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ONR ltr dtd 9 Nov 1977

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Abstract

The rotational spectrum of benzonitrile has been studied in the one-centimeter region, and approximately twenty absorption lines have been fitted to the rigid asymmetric-rotor theory. The principal moments of inertia are 89.370, 326.740, and 416.183 AU. The evidence indicates a planar symmetric structure with ring dimensions essentially the same as in benzene. The dipole moment is 4.14 ± 0.05 Debye units.

Since the addition of a CN group to the benzene ring results in a fairly long, rod-like molecule, the rotational spectrum of benzonitrile is expected to be characteristic of a near-prolate symmetric rotor with dipole moment in the small inertial axis. The principal transitions of such a rotor are of the type \( J, K \rightarrow J+1, K \), where \( K \) is the quantum number of the limiting prolate symmetric rotor. While each transition of given \( J \) would appear as a single line in the limiting symmetric case, the molecular asymmetry produces a \((2J+1)\)-fold splitting, corresponding to the various possible values of \( K \).

*The research reported in this paper was made possible by support extended to Harvard University by the Office of Naval Research under ONR Contract N5ori-76, Task Order V.
These 2J+1 lines will be grouped about a central frequency given by \((b+c)(J+1)\), and it can be shown that the lines of higher \(K\) will converge toward this central frequency in a regular manner. It is thus predicted that the benzonitrile spectrum will consist of a series of bands in which successive band-heads are separated by roughly equal frequency intervals. There is, of course, nothing to prevent the overlapping of adjacent bands.

The microwave spectrum has been observed on a commercial sample of benzonitrile which had been distilled and dried over calcium sulfate. The spectrum was found to be quite rich, but the lines were extremely broad and seemed surprisingly weak, in view of the large reported dipole moment. An extended search revealed several regions of very dense absorption separated by intervals of about 2800 mc. These regions were assigned as band-heads of high \(K\)-values, although they actually contained far more individual lines than could be expected from a single rigid rotor. On the basis of this framework, however, it was possible to pick out the lines of \(K = 0, 1,\) and \(2\), which are shifted far away from the band-heads and can thus be chosen with less ambiguity. In this way an assignment consistent with the rigid asymmetric-rotor theory was arrived at. A set of rotational constants was calculated which gave a good fit for some twenty-odd lines of the \(J = 6 \rightarrow 7, 7 \rightarrow 8,\) and \(8 \rightarrow 9\) systems. The agreement of all the calculated and observed frequencies within a few hundred kilocycles (which is not much greater than the uncertainty in frequency measurements) is adequate evidence for the assignment. The close agreement further indicates that centrifugal distortion is negligible, as is to be expected in such a heavy molecule. The rotational constants used in the assignment are given in Table I, and in Table II the assigned transitions are listed. While many additional lines could certainly be assigned,
there seems little point in further experimental and computational labor.

Attempts to study the Stark effect of benzonitrile encountered a number of difficulties. In addition to the large line breadths and highly cluttered spectrum, intensity considerations made it necessary to work with lines of high J-values, which increased the difficulties with resolution. It was possible, however, to obtain a partial resolution of the Stark effects of a few favorable lines. A detailed analysis was made on the \( 7_{25} \rightarrow 8_{26} \) transition at 23,111.1 mc. The necessary direction-cosine matrix elements were calculated by approximating the asymmetric-rotor wave functions with elliptic cylinder functions.\(^1\) It is interesting to note that a significant contribution to the Stark effect of this transition comes from the interaction of the nearly degenerate \( 7_{25} - 8_{08} \) and \( 8_{26} - 9_{09} \) pairs. While such levels with K-values differing by two cannot interact in the symmetric limit, there is sufficient asymmetry in this molecule (\( K = -0.85C \)) to permit an appreciable interaction. The dipole moment determined from this Stark effect is \( 4.14 \pm 0.05 \) Debye units. This value falls at the top of the rather wide range (3.61 to 4.14) of some two dozen reported measurements by the solution method;\(^2\) a previous measurement in the vapor state gave 4.39. It is felt that the Stark effect should give the most reliable value, since all difficulties with molecular association are eliminated.

While a great many isotopic species would be required to give a complete determination of the structure of benzonitrile, a certain amount of information can be drawn from the present data on the common species. First, the


expected planarity of the molecule is confirmed by the smallness of the inertial defect \((I_c - I_a - I_\theta = 0.073)\); furthermore, the presence of a two-fold axis (and hence, almost certainly, of \(C_{2v}\) symmetry) was demonstrated by observing the intensity alternation which results from the proton nuclear spins. If two pairs of protons can be exchanged by a 180° rotation about the small inertial axis, then the lines of even \(K\) will be enhanced over those of odd \(K\) by a factor of 10:6. An intensity alternation of this magnitude was found in the benzonitrile spectrum.

Since benzonitrile has been shown to have a planar symmetric structure, the moment of inertia about the symmetry axis should be comparable to the appropriate moment of benzene. The value of \(I_a\) determined here agrees quite closely with the benzene moment of 88.95 as determined from the rotational raman spectrum, it seems likely, then, that the addition of the CN group causes no significant distortion of the ring dimensions. Thus if we assume hexagonal symmetry for the ring and a C-H distance of 1.080 Å, the value of \(I_a\) leads to a C-C distance in the ring of 1.402 Å. If it is further assumed that the C-N distance is 1.158 Å (the value found in several similar -CN compounds) then the remaining experimental moment fixes the C-C single-bond distance as 1.419 Å. The decrease from the normal single-bond distance is clearly due to resonance between the C-N triple bond and the ring, which tends to increase the bond order of the intervening C-C bond. In fact, the length of this bond in \(C_6H_5-CN\) falls in a logical position between the corresponding lengths in \(H_3C-CN\) (1.460) and HCC-CN (1.382).


Two further points should be mentioned. The unusual line widths in benzonitrile (at least 2 mc. under the best experimental conditions) imply a very large collision cross section. In fact, rough measurements indicate an effective collision diameter of from 40 to 60 Å. While this diameter is much greater than normally encountered in microwave work, it is not too surprising when both the large physical size and the very high dipole moment of benzonitrile are taken into consideration. It should be noted that for the transitions studied here the calculated interaction of the nitrogen quadrupole moment results in a splitting of only about 0.05 mc., and thus makes a negligible contribution to the broadening.

Only about one-quarter of the observed lines were actually assigned, although these included all the strong lines. It is probable that the majority of the remaining lines are due to molecules in excited vibrational states. While there is little available vibrational data on benzonitrile, it seems likely that three or four fundamentals will be below 500 cm\(^{-1}\), and at room temperature the population of each of these states will be ten percent or more of the ground state. Since each of these states will have a rotational spectrum which is as rich as the ground state, the number of observed lines seems a reasonable order of magnitude. Although an effort was made to find regularities among the unassigned lines, the spectrum was too dense to permit any progress in this direction.
Table I. Parameters of benzonitrile

\begin{align*}
  b - c &= 332.45 	ext{ mc.} \
  b + c &= 2761.30 \
  a - \frac{1}{2}(b + c) &= 4274.7 \\
  I_a &= 89.370 \text{ A.U.Å}^2 \
  I_b &= 326.740 \
  I_c &= 416.183
\end{align*}
Table II. Assigned lines in benzonitrile

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<th>Transition</th>
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<th>Observed Frequency</th>
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<td>19,179.5 mc.</td>
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<td>19,383.4</td>
<td>19,383.5</td>
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<td>$6_{52} \rightarrow 7_{53}$</td>
<td>19,397.9</td>
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<td>$8_{18} \rightarrow 9_{19}$</td>
<td>22,938.1</td>
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Table II. (continued)

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<td>808 → 909</td>
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