138.20	141.79
9.	144.92	146.88	147.85	148.37
10.	146.67	145.37	144.53	143.43
11.	142.32	140.16	138.68	137.50
12.	137.49	138.27	140.47	144.29
13.	147.99	151.88	154.31	155.17
14.	154.77	152.83	150.61	148.57
15.	146.93	146.05	145.69	145.65
16.	145.62	145.71	145.86	145.65
17.	146.52	147.61	148.96	150.64
18.	152.20	153.79	154.86	154.74
19.	154.29	153.33	151.78	150.21
20.	149.05	148.69	149.05	149.66
21.	150.61	151.92	152.78	153.20
22.	153.69	154.38	154.47	154.84
23.	155.29	155.47	155.50	155.59
24.	155.28	155.12	154.49	154.25
25.	154.09	154.06	154.29	154.61
26.	155.72	156.68	157.44	157.75
27.	158.11	158.09	158.30	158.02
28.	157.47	157.53	157.38	157.15
29.	157.07	157.07	157.13	157.41
30.	157.31	157.50	157.80	158.20
31.	158.42	158.97	158.94	158.85
32.	159.36	159.31	159.03	158.99
33.	159.26	159.10	159.14	159.78

4-83-4 Ethylene Glycol LC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
2.	13.85	17.46	22.25	28.30
3.	34.05	39.49	44.48	48.98
4.	53.81	60.02	67.68	77.15
5.	87.14	96.89	104.77	110.07
6.	112.72	113.12	111.31	109.05
7.	106.96	105.82	105.89	107.22
8.	109.60	112.88	116.31	119.63
9.	122.23	123.82	124.89	125.07
10.	124.48	123.53	122.85	121.93
11.	120.66	119.51	118.38	117.46
12.	117.36	118.35	120.60	123.50
13.	127.20	130.67	133.30	134.28
14.	134.73			

4-79-4 Ethylene Glycol MC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
8.	185.57	192.09	198.08	203.84
9.	208.00	210.96	212.61	212.91
10.	210.98	210.34	208.52	206.93
11.	205.59	203.02	200.94	199.38
12.	199.64	201.40	204.95	209.41
13.	214.65	219.99	224.42	225.97
14.	225.34	222.81	219.76	216.91
15.	214.09	212.63	212.38	211.83
16.	212.18	211.72	211.99	212.48
17.	213.17	214.33	216.60	218.67
18.	221.04	223.46	224.45	224.65
19.	224.05	221.91	220.09	218.21
20.	216.40	216.24	216.34	217.17
21.	218.65	219.79	221.41	222.62
22.	223.28	223.74	224.13	224.70
24.	224.95	224.51	224.16	223.29
25.	223.57	223.49	223.94	224.11
26.	225.59	226.13	227.37	228.50
27.	228.36	228.72	228.84	228.26
28.	228.31	228.35	228.03	227.81
29.	227.92	228.30	228.01	227.99
30.	228.38	228.99	229.19	230.38
31.	230.40	230.71	231.81	231.29
32.	232.03	232.15	232.31	232.06
33.	231.72	231.80	232.12	232.68

4-83-5 Ethylene Glycol LC 376 K Reduced 10 Sep,85

S	.00	.25	.50	.75
2.	18.12	22.69	29.15	36.97
3.	44.59	51.93	58.30	64.27
4.	70.77	79.07	89.10	101.53
5.	114.50	127.40	137.84	144.92
6.	148.54	148.85	146.88	143.95
7.	141.11	139.46	139.67	141.75
8.	145.06	149.29	153.99	158.17
9.	161.85	164.29	165.49	165.43
10.	164.83	163.62	162.12	161.00
11.	159.43	157.64	155.91	155.04
12.	155.04	156.79	160.00	163.74
13.	168.98	173.34	176.97	178.39

4-72-2 Ethylene Glycol LC 733 K Reduced 12 Sep,85

S	.00	.25	.50	.75
2.	18.90	23.70	29.91	37.34
3.	44.65	51.75	58.76	65.12
4.	72.17	80.26	89.70	100.91
5.	113.10	124.89	135.07	142.07
6.	145.82	146.16	144.65	142.17
7.	140.49	139.65	140.57	143.03
8.	146.64	150.98	154.95	158.76
9.	161.40	163.50	164.46	164.83
10.	164.89	164.62	164.20	162.60
11.	160.88	158.92	157.49	157.25
12.	158.10	160.39	163.47	167.59
13.	171.26	174.27	176.57	177.78
14.	178.20			

4-81-3 Ethylene Glycol MC 733 K Reduced 29 Nov,85

S	.00	.25	.50	.75
7.	65.82	65.52	65.78	66.47
8.	68.11	70.04	72.16	73.70
9.	74.90	75.68	76.40	76.85
10.	76.96	77.05	76.91	76.73
11.	76.36	75.76	75.54	75.70
12.	76.24	77.37	78.89	80.61
13.	82.13	83.69	84.73	85.43
14.	85.47	85.24	84.81	84.42
15.	84.11	84.21	84.37	84.71
16.	85.15	85.38	85.79	86.27
17.	86.87	87.80	88.74	89.68
18.	90.58	91.27	91.83	92.20
19.	92.28	92.20	92.20	92.19
20.	92.20	92.52	93.00	93.70
21.	94.46	95.08	95.91	96.57
22.	97.32	97.87	98.28	98.70
23.	99.04	99.51	99.83	99.93
24.	100.17	100.46	100.52	100.91
25.	101.25	101.60	102.11	102.80
26.	103.50	104.07	104.44	105.02
27.	105.47	105.97	106.34	106.61
28.	107.05	107.34	107.48	107.78
29.	108.06	108.24	108.63	108.90
30.	109.24	109.68	110.16	110.75
31.	111.06	111.70	112.15	112.73
32.	113.09	113.60	113.98	114.33
33.	114.93			

4-72-3 Ethylene Glycol LC 733 K Reduced 12 Sep,85

S	.00	.25	.50	.75
2.	19.63	24.60	31.00	38.75
3.	46.19	53.64	60.90	67.48
4.	74.51	82.95	92.89	104.20
5.	116.62	128.77	139.21	146.45
6.	150.06	150.93	149.30	147.11
7.	145.31	144.52	145.59	148.20
8.	151.81	155.84	160.30	164.04
9.	166.82	168.50	169.95	170.40
10.	170.34	169.96	168.97	167.82
11.	166.29	164.49	162.97	162.36
12.	163.00	165.32	168.49	172.58
13.	176.48	179.72	181.96	183.17
14.	182.71			

4-81-4 Ethylene Glycol MC 733 K Reduced 29 Nov,85

S	.00	.25	.50	.75
7.	45.86	45.05	44.79	45.02
8.	45.83	47.03	48.36	49.30
9.	50.00	50.26	50.38	50.31
10.	49.99	49.52	49.05	48.37
11.	47.63	46.80	46.22	46.07
12.	46.32	47.09	48.17	49.39
13.	50.48	51.57	52.11	52.26
14.	51.98	51.41	50.90	50.27
15.	49.68	49.37	49.12	49.02
16.	48.85	48.71	48.68	48.76
17.	48.96	49.30	49.68	50.16
18.	50.56	50.75	50.89	50.77
19.	50.55	50.22	49.80	49.47
20.	49.22	49.11	49.03	49.14
21.	49.29	49.65	49.99	50.22
22.	50.53	50.81	50.90	50.95
23.	50.94	50.91	50.96	50.88
24.	50.68	50.58	50.40	50.22
25.	50.15	50.25	50.35	50.35
26.	50.51	50.73	50.97	51.03
27.	51.15	51.19	51.11	51.20
28.	51.18	51.15	51.08	50.84
29.	50.74	50.81	50.87	50.83
30.	50.83	51.07	51.19	51.40
31.	51.58	51.60	51.80	51.87
32.	51.95	51.94	52.05	52.16
33.	52.12			

4-72-4 Ethylene Glycol LC 733 K Reduced 12 Sep,85

S	.00	.25	.50	.75
2.	18.76	23.36	29.46	36.75
3.	43.91	51.15	58.16	64.62
4.	71.23	79.22	88.32	99.27
5.	111.40	122.96	132.78	139.75
6.	143.49	144.26	142.70	140.22
7.	138.32	137.55	138.42	140.73
8.	144.18	148.10	152.29	155.86
9.	158.48	160.65	161.68	162.05
10.	161.88	161.76	160.88	160.00
11.	158.21	156.51	155.09	154.36
12.	155.43	157.53	160.46	163.90
13.	167.71	170.94	173.30	174.19
14.	174.13			

4-81-5 Ethylene Glycol MC 733 K Reduced 29 Nov,85

S	.00	.25	.50	.75
7.	39.41	39.06	39.11	39.49
8.	40.41	41.62	42.70	43.74
9.	44.23	44.63	44.88	45.03
10.	44.81	44.58	44.29	43.87
11.	43.37	42.76	42.34	42.32
12.	42.55	43.23	44.17	45.33
13.	46.25	47.09	47.76	48.05
14.	48.03	47.67	47.30	46.95
15.	46.68	46.55	46.50	46.48
16.	46.50	46.52	46.72	46.99
17.	47.27	47.85	48.33	48.87
18.	49.20	49.48	49.64	49.49
19.	49.35	49.02	48.55	48.36
20.	48.18	48.19	48.28	48.45
21.	48.68	48.95	49.19	49.33
22.	49.55	49.75	49.98	50.09
23.	50.44	50.58	50.69	50.79
24.	50.85	50.92	51.03	51.26
25.	51.49	51.72	51.90	52.11
26.	52.34	52.72	53.01	53.18
27.	53.38	53.59	53.83	53.96
28.	54.11	54.27	54.43	54.62
29.	54.78	54.95	55.17	55.23
30.	55.50	55.85	56.11	56.48
31.	56.77	57.20	57.60	57.96
32.	58.39	58.90	59.41	59.90
33.	60.51			

4-72-5 Ethylene Glycol LC 733 K Reduced 12 Sep,85

S	.00	.25	.50	.75
2.	16.87	21.07	26.61	33.11
3.	39.52	45.95	52.07	57.87
4.	63.96	71.13	79.19	89.40
5.	100.34	110.69	119.78	126.14
6.	129.49	129.85	128.37	126.02
7.	124.18	123.48	124.51	126.66
8.	129.77	133.38	137.05	140.38
9.	142.85	144.74	145.73	146.38
10.	146.37	145.87	145.21	143.84
11.	142.49	140.94	139.89	139.55
12.	140.17	141.88	144.58	147.60
13.	150.87	153.58	155.96	156.71
14.	156.84			

Table XII. Calculated Background For Each Plate.

4-83-1 Ethylene Glycol LC 376 K Background 19 May, 87

S	.00	.25	.50	.75
2.	20.89	26.76	33.75	41.54
3.	49.84	58.35	66.86	75.19
4.	83.20	90.79	97.89	104.47
5.	110.51	116.01	120.99	125.48
6.	129.52	133.14	136.38	139.26
7.	141.83	144.13	146.19	148.04
8.	149.70	151.21	152.59	153.86
9.	155.04	156.15	157.21	158.23
10.	159.22	160.21	161.21	162.22
11.	163.27	164.35	165.50	166.72
12.	168.01	169.41	170.91	172.53
13.	174.30	176.21	178.30	180.56

4-79-2 Ethylene Glycol MC 376 K Background 19 May, 87

S	.00	.25	.50	.75
8.	289.25	291.99	294.58	297.01
9.	299.31	301.48	303.53	305.46
10.	307.29	309.03	310.67	312.24
11.	313.72	315.14	316.48	317.77
12.	319.00	320.17	321.30	322.38
13.	323.42	324.42	325.38	326.31
14.	327.20	328.07	328.91	329.73
15.	330.52	331.29	332.03	332.76
16.	333.47	334.17	334.85	335.51
17.	336.16	336.80	337.43	338.04
18.	338.65	339.24	339.83	340.41
19.	340.98	341.54	342.10	342.65
20.	343.19	343.73	344.26	344.79
21.	345.31	345.83	346.34	346.85
22.	347.35	347.85	348.35	348.84
23.	349.33	349.81	350.29	350.76
24.	351.23	351.70	352.16	352.62
25.	353.07	353.52	353.96	354.39
26.	354.82	355.25	355.67	356.08
27.	356.48	356.88	357.27	357.66
28.	358.03	358.40	358.77	359.13
29.	359.48	359.82	360.17	360.50
30.	360.84	361.17	361.50	361.83
31.	362.16	362.50	362.84	363.19
32.	363.56	363.94	364.33	364.75
33.	365.20	365.68	366.20	366.76

4-83-2 Ethylene Glycol LC 376 K Background 19 May,87

S	.00	.25	.50	.75
2.	18.15	22.95	28.80	35.42
3.	42.51	49.82	57.13	64.29
4.	71.16	77.65	83.70	89.28
5.	94.37	98.97	103.10	106.80
6.	110.09	113.01	115.59	117.87
7.	119.87	121.63	123.19	124.57
8.	125.79	126.87	127.84	128.71
9.	129.50	130.22	130.88	131.49
10.	132.07	132.62	133.14	133.65
11.	134.15	134.64	135.12	135.62
12.	136.12	136.63	137.15	137.70
13.	138.27	138.86	139.49	140.16
14.	140.87			

4-79-3 Ethylene Glycol MC 376 K Background 19 May,87

S	.00	.25	.50	.75
8.	136.57	137.65	138.61	139.46
9.	140.21	140.88	141.48	142.02
10.	142.51	142.95	143.36	143.73
11.	144.08	144.40	144.71	145.00
12.	145.27	145.53	145.78	146.02
13.	146.26	146.49	146.71	146.92
14.	147.14	147.34	147.55	147.74
15.	147.94	148.13	148.32	148.51
16.	148.69	148.87	149.05	149.23
17.	149.41	149.59	149.76	149.94
18.	150.11	150.29	150.46	150.64
19.	150.82	151.00	151.19	151.37
20.	151.56	151.75	151.94	152.13
21.	152.33	152.53	152.73	152.93
22.	153.14	153.35	153.55	153.76
23.	153.97	154.18	154.39	154.59
24.	154.80	155.00	155.19	155.39
25.	155.58	155.76	155.94	156.11
26.	156.28	156.43	156.59	156.73
27.	156.86	156.99	157.11	157.22
28.	157.32	157.42	157.50	157.59
29.	157.66	157.74	157.80	157.87
30.	157.94	158.00	158.07	158.14
31.	158.22	158.31	158.40	158.50
32.	158.62	158.75	158.90	159.06
33.	159.24	159.44	159.65	159.89

4-83-4 Ethylene Glycol LC 376 K Background 19 May,87

S	.00	.25	.50	.75
2.	16.45	20.81	26.13	32.15
3.	38.62	45.30	51.99	58.54
4.	64.84	70.80	76.37	81.51
5.	86.21	90.46	94.29	97.72
6.	100.78	103.49	105.89	108.01
7.	109.87	111.50	112.94	114.21
8.	115.32	116.30	117.17	117.94
9.	118.63	119.24	119.80	120.31
10.	120.78	121.21	121.63	122.02
11.	122.40	122.78	123.16	123.55
12.	123.95	124.37	124.82	125.29
13.	125.81	126.36	126.97	127.64
14.	128.37			

4-79-4 Ethylene Glycol MC 376 K Background 19 May,87

S	.00	.25	.50	.75
8.	195.90	197.38	198.76	200.03
9.	201.22	202.34	203.38	204.35
10.	205.26	206.12	206.92	207.68
11.	208.39	209.06	209.69	210.28
12.	210.84	211.36	211.85	212.32
13.	212.75	213.16	213.54	213.91
14.	214.25	214.57	214.87	215.16
15.	215.43	215.69	215.94	216.18
16.	216.41	216.63	216.85	217.06
17.	217.27	217.48	217.69	217.90
18.	218.11	218.32	218.54	218.76
19.	218.99	219.22	219.46	219.70
20.	219.95	220.20	220.46	220.73
21.	220.99	221.27	221.55	221.83
22.	222.11	222.40	222.68	222.97
23.	223.25	223.54	223.82	224.10
24.	224.37	224.64	224.90	225.15
25.	225.40	225.64	225.87	226.10
26.	226.31	226.52	226.72	226.92
27.	227.10	227.28	227.46	227.63
28.	227.80	227.97	228.14	228.31
29.	228.48	228.66	228.84	229.03
30.	229.23	229.44	229.67	229.90
31.	230.14	230.39	230.66	230.93
32.	231.20	231.47	231.74	232.00
33.	232.24	232.44	232.60	232.71

4-83-5 Ethylene Glycol LC 376 K Background 19 May, 87

S		.00	.25	.50	.75
2.		21.35	27.24	34.33	42.28
3.		50.79	59.54	68.31	76.90
4.		85.17	93.00	100.33	107.11
5.		113.33	118.98	124.08	128.66
6.		132.77	136.43	139.67	142.54
7.		145.06	147.29	149.25	150.98
8.		152.49	153.82	154.99	156.02
9.		156.93	157.75	158.47	159.13
10.		159.72	160.28	160.80	161.30
11.		161.79	162.29	162.80	163.33
12.		163.90	164.51	165.19	165.93
13.		166.76	167.69	168.73	169.89

4-73-2 Ethylene Glycol LC 733 K Background 19 May,87

S	.00	.25	.50	.75
2.	21.96	27.38	34.11	41.79
3.	50.09	58.69	67.35	75.87
4.	84.09	91.90	99.21	106.00
5.	112.23	117.91	123.05	127.68
6.	131.84	135.57	138.89	141.84
7.	144.46	146.79	148.87	150.72
8.	152.37	153.84	155.15	156.33
9.	157.39	158.36	159.24	160.04
10.	160.78	161.47	162.12	162.74
11.	163.34	163.92	164.50	165.08
12.	165.67	166.28	166.92	167.59
13.	168.31	169.10	169.95	170.88
14.	171.90			

4-81-3 Ethylene Glycol MC 733 K Background 19 May,87

S	.00	.25	.50	.75
7.	67.69	68.42	69.13	69.82
8.	70.49	71.14	71.78	72.40
9.	73.01	73.60	74.18	74.75
10.	75.30	75.84	76.36	76.88
11.	77.38	77.88	78.36	78.84
12.	79.31	79.77	80.23	80.68
13.	81.12	81.56	82.00	82.43
14.	82.86	83.28	83.70	84.13
15.	84.55	84.97	85.39	85.81
16.	86.23	86.65	87.07	87.49
17.	87.92	88.34	88.77	89.20
18.	89.63	90.06	90.49	90.93
19.	91.37	91.81	92.25	92.69
20.	93.14	93.58	94.03	94.48
21.	94.92	95.37	95.82	96.27
22.	96.72	97.16	97.61	98.05
23.	98.50	98.94	99.38	99.81
24.	100.24	100.67	101.10	101.52
25.	101.94	102.36	102.77	103.18
26.	103.58	103.98	104.37	104.76
27.	105.15	105.53	105.91	106.28
28.	106.65	107.02	107.39	107.76
29.	108.12	108.49	108.86	109.23
30.	109.60	109.98	110.36	110.75
31.	111.15	111.56	111.98	112.42
32.	112.87	113.34	113.83	114.35
33.	114.89			

4-73-3 Ethylene Glycol LC 733 K Background 19 May,87

S	.00	.25	.50	.75
2.	22.75	28.32	35.24	43.15
3.	51.70	60.57	69.49	78.28
4.	86.77	94.83	102.39	109.41
5.	115.86	121.74	127.06	131.86
6.	136.18	140.05	143.50	146.57
7.	149.29	151.72	153.88	155.80
8.	157.50	159.03	160.38	161.60
9.	162.69	163.68	164.57	165.39
10.	166.13	166.82	167.46	168.07
11.	168.65	169.21	169.76	170.32
12.	170.88	171.46	172.07	172.71
13.	173.41	174.16	174.99	175.90
14.	176.91			

4-81-4 Ethylene Glycol MC 733 K Background 19 May,87

S	.00	.25	.50	.75
7.	47.54	47.72	47.85	47.94
8.	48.01	48.07	48.11	48.15
9.	48.19	48.24	48.29	48.34
10.	48.41	48.48	48.55	48.64
11.	48.73	48.82	48.92	49.01
12.	49.11	49.20	49.30	49.38
13.	49.47	49.54	49.62	49.68
14.	49.74	49.78	49.82	49.86
15.	49.88	49.90	49.91	49.91
16.	49.91	49.90	49.89	49.88
17.	49.86	49.85	49.83	49.81
18.	49.80	49.79	49.78	49.77
19.	49.77	49.77	49.78	49.79
20.	49.81	49.83	49.86	49.90
21.	49.94	49.98	50.03	50.08
22.	50.14	50.20	50.26	50.32
23.	50.38	50.44	50.50	50.55
24.	50.61	50.66	50.70	50.74
25.	50.78	50.81	50.83	50.85
26.	50.87	50.88	50.88	50.88
27.	50.88	50.88	50.87	50.87
28.	50.87	50.87	50.88	50.89
29.	50.91	50.94	50.98	51.03
30.	51.10	51.17	51.26	51.35
31.	51.46	51.57	51.68	51.78
32.	51.88	51.95	52.00	52.00
33.	51.95			

4-73-4 Ethylene Glycol LC 733 K Background 19 May, 87

S	.00	.25	.50	.75
2.	21.71	26.98	33.57	41.13
3.	49.31	57.81	66.36	74.78
4.	82.90	90.61	97.83	104.51
5.	110.65	116.23	121.27	125.80
6.	129.88	133.51	136.75	139.62
7.	142.17	144.43	146.45	148.24
8.	149.83	151.25	152.52	153.67
9.	154.70	155.64	156.49	157.28
10.	158.00	158.67	159.31	159.91
11.	160.49	161.05	161.59	162.15
12.	162.70	163.26	163.84	164.44
13.	165.09	165.77	166.51	167.31
14.	168.18			

4-81-5 Ethylene Glycol MC 733 K Background 19 May, 87

S	.00	.25	.50	.75
7.	40.54	41.10	41.56	41.93
8.	42.22	42.47	42.67	42.84
9.	42.99	43.12	43.25	43.37
10.	43.50	43.63	43.76	43.90
11.	44.05	44.21	44.38	44.55
12.	44.73	44.92	45.11	45.30
13.	45.49	45.69	45.88	46.07
14.	46.26	46.44	46.62	46.79
15.	46.95	47.11	47.25	47.39
16.	47.52	47.64	47.75	47.86
17.	47.96	48.04	48.13	48.21
18.	48.28	48.35	48.41	48.48
19.	48.54	48.60	48.67	48.73
20.	48.80	48.87	48.95	49.04
21.	49.12	49.22	49.32	49.43
22.	49.55	49.68	49.82	49.96
23.	50.11	50.27	50.44	50.62
24.	50.80	50.99	51.18	51.38
25.	51.58	51.79	52.00	52.21
26.	52.42	52.63	52.84	53.05
27.	53.26	53.47	53.67	53.88
28.	54.08	54.28	54.48	54.67
29.	54.87	55.07	55.28	55.49
30.	55.71	55.94	56.19	56.46
31.	56.75	57.07	57.42	57.81
32.	58.24	58.73	59.27	59.88
33.	60.57			

4-73-5 Ethylene Glycol LC 733 K Background 19 May, 87

S	.00	.25	.50	.75
2.	19.64	24.39	30.32	37.11
3.	44.46	52.09	59.78	67.33
4.	74.62	81.54	88.02	94.03
5.	99.54	104.55	109.08	113.16
6.	116.83	120.11	123.03	125.62
7.	127.93	129.98	131.81	133.44
8.	134.90	136.20	137.37	138.42
9.	139.38	140.25	141.04	141.77
10.	142.44	143.07	143.65	144.21
11.	144.74	145.24	145.73	146.21
12.	146.69	147.17	147.65	148.15
13.	148.67	149.21	149.78	150.39
14.	151.05			

Table XIII. Average Molecular Intensities.
Ethylene Glycol 376 K.

1.Ave.	4-83-1	4-83-2	4-83-4	4-83-5	17 Sep, 85
S	.00	.25	.50	.75	
2.	-2.88	-4.34	-5.55	-6.18	
3.	-7.98	-10.81	-15.19	-20.59	
4.	-25.18	-25.90	-21.73	-11.41	
5.	2.92	19.29	32.90	40.74	
6.	40.92	33.63	19.51	3.31	
7.	-12.22	-24.22	-30.51	-31.04	
8.	-25.65	-15.63	-3.41	8.93	
9.	19.27	26.02	28.09	26.45	
10.	21.45	14.54	6.46	-1.45	
11.	-11.83	-22.73	-33.61	-42.00	
12.	-45.90	-40.96	-29.06	-11.18	
13.	12.23	33.30	48.04	51.03	

2.Ave.	4-79-2	4-79-3	4-79-4	17 Sep, 85
S	.00	.25	.50	.75
8.	-26.43	-15.37	-1.51	10.14
9.	19.81	25.94	28.88	27.99
10.	20.06	13.61	6.60	-1.88
11.	-9.71	-22.30	-33.35	-41.92
12.	-44.95	-41.60	-30.36	-11.89
13.	9.76	31.82	48.61	54.42
14.	51.33	38.87	22.68	6.91
15.	-7.16	-15.82	-19.29	-21.84
16.	-24.10	-25.54	-26.80	-27.86
17.	-23.28	-17.42	-6.72	5.86
18.	19.08	31.96	38.78	38.00
19.	32.94	20.52	4.64	-10.02
20.	-23.83	-29.31	-29.32	-25.56
21.	-17.57	-8.59	-0.93	4.05
22.	6.75	10.54	10.60	13.04
23.	14.66	15.18	13.47	10.25
24.	5.13	-0.13	-8.21	-14.60
25.	-17.38	-19.73	-17.85	-16.44
26.	-5.74	1.33	8.45	13.95
27.	14.10	13.63	13.91	8.97
28.	2.88	2.70	-0.45	-4.93
29.	-6.24	-6.25	-8.81	-9.94
30.	-9.69	-7.74	-4.66	1.46
31.	3.76	5.88	9.28	6.68
32.	9.85	7.90	4.78	1.66
33.	-2.98	-5.72	-6.25	-2.85

Table XIII cont. Ethylene Glycol 733 K.

1.Ave.	4-72-2	4-72-3	4-72-4	4-72-5	2 Dec,85
S	.00	.25	.50	.75	
2.	-3.04	-4.12	-5.19	-6.05	
3.	-8.16	-11.07	-14.80	-19.71	
4.	-23.75	-24.59	-21.48	-12.23	
5.	2.03	18.02	32.58	41.05	
6.	41.48	33.51	18.98	1.61	
7.	-13.93	-25.67	-30.56	-29.26	
8.	-22.67	-12.66	-0.92	10.06	
9.	17.67	23.00	24.84	23.91	
10.	20.63	15.95	9.01	-0.93	
11.	-13.13	-26.66	-38.35	-45.31	
12.	-44.73	-36.02	-21.52	-2.06	
13.	17.93	34.22	45.80	48.05	
14.	42.70				

2.Ave.	4-81-3	4-81-4	4-81-5	2 Dec,85
S	.00	.25	.50	.75
7.	-14.87	-25.18	-30.43	-30.91
8.	-23.45	-11.27	2.37	13.37
9.	20.18	23.72	25.85	25.70
10.	20.99	15.13	7.70	-2.12
11.	-14.01	-28.68	-39.76	-44.63
12.	-44.14	-34.70	-19.44	-0.08
13.	16.52	33.51	43.79	47.34
14.	42.35	31.13	18.80	5.72
15.	-6.19	-12.85	-18.24	-21.17
16.	-23.52	-26.95	-26.76	-24.55
17.	-20.31	-9.78	0.69	12.75
18.	22.17	28.13	31.74	28.39
19.	22.04	11.38	-1.44	-10.88
20.	-19.70	-22.82	-23.73	-20.17
21.	-15.14	-8.82	-1.25	2.61
22.	8.92	13.10	14.52	13.90
23.	14.90	14.07	12.30	6.89
24.	0.80	-3.76	-10.95	-13.36
25.	-15.14	-14.75	-13.58	-11.07
26.	-6.15	0.54	4.31	6.40
27.	8.49	10.30	9.97	8.92
28.	8.76	6.92	2.73	-1.28
29.	-4.53	-6.66	-5.99	-10.83
30.	-11.24	-6.48	-4.70	1.08
31.	1.06	4.40	7.13	7.91
32.	6.30	6.13	5.22	2.26
33.	1.91			

Table XIV. Total Scattered Intensities From Each Plate.

3-121-1 Ethylenediamine LC 343 K Reduced 18 Jun,85

S	.00	.25	.50	.75
2.	20.21	25.12	31.91	39.55
3.	46.86	53.80	60.30	66.00
4.	72.35	80.52	90.22	101.75
5.	114.27	125.32	134.28	139.29
6.	141.30	140.36	137.96	135.15
7.	132.89	131.63	131.87	133.63
8.	136.48	140.00	143.53	146.05
9.	147.44	148.20	147.92	146.55
10.	144.64	141.83	139.08	136.12
11.	133.92	132.09	131.83	132.90
12.	135.46	139.66	144.65	149.83
13.	153.98			

3-114-6 Ethylenediamine MC 343 K Reduced 21 Jun,85

S	.00	.25	.50	.75
7.			174.79	176.99
8.	181.46	186.94	191.72	194.95
9.	196.87	198.18	198.23	195.99
10.	193.88	191.68	188.52	184.61
11.	181.07	178.94	178.19	179.70
12.	182.85	187.72	194.15	200.33
13.	205.72	208.99	209.64	207.38
14.	203.97	199.78	195.83	193.37
15.	192.20	191.57	190.98	190.20
16.	189.37	188.71	189.43	191.01
17.	193.01	196.00	198.72	201.80
18.	203.48	204.39	204.21	202.37
19.	200.22	197.07	194.92	193.90
20.	193.77	194.25	195.81	197.17
21.	198.53	199.63	200.25	201.19
22.	201.38	201.61	201.49	201.29
23.	200.55	200.19	199.47	198.74
24.	197.83	197.29	197.08	197.24
25.	197.83	198.95	200.24	201.21
26.	202.11	202.34	202.08	201.70
27.	201.31	200.46	199.89	199.44
28.	199.02	198.72	199.04	199.16
29.	199.22	199.91	200.53	201.20
30.	201.84	202.61	202.97	203.25
31.	203.46	203.28	203.10	202.86
32.	202.44	202.29	202.42	

3-121-3 Ethylenediamine LC 343 K Reduced 18 Jun,85

S	.00	.25	.50	.75
2.	21.35	26.73	33.63	41.71
3.	49.34	56.56	63.29	69.56
4.	76.06	84.52	94.88	107.01
5.	119.88	131.86	141.13	146.48
6.	148.71	148.22	145.80	143.30
7.	141.12	140.00	140.33	142.39
8.	145.46	149.52	153.05	156.00
9.	157.85	158.48	158.23	157.12
10.	155.20	152.96	150.13	147.47
11.	145.07	143.43	143.23	144.48
12.	147.66	152.18	157.62	163.01
13.	167.39			

3-114-8 Ethylenediamine MC 343 K Reduced 21 Jun,85

S	.00	.25	.50	.75
7.			198.12	203.19
8.	210.29	218.11	223.98	228.43
9.	231.51	233.09	233.27	231.51
10.	228.89	227.03	223.36	219.21
11.	215.71	213.12	212.68	214.63
12.	218.79	224.99	232.48	239.83
13.	246.19	250.44	251.08	248.05
14.	244.44	239.78	235.79	233.20
15.	231.54	231.03	230.71	229.93
16.	229.55	229.24	229.92	231.74
17.	234.03	238.14	241.69	244.66
18.	247.11	248.05	247.46	245.82
19.	243.19	239.94	237.97	236.66
20.	236.51	237.10	239.03	240.84
21.	242.62	244.30	245.20	245.83
22.	246.29	246.51	246.61	246.36
23.	246.29	245.44	244.92	243.86
24.	243.66	243.17	243.35	243.81
25.	245.06	246.33	247.70	248.92
26.	249.82	250.41	250.22	249.54
27.	248.70	248.48	247.46	246.60
28.	246.24	245.99	246.13	246.29
29.	246.25	247.23	247.53	248.04
30.	248.49	249.09	249.73	249.67
31.	249.76	249.66	249.35	248.77
32.	248.45	248.44	248.26	

3-121-4 Ethylenediamine LC 343 K Reduced 18 Jun,85

S	.00	.25	.50	.75
2.	16.40	20.46	25.96	32.26
3.	38.28	43.93	49.32	54.09
4.	59.35	66.17	74.48	84.29
5.	94.84	104.37	111.86	116.23
6.	117.81	117.02	114.94	112.33
7.	110.26	109.25	109.42	110.96
8.	113.45	116.44	119.46	121.73
9.	123.07	123.34	122.95	121.96
10.	120.30	118.09	115.67	113.24
11.	111.47	109.82	109.51	110.45
12.	112.65	116.41	120.51	124.57
13.	128.15			

3-114-9 Ethylenediamine MC 343 K Reduced 21 Jun,85

S	.00	.25	.50	.75
7.			191.25	196.24
8.	202.04	207.61	212.76	216.47
9.	219.08	220.12	220.17	218.49
10.	216.02	213.75	210.77	206.33
11.	202.90	200.56	200.12	201.44
12.	205.35	211.29	217.97	225.29
13.	231.27	234.82	235.44	232.92
14.	229.37	225.18	221.08	218.24
15.	216.99	216.04	215.50	215.03
16.	214.63	214.03	214.68	216.16
17.	219.13	222.40	225.73	228.48
18.	230.79	231.52	230.90	229.40
19.	226.54	223.59	221.58	220.39
20.	220.27	221.19	222.71	224.28
21.	225.57	227.10	228.04	228.80
22.	229.14	229.01	228.98	228.71
23.	228.13	227.72	226.80	226.20
24.	225.55	225.27	225.51	226.13
25.	226.57	227.64	228.61	229.48
26.	230.11	230.57	230.23	230.01
27.	229.21	228.49	227.81	227.41
28.	227.15	227.11	227.20	227.38
29.	227.36	227.90	228.43	228.92
30.	229.47	230.12	230.53	230.58
31.	230.87	230.77	230.51	230.25
32.	230.07	229.58	229.44	

4-60-1 II Ethylenediamine LC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
2.	9.27	11.68	14.54	17.83
3.	20.93	23.97	26.77	29.34
4.	32.01	35.22	39.10	43.79
5.	48.78	53.44	57.15	59.41
6.	60.36	60.20	59.44	58.73
7.	58.12	58.08	58.47	59.46
8.	60.91	62.47	63.87	65.22
9.	66.03	66.66	66.98	66.93
10.	66.75	66.32	65.73	65.16
11.	64.66	64.38	64.68	65.78
12.	67.04	68.95	71.09	73.12
13.	74.84	76.07	76.39	76.25

4-59-1 Ethylenediamine MC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
7.	64.71	64.49	64.91	65.77
8.	67.36	69.44	71.36	72.62
9.	73.57	74.29	74.82	74.51
10.	73.99	73.43	72.72	71.88
11.	71.15	70.82	71.00	71.81
12.	73.35	75.60	77.92	80.17
13.	82.07	83.40	83.71	83.19
14.	82.38	81.40	80.67	80.40
15.	80.20	80.39	80.23	80.50
16.	80.39	81.00	81.59	82.63
17.	83.62	84.99	85.99	87.08
18.	88.01	88.47	88.49	88.28
19.	88.04	87.63	87.49	87.50
20.	87.49	88.14	88.68	89.41
21.	90.38	91.01	91.43	91.86
22.	92.16	92.42	92.55	92.86
23.	93.02	92.98	92.97	92.95
24.	93.14	93.33	93.43	93.92
25.	94.51	94.95	95.44	96.04
26.	96.58	96.62	97.10	97.40
27.	97.43	97.64	97.71	97.59
28.	97.70	97.99	98.25	98.66
29.	98.85	99.06	99.77	100.03
30.	100.57	101.00	101.28	101.45
31.	101.66	101.41	101.56	101.94
32.	101.79	102.03	102.13	102.39
33.	102.66	103.20	103.60	

4-60-2 Ethylenediamine LC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
2.	10.78	13.50	16.88	20.88
3.	24.59	28.22	31.64	34.60
4.	37.89	41.89	46.62	52.47
5.	58.64	64.26	68.73	71.57
6.	72.74	72.68	71.44	70.10
7.	69.18	68.54	68.75	69.89
8.	71.31	73.07	74.99	76.17
9.	77.17	77.64	77.69	77.49
10.	77.19	76.28	75.12	74.00
11.	72.85	72.32	72.43	73.41
12.	75.01	77.40	79.78	82.33
13.	84.33	85.58	86.04	85.58

4-59-3 Ethylenediamine MC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
7.	107.23	106.53	106.74	107.96
8.	110.41	113.95	116.69	118.47
9.	119.77	120.12	120.44	120.25
10.	119.22	117.90	116.49	114.67
11.	113.00	112.15	112.41	113.87
12.	116.57	119.92	123.63	127.48
13.	130.25	131.91	132.30	131.46
14.	130.15	128.11	126.56	125.50
15.	124.81	124.63	124.70	124.65
16.	124.33	124.82	125.85	126.93
17.	128.64	130.50	132.38	134.20
18.	135.22	135.49	135.59	135.00
19.	133.96	132.86	132.21	131.87
20.	132.20	132.75	133.60	134.53
21.	135.79	136.52	137.27	137.98
22.	138.31	138.55	138.92	139.01
23.	138.78	138.71	138.61	138.29
24.	138.46	138.79	138.90	139.67
25.	140.55	141.65	142.44	143.04
26.	143.61	144.14	144.37	144.53
27.	144.56	144.41	144.36	144.39
28.	144.59	144.43	145.11	145.08
29.	145.43	145.82	146.38	146.67
30.	147.43	147.97	148.48	148.50
31.	148.71	148.73	148.79	149.19
32.	148.91	148.98	149.03	149.30
33.	149.71	149.84	150.58	151.48

4-60-3 Ethylenediamine LC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
2.	9.11	11.50	14.41	17.80
3.	21.03	24.15	27.10	29.82
4.	32.59	36.09	40.25	45.33
5.	50.80	55.82	59.85	62.30
6.	63.22	63.08	62.21	61.15
7.	60.44	60.09	60.32	61.25
8.	62.53	64.22	65.72	66.98
9.	67.83	68.31	68.20	67.98
10.	67.60	66.82	65.76	64.75
11.	63.85	63.31	63.44	64.18
12.	65.51	67.53	69.43	71.57
13.	73.38	74.46	74.94	74.63

4-59-4 Ethylenediamine MC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
7.	78.65	78.33	78.70	79.70
8.	81.61	83.92	86.40	87.70
9.	88.67	89.13	89.48	89.29
10.	88.50	87.71	86.70	85.32
11.	84.11	83.59	83.57	84.83
12.	86.71	89.28	91.84	94.60
13.	96.47	98.00	98.33	97.82
14.	96.76	95.41	94.25	93.70
15.	93.27	93.24	93.35	93.45
16.	93.43	93.62	94.21	95.19
17.	96.44	97.70	99.18	100.32
18.	101.29	101.67	101.82	101.40
19.	100.83	100.40	99.98	99.93
20.	100.42	100.97	101.62	102.35
21.	103.26	104.09	104.60	105.26
22.	105.35	105.60	105.84	105.88
23.	106.28	105.94	105.79	105.67
24.	105.92	106.02	106.31	106.58
25.	107.43	107.97	108.54	109.31
26.	109.76	110.10	110.30	110.38
27.	110.38	110.58	110.43	110.49
28.	110.67	110.67	110.75	111.29
29.	111.63	111.93	112.44	112.91
30.	113.12	113.86	114.14	114.58
31.	114.48	114.73	114.99	115.24
32.	115.43	115.83	115.86	116.19
33.	116.94	117.30	117.77	

4-60-4 Ethylenediamine LC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
2.	9.71	12.28	15.31	18.85
3.	22.20	25.54	28.61	31.48
4.	34.35	37.96	42.37	47.53
5.	53.07	58.39	62.51	65.11
6.	66.17	66.05	65.18	64.24
7.	63.68	63.43	63.64	64.75
8.	66.15	67.80	69.56	70.89
9.	71.58	72.10	72.30	72.03
10.	71.75	71.11	70.22	69.08
11.	68.26	67.81	67.86	68.72
12.	70.10	72.31	74.49	76.83
13.	78.71	79.90	80.24	79.80

4-59-5 Ethylenediamine MC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
7.	80.96	80.36	80.50	81.33
8.	82.91	85.46	87.52	89.00
9.	89.85	90.41	90.44	90.10
10.	89.19	87.72	86.63	84.74
11.	83.41	82.58	82.81	83.99
12.	85.82	88.32	91.05	93.70
13.	95.86	97.22	97.45	96.74
14.	95.31	93.89	92.74	92.15
15.	91.98	91.68	91.50	91.45
16.	91.14	91.57	91.90	92.71
17.	93.94	95.45	96.72	97.86
18.	98.58	98.78	98.72	98.03
19.	97.24	96.70	95.97	96.02
20.	96.11	96.61	97.42	98.07
21.	98.59	99.24	99.63	100.04
22.	100.14	100.38	100.36	100.28
23.	100.29	99.97	99.69	99.85
24.	99.72	99.89	100.13	100.47
25.	101.18	101.66	102.31	102.84
26.	103.30	103.44	103.41	103.90
27.	103.76	103.88	103.61	103.75
28.	103.74	103.91	104.42	104.53
29.	104.67	105.10	105.54	106.13
30.	106.48	107.08	107.25	107.69
31.	107.88	108.06	108.10	108.47
32.	108.88	109.14	109.35	109.94
33.	110.51	110.75	111.53	

4-60-5 Ethylenediamine LC 463 K Reduced 28 Jul,85

S	.00	.25	.50	.75
2.	10.41	13.08	16.37	20.16
3.	23.78	27.31	30.42	33.50
4.	36.62	40.45	44.91	50.35
5.	56.16	61.63	66.00	68.74
6.	69.97	69.84	69.16	68.32
7.	67.62	67.49	67.98	69.14
8.	70.70	72.43	74.31	75.71
9.	76.68	77.32	77.50	77.42
10.	77.08	76.22	75.11	74.12
11.	73.19	72.79	72.88	73.92
12.	75.39	77.54	79.98	82.33
13.	84.35	85.71	86.15	86.08

3-115-7 Ethylenediamine LC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
2.	11.24	14.03	17.48	21.52
3.	25.33	29.44	33.11	36.62
4.	40.09	44.72	50.08	56.31
5.	63.08	69.62	74.64	77.94
6.	79.49	79.36	78.32	77.49
7.	76.88	76.95	77.88	79.63
8.	81.90	84.59	87.31	89.38
9.	91.02	92.28	93.12	93.88
10.	94.40	94.53	94.47	94.06
11.	94.15	94.78	96.13	98.61
12.	101.87	105.81	110.05	114.36
13.	117.70	120.29	121.80	

3-115-2 Ethylenediamine MC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
8.	177.24	181.64	185.84	188.82
9.	190.68	191.83	192.13	190.97
10.	189.34	187.45	184.51	180.82
11.	177.89	176.32	176.94	179.72
12.	184.04	189.31	195.26	200.69
13.	204.77	207.82	208.19	206.57
14.	203.91	201.22	198.35	196.47
15.	195.62	194.52	193.98	193.67
16.	193.65	193.59	194.88	196.82
17.	199.21	202.18	204.83	207.03
18.	208.71	209.13	208.60	207.58
19.	206.21	204.62	203.43	202.82
20.	202.92	203.83	205.01	205.91
21.	207.44	208.25	209.41	209.91
22.	210.82	210.75	210.65	210.63
23.	210.30	209.92	209.33	209.27
24.	209.17	209.48	209.64	209.91
25.	210.90	211.96	212.92	213.83
26.	214.68	215.19	215.55	215.31
27.	215.24	214.89	214.83	214.31
28.	214.65	214.61	215.03	215.16
29.	215.64	216.36	216.83	217.35
30.	217.93	218.66	218.96	219.31
31.	219.51	219.72	219.94	219.80
32.	219.87	220.05	220.59	

3-115-8 Ethylenediamine LC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
2.	15.20	18.91	23.48	28.92
3.	34.15	39.64	44.50	49.09
4.	53.71	59.68	66.51	75.04
5.	83.83	92.07	98.52	102.55
6.	104.16	103.78	102.16	100.44
7.	98.86	98.07	98.66	100.19
8.	102.56	105.18	107.48	109.38
9.	110.59	110.91	110.84	110.52
10.	109.68	108.28	106.34	104.43
11.	102.78	101.86	102.14	103.69
12.	106.44	109.74	113.15	116.48
13.	119.01	120.70	121.02	

3-115-3 Ethylenediamine MC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
8.	151.41	155.77	158.89	161.15
9.	162.81	163.35	163.46	162.08
10.	161.13	159.64	157.00	153.94
11.	151.15	149.39	149.36	151.50
12.	155.16	159.81	164.69	169.36
13.	173.11	175.23	175.56	174.01
14.	171.81	169.53	167.07	165.42
15.	164.72	164.37	163.53	163.40
16.	163.50	163.88	164.81	166.70
17.	168.89	171.44	174.02	175.79
18.	177.36	178.39	178.00	177.65
19.	176.47	175.43	174.56	174.13
20.	174.38	175.09	176.15	177.40
21.	178.63	180.17	181.04	181.96
22.	182.58	183.37	183.63	183.95
23.	183.83	184.08	184.22	184.16
24.	184.44	184.99	185.76	186.92
25.	188.03	189.14	190.42	191.69
26.	193.05	193.89	194.74	195.14
27.	195.60	196.14	196.59	197.15
28.	197.67	198.40	199.42	200.31
29.	201.41	203.01	204.60	206.06
30.	207.34	208.49	209.96	210.79
31.	212.61	213.67	214.68	215.74
32.	216.78	217.87	219.64	

3-115-9 Ethylenediamine LC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
2.	12.07	15.06	18.69	23.06
3.	27.23	31.53	35.29	38.93
4.	42.45	47.38	52.94	59.72
5.	66.96	73.73	78.75	82.04
6.	83.24	82.83	81.16	79.48
7.	78.14	77.62	78.01	79.20
8.	80.98	82.97	85.00	86.33
9.	87.14	87.25	87.41	87.15
10.	86.40	85.01	83.34	81.81
11.	80.23	79.48	79.60	80.77
12.	82.99	85.68	88.60	91.40
13.	93.44	94.65	94.77	

3-115-4 Ethylenediamine MC 713 K Reduced 21 Jun,85

S	.00	.25	.50	.75
8.	107.17	110.02	112.34	114.15
9.	115.17	115.66	116.09	115.11
10.	114.01	112.77	110.72	108.13
11.	105.95	104.85	105.06	106.54
12.	109.09	112.23	115.74	119.07
13.	121.65	123.26	123.38	122.10
14.	120.45	118.48	116.90	115.79
15.	115.05	114.77	114.53	114.28
16.	113.97	114.01	114.93	116.04
17.	117.49	118.89	120.65	121.80
18.	122.81	122.95	122.89	122.29
19.	120.95	119.76	118.97	118.43
20.	118.67	119.11	119.67	120.34
21.	121.19	121.83	122.56	122.87
22.	123.30	123.41	123.16	123.16
23.	122.76	122.43	122.27	121.98
24.	121.90	121.93	121.96	122.63
25.	123.21	123.71	124.12	124.60
26.	125.06	125.40	125.64	125.54
27.	125.53	125.45	124.92	124.87
28.	124.88	124.65	125.11	125.18
29.	125.51	125.71	126.29	126.41
30.	126.64	127.03	127.36	127.46
31.	127.44	127.36	127.60	127.38
32.	127.52	127.54	127.52	

Table XV. Calculated Background For Each Plate.

3-121-1 Ethylenediamine LC 343 K Background 19 May, 87

S	.00	.25	.50	.75
2.	22.86	28.92	36.57	45.24
3.	54.45	63.77	72.87	81.52
4.	89.56	96.90	103.47	109.28
5.	114.35	118.71	122.42	125.55
6.	128.21	130.45	132.32	133.88
7.	135.18	136.28	137.21	138.01
8.	138.70	139.31	139.84	140.33
9.	140.77	141.17	141.54	141.88
10.	142.19	142.49	142.77	143.02
11.	143.25	143.45	143.63	143.78
12.	143.91	144.01	144.10	144.17
13.	144.23			

3-114-6 Ethylenediamine MC 344 K Background 19 May, 87

S	.00	.25	.50	.75
7.			181.35	183.11
8.	184.61	185.90	187.00	187.94
9.	188.75	189.44	190.04	190.56
10.	191.01	191.41	191.76	192.08
11.	192.37	192.63	192.88	193.10
12.	193.32	193.53	193.72	193.91
13.	194.10	194.28	194.45	194.62
14.	194.79	194.95	195.12	195.27
15.	195.43	195.58	195.73	195.88
16.	196.03	196.17	196.31	196.45
17.	196.59	196.73	196.87	197.00
18.	197.14	197.28	197.41	197.55
19.	197.68	197.82	197.95	198.09
20.	198.22	198.35	198.48	198.60
21.	198.73	198.85	198.96	199.07
22.	199.18	199.28	199.37	199.46
23.	199.54	199.61	199.67	199.73
24.	199.78	199.82	199.85	199.87
25.	199.89	199.90	199.91	199.92
26.	199.92	199.93	199.93	199.95
27.	199.96	199.99	200.02	200.07
28.	200.13	200.21	200.31	200.43
29.	200.56	200.72	200.90	201.11
30.	201.32	201.56	201.80	202.04
31.	202.28	202.51	202.70	202.85
32.	202.93	202.93	202.81	

3-121-4 Ethylenediamine LC 343 K Background 19 May,87

S	.00	.25	.50	.75
2.	18.77	23.62	29.88	37.06
3.	44.74	52.54	60.17	67.43
4.	74.18	80.35	85.87	90.74
5.	94.98	98.63	101.72	104.33
6.	106.55	108.41	109.96	111.25
7.	112.34	113.25	114.04	114.71
8.	115.29	115.81	116.28	116.70
9.	117.10	117.46	117.79	118.10
10.	118.38	118.65	118.91	119.13
11.	119.33	119.50	119.65	119.76
12.	119.84	119.89	119.93	119.92
13.	119.90			

3-114-9 Ethylenediamine MC 343 K Background 19 May,87

S	.00	.25	.50	.75
7.			199.68	202.20
8.	204.33	206.14	207.66	208.96
9.	210.06	211.00	211.82	212.53
10.	213.16	213.72	214.23	214.70
11.	215.14	215.56	215.96	216.34
12.	216.72	217.09	217.45	217.81
13.	218.16	218.50	218.84	219.17
14.	219.50	219.81	220.12	220.42
15.	220.71	220.99	221.26	221.53
16.	221.78	222.02	222.26	222.49
17.	222.71	222.93	223.14	223.34
18.	223.54	223.74	223.94	224.13
19.	224.32	224.52	224.71	224.90
20.	225.09	225.29	225.48	225.67
21.	225.86	226.05	226.23	226.42
22.	226.60	226.77	226.94	227.10
23.	227.26	227.40	227.54	227.66
24.	227.77	227.88	227.96	228.04
25.	228.10	228.15	228.19	228.22
26.	228.24	228.25	228.25	228.25
27.	228.24	228.24	228.24	228.25
28.	228.26	228.29	228.33	228.39
29.	228.47	228.57	228.69	228.84
30.	229.00	229.18	229.37	229.57
31.	229.77	229.96	230.11	230.22
32.	230.26	230.20	230.01	

3-121-3 Ethylenediamine LC 343 K Background 19 May, 87

S	.00	.25	.50	.75
2.	24.16	30.50	38.47	47.51
3.	57.12	66.84	76.35	85.41
4.	93.86	101.59	108.55	114.74
5.	120.16	124.87	128.90	132.35
6.	135.32	137.84	139.99	141.82
7.	143.38	144.73	145.90	146.93
8.	147.84	148.67	149.41	150.10
9.	150.75	151.34	151.90	152.43
10.	152.93	153.41	153.88	154.31
11.	154.73	155.13	155.51	155.87
12.	156.21	156.54	156.88	157.20
13.	157.53			

3-114-8 Ethylenediamine MC 343 K Background 19 May, 87

S	.00	.25	.50	.75
7.			206.43	210.24
8.	213.44	216.13	218.38	220.26
9.	221.85	223.18	224.31	225.28
10.	226.12	226.86	227.51	228.11
11.	228.66	229.18	229.67	230.15
12.	230.61	231.07	231.52	231.97
13.	232.42	232.86	233.30	233.73
14.	234.15	234.57	234.98	235.37
15.	235.76	236.14	236.50	236.85
16.	237.19	237.51	237.83	238.13
17.	238.43	238.71	238.99	239.25
18.	239.52	239.78	240.03	240.29
19.	240.54	240.80	241.06	241.32
20.	241.58	241.85	242.12	242.39
21.	242.67	242.95	243.24	243.52
22.	243.81	244.10	244.39	244.67
23.	244.95	245.22	245.48	245.74
24.	245.98	246.21	246.43	246.63
25.	246.81	246.98	247.12	247.25
26.	247.36	247.45	247.53	247.59
27.	247.63	247.66	247.68	247.70
28.	247.71	247.72	247.73	247.74
29.	247.77	247.80	247.86	247.92
30.	248.01	248.11	248.23	248.36
31.	248.51	248.65	248.78	248.89
32.	248.97	248.97	248.89	

4-60-1 Ethylenediamine LC 463 K Background 19 May,87

S	.00	.25	.50	.75
2.	10.44	13.05	16.29	19.92
3.	23.77	27.65	31.44	35.05
4.	38.41	41.50	44.29	46.77
5.	48.97	50.88	52.55	54.00
6.	55.27	56.38	57.36	58.23
7.	59.01	59.72	60.38	61.00
8.	61.59	62.16	62.72	63.26
9.	63.81	64.35	64.88	65.41
10.	65.94	66.46	66.99	67.50
11.	68.00	68.50	68.98	69.44
12.	69.88	70.30	70.70	71.07
13.	71.43	71.75	72.04	72.30

4-59-1 Ethylenediamine MC 463 K Background 19 May,87

S	.00	.25	.50	.75
7.	65.81	66.45	67.10	67.75
8.	68.40	69.05	69.68	70.29
9.	70.89	71.46	72.02	72.56
10.	73.07	73.57	74.05	74.52
11.	74.96	75.40	75.82	76.23
12.	76.64	77.03	77.42	77.81
13.	78.19	78.57	78.95	79.33
14.	79.71	80.10	80.48	80.87
15.	81.26	81.65	82.04	82.43
16.	82.83	83.23	83.63	84.02
17.	84.42	84.82	85.21	85.61
18.	86.00	86.38	86.76	87.14
19.	87.51	87.88	88.24	88.60
20.	88.94	89.29	89.62	89.95
21.	90.27	90.58	90.89	91.20
22.	91.49	91.78	92.07	92.36
23.	92.63	92.91	93.19	93.46
24.	93.73	94.00	94.27	94.54
25.	94.82	95.09	95.36	95.64
26.	95.92	96.20	96.48	96.76
27.	97.05	97.33	97.62	97.90
28.	98.19	98.47	98.75	99.02
29.	99.29	99.55	99.81	100.06
30.	100.30	100.54	100.76	100.98
31.	101.19	101.39	101.59	101.79
32.	102.00	102.20	102.43	102.67
33.	102.94	103.24	103.60	

4-60-2 Ethylenediamine LC 463 K Background 19 May,87

S	.00	.25	.50	.75
2.	12.26	15.32	19.19	23.59
3.	28.28	33.02	37.66	42.07
4.	46.18	49.93	53.30	56.28
5.	58.89	61.15	63.09	64.74
6.	66.16	67.38	68.41	69.31
7.	70.08	70.76	71.37	71.92
8.	72.43	72.91	73.36	73.80
9.	74.23	74.64	75.05	75.45
10.	75.84	76.24	76.63	77.00
11.	77.38	77.73	78.08	78.41
12.	78.73	79.03	79.31	79.58
13.	79.83	80.07	80.29	80.49

4-59-3 Ethylenediamine MC 463 K Background 19 May,87

S	.00	.25	.50	.75
7.	109.10	109.90	110.68	111.45
8.	112.20	112.93	113.64	114.33
9.	115.01	115.66	116.29	116.91
10.	117.50	118.08	118.64	119.19
11.	119.72	120.24	120.74	121.24
12.	121.72	122.19	122.66	123.11
13.	123.56	124.00	124.43	124.86
14.	125.28	125.70	126.11	126.52
15.	126.93	127.33	127.73	128.12
16.	128.51	128.90	129.28	129.67
17.	130.04	130.42	130.79	131.16
18.	131.53	131.90	132.26	132.62
19.	132.98	133.33	133.68	134.03
20.	134.38	134.73	135.07	135.42
21.	135.76	136.10	136.44	136.77
22.	137.11	137.45	137.79	138.12
23.	138.46	138.80	139.13	139.47
24.	139.81	140.15	140.49	140.82
25.	141.16	141.50	141.83	142.17
26.	142.50	142.84	143.17	143.49
27.	143.82	144.14	144.45	144.76
28.	145.07	145.36	145.65	145.94
29.	146.21	146.48	146.74	146.99
30.	147.23	147.46	147.69	147.91
31.	148.13	148.34	148.55	148.77
32.	148.99	149.23	149.48	149.76
33.	150.06	150.41	150.80	151.26

4-60-3 Ethylenediamine LC 463 K Background 19 May,87

S	.00	.25	.50	.75
2.	10.42	13.06	16.39	20.19
3.	24.23	28.33	32.35	36.18
4.	39.76	43.05	46.01	48.66
5.	50.99	53.02	54.78	56.29
6.	57.60	58.74	59.72	60.57
7.	61.31	61.96	62.54	63.07
8.	63.55	64.00	64.41	64.81
9.	65.18	65.54	65.88	66.21
10.	66.52	66.83	67.14	67.42
11.	67.70	67.96	68.22	68.46
12.	68.69	68.91	69.13	69.33
13.	69.53	69.73	69.93	70.13

4-59-4 Ethylenediamine MC 463 K Background 19 May,87

S	.00	.25	.50	.75
7.	79.99	80.75	81.47	82.18
8.	82.85	83.50	84.11	84.71
9.	85.27	85.82	86.33	86.83
10.	87.30	87.76	88.19	88.61
11.	89.02	89.41	89.79	90.16
12.	90.52	90.88	91.23	91.57
13.	91.92	92.26	92.59	92.93
14.	93.27	93.61	93.95	94.29
15.	94.64	94.99	95.33	95.69
16.	96.04	96.40	96.76	97.12
17.	97.48	97.84	98.21	98.57
18.	98.93	99.30	99.66	100.03
19.	100.39	100.75	101.10	101.46
20.	101.81	102.16	102.50	102.84
21.	103.17	103.50	103.83	104.15
22.	104.47	104.78	105.08	105.38
23.	105.68	105.97	106.26	106.54
24.	106.82	107.09	107.37	107.63
25.	107.90	108.16	108.42	108.68
26.	108.94	109.19	109.45	109.70
27.	109.95	110.21	110.46	110.72
28.	110.97	111.23	111.49	111.75
29.	112.02	112.29	112.56	112.83
30.	113.11	113.39	113.68	113.97
31.	114.27	114.57	114.89	115.21
32.	115.54	115.89	116.25	116.62
33.	117.02	117.44	117.88	

4-60-4 Ethylenediamine LC 463 K Background 19 May,87

S	.00	.25	.50	.75
2.	11.03	13.84	17.35	21.31
3.	25.52	29.78	33.94	37.92
4.	41.65	45.07	48.16	50.93
5.	53.38	55.52	57.39	59.00
6.	60.42	61.65	62.72	63.66
7.	64.50	65.24	65.91	66.52
8.	67.08	67.61	68.11	68.58
9.	69.04	69.48	69.90	70.31
10.	70.70	71.09	71.47	71.83
11.	72.19	72.53	72.86	73.18
12.	73.49	73.79	74.08	74.36
13.	74.63	74.91	75.18	75.45

4-59-5 Ethylenediamine MC 463 K Background 19 May,87

S	.00	.25	.50	.75
7.	82.18	82.86	83.47	84.03
8.	84.55	85.02	85.46	85.86
9.	86.24	86.59	86.93	87.25
10.	87.56	87.86	88.15	88.43
11.	88.71	88.99	89.26	89.53
12.	89.81	90.08	90.35	90.62
13.	90.89	91.16	91.43	91.70
14.	91.97	92.24	92.51	92.78
15.	93.05	93.31	93.58	93.84
16.	94.09	94.35	94.60	94.85
17.	95.09	95.33	95.57	95.80
18.	96.03	96.26	96.48	96.69
19.	96.91	97.12	97.32	97.52
20.	97.72	97.92	98.11	98.30
21.	98.49	98.68	98.87	99.05
22.	99.24	99.42	99.61	99.80
23.	99.98	100.17	100.36	100.55
24.	100.75	100.94	101.14	101.34
25.	101.55	101.75	101.96	102.18
26.	102.39	102.61	102.83	103.06
27.	103.29	103.52	103.76	104.00
28.	104.24	104.49	104.74	104.99
29.	105.25	105.51	105.78	106.05
30.	106.32	106.61	106.90	107.20
31.	107.51	107.83	108.16	108.51
32.	108.88	109.27	109.68	110.12
33.	110.59	111.10	111.65	

4-60-5 Ethylenediamine LC 463 K Background 19 May, 87

S	.00	.25	.50	.75
2.	11.65	14.80	18.59	22.79
3.	27.20	31.65	35.99	40.14
4.	44.02	47.60	50.86	53.79
5.	56.40	58.70	60.72	62.49
6.	64.06	65.44	66.65	67.72
7.	68.68	69.53	70.31	71.02
8.	71.67	72.29	72.86	73.40
9.	73.91	74.40	74.87	75.32
10.	75.75	76.18	76.59	76.99
11.	77.38	77.75	78.12	78.48
12.	78.83	79.18	79.53	79.88
13.	80.23	80.59	80.96	81.34

3-115-7 Ethylenediamine LC 713 K Background 19 May,87

S	.00	.25	.50	.75
2.	12.78	15.94	20.02	24.73
3.	29.78	34.92	39.96	44.78
4.	49.29	53.43	57.18	60.54
5.	63.52	66.14	68.44	70.46
6.	72.28	73.90	75.38	76.74
7.	78.02	79.24	80.43	81.61
8.	82.79	83.98	85.20	86.44
9.	87.73	89.05	90.41	91.81
10.	93.24	94.71	96.23	97.77
11.	99.34	100.93	102.55	104.17
12.	105.81	107.46	109.12	110.78
13.	112.44	114.09	115.74	

3-115-2 Ethylenediamine MC 713 K Background 19 May,87

S	.00	.25	.50	.75
8.	179.64	180.51	181.39	182.28
9.	183.15	184.02	184.87	185.71
10.	186.52	187.30	188.06	188.80
11.	189.51	190.19	190.85	191.48
12.	192.09	192.69	193.26	193.81
13.	194.35	194.87	195.37	195.87
14.	196.35	196.82	197.29	197.75
15.	198.20	198.64	199.08	199.51
16.	199.94	200.36	200.78	201.19
17.	201.60	202.01	202.41	202.81
18.	203.20	203.58	203.96	204.34
19.	204.71	205.07	205.43	205.78
20.	206.13	206.46	206.80	207.12
21.	207.44	207.75	208.06	208.36
22.	208.66	208.95	209.24	209.53
23.	209.81	210.08	210.36	210.63
24.	210.90	211.18	211.45	211.72
25.	211.99	212.26	212.54	212.82
26.	213.09	213.37	213.66	213.94
27.	214.23	214.51	214.80	215.09
28.	215.38	215.67	215.97	216.26
29.	216.55	216.83	217.12	217.41
30.	217.69	217.98	218.26	218.55
31.	218.84	219.14	219.45	219.78
32.	220.12	220.49	220.89	

3-115-8 Ethylenediamine LC 713 K Background 19 May, 87

S	.00	.25	.50	.75
2.	17.19	21.47	26.98	33.28
3.	40.01	46.85	53.54	59.91
4.	65.85	71.28	76.16	80.48
5.	84.26	87.53	90.33	92.72
6.	94.77	96.51	98.00	99.28
7.	100.38	101.34	102.19	102.95
8.	103.64	104.29	104.90	105.47
9.	106.03	106.56	107.08	107.57
10.	108.06	108.53	108.99	109.43
11.	109.86	110.26	110.63	110.98
12.	111.30	111.60	111.87	112.11
13.	112.33	112.52	112.69	

3-115-3 Ethylenediamine MC 713 K Background 19 May, 87

S	.00	.25	.50	.75
8.	153.57	154.35	155.08	155.77
9.	156.42	157.04	157.62	158.17
10.	158.70	159.20	159.68	160.14
11.	160.59	161.02	161.43	161.84
12.	162.25	162.65	163.04	163.44
13.	163.84	164.23	164.63	165.04
14.	165.45	165.87	166.29	166.72
15.	167.16	167.60	168.05	168.50
16.	168.97	169.44	169.91	170.40
17.	170.88	171.37	171.87	172.37
18.	172.87	173.38	173.89	174.40
19.	174.92	175.44	175.96	176.48
20.	177.00	177.53	178.05	178.59
21.	179.12	179.66	180.20	180.74
22.	181.29	181.85	182.42	182.99
23.	183.57	184.16	184.76	185.37
24.	185.99	186.63	187.29	187.96
25.	188.64	189.34	190.07	190.81
26.	191.57	192.35	193.16	193.99
27.	194.84	195.71	196.61	197.53
28.	198.47	199.44	200.44	201.46
29.	202.50	203.57	204.66	205.78
30.	206.92	208.09	209.28	210.49
31.	211.74	213.01	214.31	215.65
32.	217.01	218.42	219.86	

3-115-9 Ethylenediamine LC 713 K Background 19 May,87

S	.00	.25	.50	.75
2.	13.75	17.19	21.61	26.67
3.	32.07	37.55	42.90	47.99
4.	52.72	57.03	60.89	64.30
5.	67.27	69.82	71.98	73.82
6.	75.38	76.69	77.80	78.74
7.	79.54	80.22	80.83	81.36
8.	81.84	82.29	82.71	83.11
9.	83.49	83.86	84.22	84.57
10.	84.91	85.24	85.57	85.88
11.	86.17	86.45	86.71	86.94
12.	87.15	87.34	87.51	87.64
13.	87.75	87.83	87.89	

3-115-4 Ethylenediamine MC 713 K Background 19 May,87

S	.00	.25	.50	.75
8.	108.48	109.19	109.79	110.31
9.	110.77	111.17	111.53	111.85
10.	112.14	112.41	112.67	112.91
11.	113.15	113.38	113.61	113.84
12.	114.07	114.30	114.54	114.77
13.	115.01	115.25	115.49	115.73
14.	115.98	116.22	116.46	116.70
15.	116.94	117.17	117.40	117.63
16.	117.85	118.07	118.28	118.49
17.	118.69	118.89	119.08	119.27
18.	119.45	119.63	119.80	119.96
19.	120.13	120.28	120.44	120.59
20.	120.74	120.89	121.03	121.18
21.	121.32	121.46	121.61	121.75
22.	121.89	122.03	122.17	122.31
23.	122.46	122.60	122.75	122.89
24.	123.04	123.19	123.34	123.48
25.	123.63	123.78	123.93	124.08
26.	124.23	124.38	124.53	124.68
27.	124.82	124.97	125.11	125.26
28.	125.40	125.55	125.69	125.83
29.	125.97	126.11	126.25	126.39
30.	126.52	126.66	126.80	126.93
31.	127.07	127.20	127.33	127.45
32.	127.57	127.68	127.79	

Table XVI. Average Molecular Intensities.
Ethylenediamine 343 K

1.Ave. 3-121-1 3-121-3 3-121-4 21 Jun,85

S	.00	.25	.50	.75
2.	-2.12	-3.27	-4.53	-6.05
3.	-8.85	-12.68	-17.26	-22.67
4.	-26.96	-27.36	-23.31	-13.93
5.	-0.34	13.73	25.44	30.92
6.	30.62	24.42	14.44	3.48
7.	-6.30	-13.13	-15.74	-13.28
8.	-6.95	2.41	12.10	19.70
9.	24.06	25.05	22.90	17.40
10.	8.98	-2.32	-15.16	-28.54
11.	-39.90	-49.78	-53.18	-50.14
12.	-39.25	-20.16	3.17	27.73
13.	48.96			

2.Ave. 3-114-6 3-114-8 3-114-9 21 Jun,85

S	.00	.25	.50	.75
7.			-17.87	-15.19
8.	-7.08	3.76	13.42	20.34
9.	24.71	26.33	24.86	17.61
10.	8.76	0.51	-11.65	-27.23
11.	-41.15	-51.58	-55.99	-52.77
12.	-41.42	-22.25	2.39	28.39
13.	51.28	65.97	68.56	57.56
14.	42.14	22.53	3.70	-9.47
15.	-17.12	-21.92	-25.85	-30.79
16.	-35.18	-39.55	-37.83	-31.16
17.	-20.11	-3.27	12.77	27.92
18.	39.03	43.33	40.23	30.02
19.	14.42	-5.00	-18.55	-27.06
20.	-29.42	-26.85	-17.91	-9.34
21.	-1.19	6.87	11.08	15.17
22.	16.30	15.97	14.74	12.05
23.	7.56	2.71	-3.55	-10.42
24.	-15.97	-20.29	-20.85	-18.72
25.	-13.78	-5.54	3.42	11.12
26.	17.38	20.68	18.50	15.00
27.	9.45	4.22	-2.21	-7.23
28.	-10.55	-12.72	-11.67	-11.01
29.	-11.84	-6.25	-2.93	0.90
30.	4.51	9.25	11.94	11.12
31.	11.20	8.30	4.38	-0.27
32.	-3.96	-5.99	-5.27	

Table XVI cont. Ethylenediamine 463 K.

1.Ave. 4-60-1 4-60-2 4-60-3 4-60-4 4-60-5 15 Jul,85

S	.00	.25	.50	.75
2.	-2.34	-3.24	-4.61	-6.05
3.	-8.85	-12.38	-16.85	-21.94
4.	-26.23	-27.10	-23.69	-14.45
5.	-1.06	13.45	25.40	31.58
6.	31.15	24.92	14.61	3.67
7.	-5.73	-12.44	-15.25	-12.56
8.	-6.76	1.57	10.64	17.48
9.	21.16	22.60	20.59	15.98
10.	9.98	-0.13	-12.93	-26.29
11.	-39.10	-48.15	-51.48	-46.21
12.	-35.65	-16.44	4.52	27.31
13.	46.49	58.66	61.37	55.79

2.Ave. 4-59-1 4-59-3 4-59-4 4-59-5 15 Jul,85

S	.00	.25	.50	.75
7.	-7.03	-13.48	-16.10	-14.96
8.	-8.29	3.28	13.98	19.99
9.	23.55	24.35	23.69	18.80
10.	9.88	-0.96	-12.42	-27.61
11.	-41.38	-50.74	-53.92	-48.43
12.	-35.57	-15.79	6.26	29.57
13.	47.37	59.19	59.88	50.89
14.	36.30	18.45	3.02	-7.16
15.	-15.05	-19.97	-25.00	-28.49
16.	-35.29	-34.83	-32.10	-24.73
17.	-13.52	0.82	14.05	26.39
18.	34.47	34.98	31.49	20.83
19.	7.72	-5.16	-16.56	-22.38
20.	-24.28	-20.55	-14.85	-8.01
21.	1.36	8.03	11.43	15.70
22.	14.55	14.17	12.51	9.74
23.	7.04	-0.92	-7.95	-13.78
24.	-16.58	-18.16	-19.81	-16.62
25.	-7.89	-1.64	4.79	11.89
26.	16.53	15.98	15.44	15.56
27.	9.79	6.77	-0.69	-5.97
28.	-9.31	-12.90	-11.34	-10.93
29.	-11.43	-10.32	-3.69	-0.58
30.	3.64	10.86	11.80	12.37
31.	9.37	4.09	1.08	2.86
32.	-2.55	-3.61	-8.71	-8.00
33.	-4.76	-5.93	-2.52	

Table XVI cont. Ethylenediamine 713 K.

1.Ave. 3-115-7 3-115-8 3-115-9 23 JUN,85

S	.00	.25	.50	.75
2.	-2.17	-3.09	-4.65	-6.39
3.	-9.45	-12.64	-17.10	-21.86
4.	-26.30	-26.48	-23.09	-14.10
5.	-1.24	13.04	24.21	30.54
6.	30.56	24.52	14.07	3.69
7.	-5.91	-12.27	-13.85	-11.08
8.	-4.72	3.70	12.39	18.29
9.	21.47	21.06	19.05	15.38
10.	8.89	-1.43	-14.42	-28.55
11.	-41.60	-50.28	-52.67	-46.54
12.	-32.49	-13.27	8.59	31.20
13.	47.78	58.46	59.66	

2.Ave. 3-115-2 3-115-3 3-115-4 23 Jun,85

S	.00	.25	.50	.75
8.	-6.49	3.93	12.73	19.14
9.	22.87	23.87	23.44	16.93
10.	9.98	1.46	-11.88	-28.36
11.	-43.35	-53.35	-55.30	-48.15
12.	-33.66	-14.13	8.43	30.39
13.	47.82	59.03	59.47	49.76
14.	35.75	20.19	4.47	-7.22
15.	-14.63	-20.63	-26.78	-31.19
16.	-34.87	-37.02	-32.78	-24.35
17.	-13.02	0.47	15.04	25.12
18.	33.48	35.16	30.70	23.28
19.	10.15	-2.87	-13.46	-20.71
20.	-22.12	-19.46	-14.64	-9.44
21.	-1.88	4.14	9.56	11.96
22.	15.03	14.83	11.45	9.37
23.	3.42	-1.46	-6.58	-11.62
24.	-14.90	-15.76	-16.38	-12.49
25.	-7.11	-1.94	3.20	8.54
26.	13.96	15.89	16.86	12.64
27.	9.44	5.18	-0.96	-5.95
28.	-8.13	-12.09	-9.98	-11.52
29.	-9.76	-5.96	-0.98	1.05
30.	3.11	6.08	8.39	6.90
31.	8.14	5.77	4.91	0.11
32.	-2.21	-4.87	-3.67	