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The Specificity of Electron Impact Mass Spectroscopy for the Identification of *N*-Ethyl-1-Phenylcyclohexylamine (PCE)

REFERENCE: Clark, C. C., **"The Specificity of Electron Impact Mass Spectroscopy for the Identification of *N*-Ethyl-1-Phenylcyclohexylamine (PCE),"** *Journal of Forensic Sciences*, JFSCA, Vol. 32, No. 4, July 1987, pp. 917-932.

ABSTRACT: The electron impact mass spectrum of *N*-ethyl-1-phenylcyclohexylamine (PCE) was studied using both deuterium-labeled compounds and structurally related analogs. The deuterium-labeled compounds used were d_2 · PCE with two deuterium atoms on the methylene carbon of the *N*-ethyl group, d_3 · PCE with three deuterium atoms on the methyl carbon of the *N*-ethyl group, d_4 · PCE with four deuterium atoms on the β carbons of the cyclohexyl rings, and d_5 · PCE with five deuterium atoms on the phenyl ring. Structurally related compounds used included the *N,N*-dimethyl, *N*-propyl, and cyclopentyl analogs. The identities of some major fragments and possible pathways leading to their formation are shown. Electron impact mass spectroscopy is shown to be a definitive test for the identification of PCE.

KEYWORDS: toxicology, *N*-ethyl-1-phenylcyclohexylamine (PCE), spectroscopic analysis

1-phenylcyclohexylethylamine (PCE) or more properly, *N*-ethyl-1-phenylcyclohexylamine, is a potent central nervous system depressant related to phencyclidine (PCP). Clinical tests show that its effects closely resemble those of PCP [*1*], including trance-like ecstatic states, visual distortions, dizziness, slurred speech, and manic behavior. PCE is said to have been available on the streets in California since 1969 [*2*], but it did not gain in popularity until after 1970, when PCP was controlled under the Controlled Substances Act [*3*]. PCE, along with the thiophene and morpholine analogs of PCP, were then sold on the street as noncontrolled PCP substitutes. PCE was controlled as a Schedule 1 substance in 1978. It continues to be seen on the illicit drug scene, however, because of its ease of manufacture and the fact that its synthesis does not require the use of piperidine, as does the manufacture of PCP. The sale of piperidine is closely watched and is subject to the Piperidine Reporting Act [*4*].

The dosage of PCE is low, 3 to 5 mg, and it is usually found mixed with a variety of diluents. Thus, it is frequently a difficult task to isolate sufficient pure sample for definitive tests such as infrared spectroscopy or nuclear magnetic resonance spectroscopy. PCE has excellent gas-liquid chromatographic properties. This, combined with the excellent sensitivity of the mass spectrometer, has frequently made the use of combined gas chromatography/mass spectroscopy (GC/MS) the method of choice for the identification of PCE.

Positive identification can be demonstrated by obtaining analytical data on all possible closely related analogs, a nearly impossible task, or by the more practical means of obtaining

Received for publication 5 July 1986; revised manuscript received 22 Aug. 1986; accepted for publication 26 Aug. 1986.

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a thorough understanding of the processes which lead to the observed responses. Thus, when mass spectroscopy is used for the identification of PCE, a knowledge of the identities of the major fragments and possible routes for their formation is highly desirable from a forensic science viewpoint. The electron impact (EI) mass spectrum of PCE has been published, but no attempt was made at its interpretation [5]. This paper presents an attempt to interpret, using various deuterated counterparts of PCE as aids, the EI mass spectrum of PCE. Comparison of the *m/z* of a fragment in the mass spectrum of a deuterium labeled compound with the *m/z* of the corresponding fragment in the mass spectrum of PCE shows the number of deuterium atoms in the fragment. Knowledge of the positions of the deuterium atoms in the deuterium labeled compound assists in proposing structures for the fragments and routes for their formation. Although mechanisms drawn to account for the formation of observed fragments are speculative and the structures of the fragments reaching the detector cannot be unambiguously determined, they are presented to assist in the interpretation of the EI mass spectrum of PCE. Figure 1 and Table 1 show the structure of PCE and the position of the deuterium atoms in the deuterium labeled compounds used in this study.

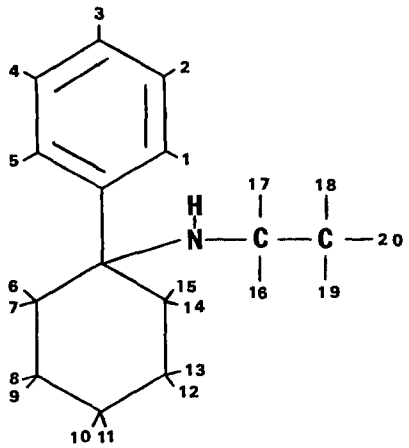

FIG. 1—*Structure of PCE and the deuterated analogs used in this study.* H = *hydrogen,* D = *deuterium. See also Table 1.*

TABLE 1—*Composition and structure of deuterium-labeled compounds used in this study. See also Fig. 1.*[a]

	Compound				
Position No.	PCE	d_2 · PCE	d_3 · PCE	d_4 · PCE	d_5 · PCE
1-5	H	H	H	H	D
6, 7, 14, 15	H	H	H	D	H
16, 17	H	D	H	H	H
18-20	H	H	D	H	H

[a] H = hydrogen, D = deuterium.

Experimental Procedure

Apparatus

For all mass spectroscopic work except high resolution, a Finnigan 4530 quadrupole mass spectrometer was used. Ionizing voltage was 70 eV. Source temperature was 200°C. Scan rate was 0.5 s/scan and the scan range was 40 to 220 mass units. Sample introductions were made via a 25-m by 0.31-mm inside diameter fused silica capillary column with a 0.52-μm film thickness of OV-1. Split ratio was approximately 50:1. Column temperature was 170°C, and the injection temperature was 260°C. High-resolution data was obtained on a Finnigan Mat 8230 magnetic sector mass spectrometer at a resolution of 8500. Ionizing voltage was 70 eV. Source temperature was 175°C. Sample introduction was via solid probe.

Synthesis and Procedure

The procedure used for the syntheses of the PCE, d_4 · PCE and d_5 · PCE used in this study was Method B(a) of Maddox et al. [*1*]. Before its reaction with ethylamine, the cyclohexanone used in the synthesis of d_4 · PCE was allowed to stand overnight at room temperature in basic C_2H_5OD to effect exchange of the β hydrogens. The d_5 · bromobenzene used in the synthesis of d_5 · PCE and the C_2H_5OD used in the synthesis of d_4 · PCE both had stated isotopic purities of 99%. The d_2 · PCE and d_3 · PCE were produced by the lithium aluminum hydride or lithium aluminum deuteride reduction of the amide produced by reacting acetic anhydride or d_3 · acetic anhydride with 1-phenylcyclohexylamine. The 1-phenylcyclohexylamine was produced from 1-phenylcyclohexene by the method of Maddox et al. [*1*]. The lithium aluminum deuteride used to produce the d_2 · PCE and the d_3 · acetic anhydride used to produce the d_3 · PCE both had stated isotopic purities of 99%.

All deuterated starting materials were purchased from MSD isotopes. The d_2 · PCE had an isotopic purity of approximately 96%, calculated from the (m/z 161)/(m/z 161 + m/z 162) intensity ratio in its mass spectrum. For accuracy of measurement, isotopic purities of the deuterated compounds were determined using the base peak fragment in the mass spectra, whenever possible. For d_4 · PCE the molecular ion fragment was used, since all four β hydrogens are not present in the base peak fragment. The isotopic purity of the d_3 · PCE was approximately 89%, calculated from the (m/z 162)/(m/z 162 + m/z 163) intensity ratio. The approximate isotopic purity of the d_4 · PCE, 85%, was calculated from the (m/z 206)/(m/z 206 + m/z 207) intensity ratio after working a correction for the normal M-1 contribution. The d_5 · PCE had an approximate isotopic purity of 88%, calculated from the (m/z 164)/(m/z 164 + m/z 165) intensity ratio in its mass spectrum. Figures 2 to 6 show the EI mass spectra of PCE, d_2 · PCE, d_3 · PCE, d_4 · PCE and d_5 · PCE, respectively. The PCE and its deuterium labeled counterparts were isolated as the free bases and had a minimum chemical purity of 95%, determined by gas-liquid chromatography. A 1-mg/mL solution of each of the free bases was used for sample introduction into the mass spectrometer.

Results and Discussion

m/z *202*

A prominent ion of m/z 202 appears in the EI mass spectrum of PCE. Since α-bond cleavage is favored (although usually with loss of the largest group), a loss of an ethyl hydrogen from the carbon α to the nitrogen might be expected. This loss is shown in Fig. 7. This is not the source of the m/z 202 fragment, however, since the spectrum of d_2 · PCE shows no significant M-2 ion. Examination of the spectrum of d_5 · PCE clearly shows that it is a phenyl hydrogen which is lost, as shown in Fig. 8. This same hydrogen loss has been shown to be

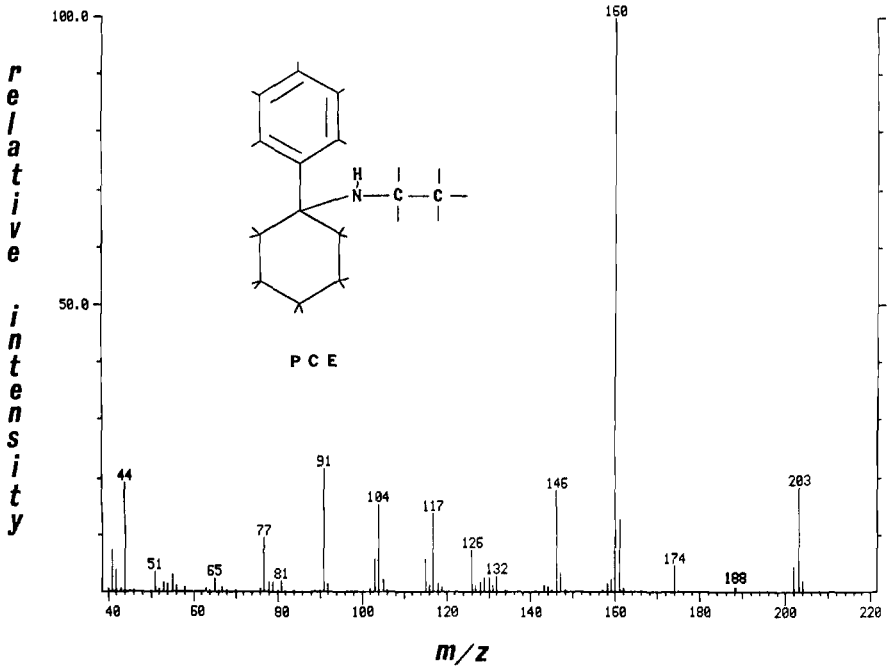

FIG. 2—*EI mass spectrum of PCE.*

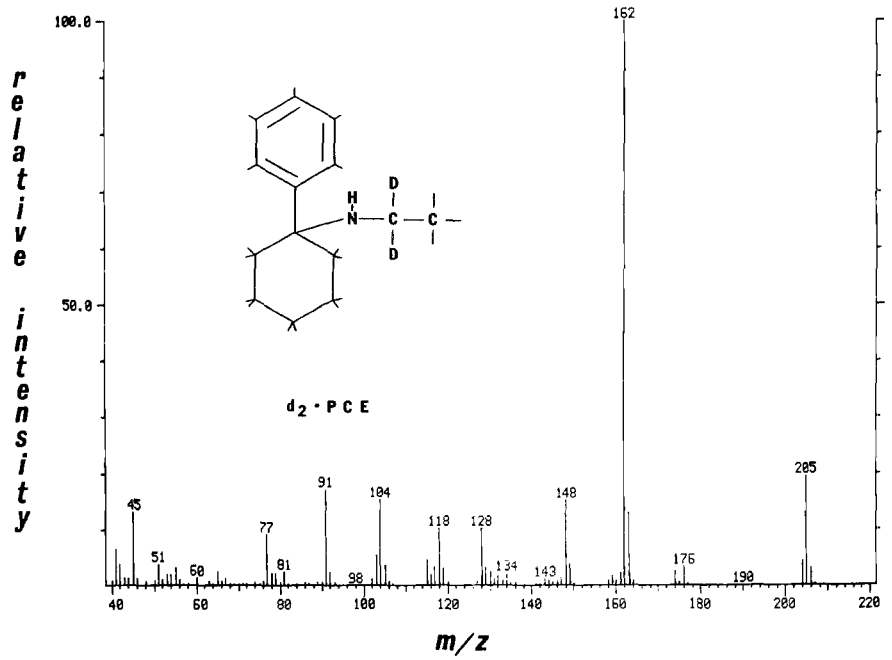

FIG. 3—*EI mass spectrum of* $d_2 \cdot$ *PCE.*

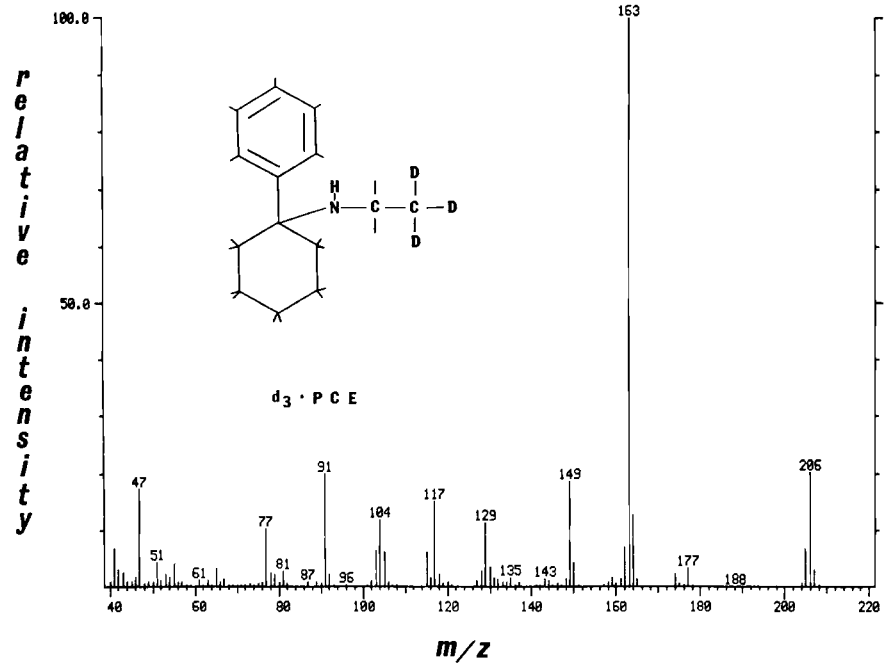

FIG. 4—*EI mass spectrum of* d_3 · *PCE.*

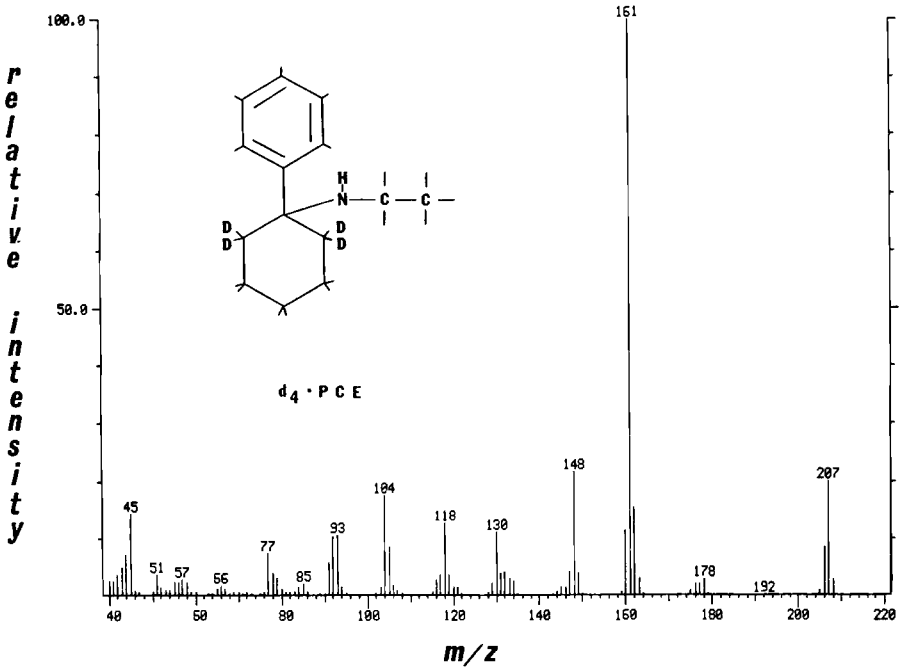

FIG. 5—*EI mass spectrum of* d_4 · *PCE.*

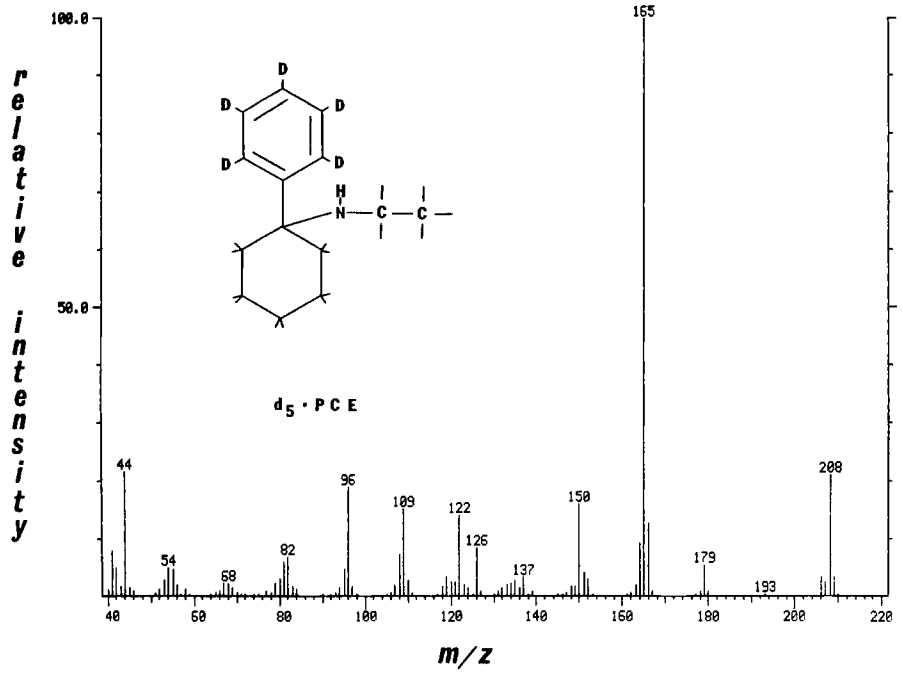

FIG. 6—*EI mass spectrum of* d_5 · *PCE.*

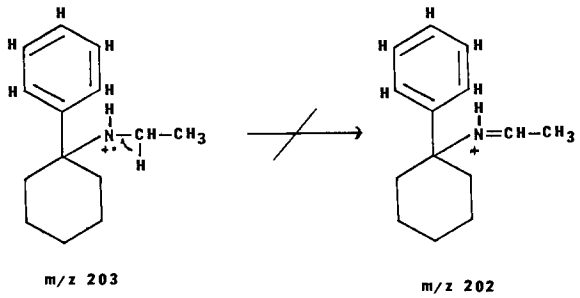

FIG. 7—*Expected loss of a hydrogen* α *to the nitrogen to produce* m/z *202.*

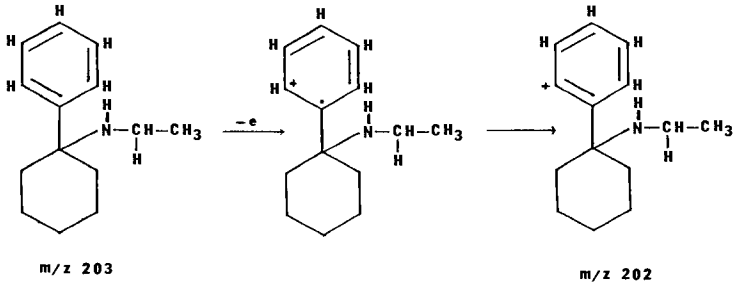

FIG. 8—*Loss of a phenyl hydrogen to produce* m/z *202.*

responsible for the *m/z* (M-1) in the EI mass spectrum of PCP [6]. Loss of an ethyl hydrogen from the carbon α to the nitrogen does occur but leads to other species as shown below.

m/z *188*

A very weak (RI < 1%) M-15 occurs in the EI mass spectrum of PCE. The observance of the *m/z* at M-18 in the mass spectrum of d_3 · PCE shows that this fragment is formed by the loss of a methyl radical from the ethyl group, as shown in Fig. 9. This is the expected cleavage of the bond β to the nitrogen.

m/z *174 (M-29)*

Examination of the mass spectrum of d_2 · PCE shows that this fragment has at least two different origins. In the d_2 compound, one route (or routes) produces a M-29, the other produces a M-31. The M-31 is due to the rupture of the N-C bond and loss of the ethyl radical. This is substantiated by the observance of a M-32 (*m/z* 174) in the mass spectrum of the d_3 · PCE. The observance of the loss of the ethyl group is in marked contrast to that observed for *N*-ethylcyclohexylamine by Pelah et al. Concerning that mass spectrum, it was stated that "The formation of the M-29 peak does not involve loss of the *N*-ethyl group to a significant extent" [7]. The other route or routes responsible for the formation of the M-29 fragment in the mass spectrum of PCE must result from the fragmentation of the cyclohexyl ring since the M-29 fragment in the mass spectrum of d_5 · PCE shows the phenyl ring to be intact. Examination of the corresponding fragments in the mass spectrum of d_4 · PCE shows the M-29 fragment at *m/z* 178 as a result of the loss of the ethyl group and two other fragments, one containing two deuterium atoms (*m/z* 176) and another with three deuterium atoms (*m/z* 177). This is in agreement with the statement by Pelah et al. that the M-29 fragment in the EI mass spectrum of *N*-ethylcyclohexylamine is formed by the loss of an ethyl radical (· C_2H_5) from the cyclohexyl ring in a random manner [7].

m/z *160 (M-43)*

The formation of this base peak in the EI mass spectrum of PCE can be rationalized as shown in Fig. 10. This is a common pathway for cyclohexylamine itself and both *N*-mono and *N,N*-di substituted cyclohexylamines [8]. Supporting this structure are the observations that the corresponding fragment occurs at *m/z* 162 in the d_2 · PCE mass spectrum and at *m/z* 163 in the d_3 · PCE mass spectrum. This shows the PCE *m/z* 160 fragment to have all five *N*-ethyl hydrogens. Additionally, the fragment *m/z* 161 in the mass spectrum of d_4 · PCE shows the PCE *m/z* 160 fragment to contain only one of the original four hydrogens on the carbons β to the nitrogen in the PCE molecule. The fragment *m/z* 165 in the mass spectrum

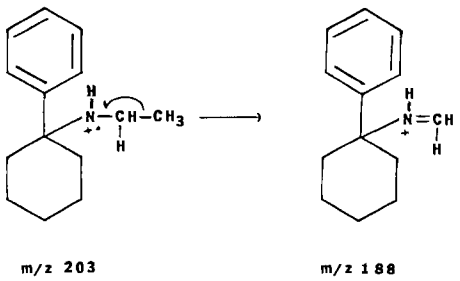

FIG. 9—*Suggested route for the formation of the* m/z *188 fragment in the EI mass spectrum of PCE.*

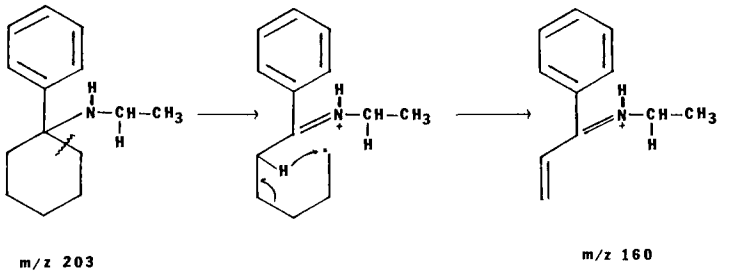

FIG. 10—*Suggested route for the formation of the* m/z *160 fragment in the EI mass spectrum of PCE.*

of d_5 · PCE shows the PCE *m/z* 160 fragment to contain all five phenyl hydrogens. In the case of cyclohexylamine itself, this fragmentation route is further strengthened by the presence of a "meta-stable peak" at the appropriate *m/z* value [*9*].

m/z *146 (M-58)*

Examination of the *m/z* values of the corresponding fragments in the mass spectra of the deuterium labeled compounds reveal that the *m/z* 146 fragment in the mass spectrum of PCE contains all five of the *N*-ethyl hydrogens, only two of the four hydrogens located on the β carbons of the cyclohexyl ring, and only four of the phenyl hydrogens. This fragment is analogous to the *m/z* 186 in the mass spectrum of PCP and is thought to be formed by the pathway shown in Fig. 11. It uses the route, suggested by Budzikiewicz et al. for *N*-ethylcyclohexylamine, of a concerted homolytic fission of the cyclohexyl ring with elimination of two molecules of ethylene [*8*].

m/z *126 (M-77)*

Examination of the *m/z* of the corresponding fragment in the mass spectra of the deuterium-labeled compounds shows that the *m/z* 126 fragment in the mass spectrum of PCE contains all five of the *N*-ethyl hydrogens, all four of the hydrogens located on the β carbons of the cyclohexyl ring and none of the phenyl hydrogens. This fragment is due then to simple loss of the phenyl ring caused by the rupture of the bond α to the phenyl ring and the charge residing on the nitrogen atom. A *m/z* 77, caused by the charge residing on the aromatic portion, is also seen in the mass spectrum of PCP.

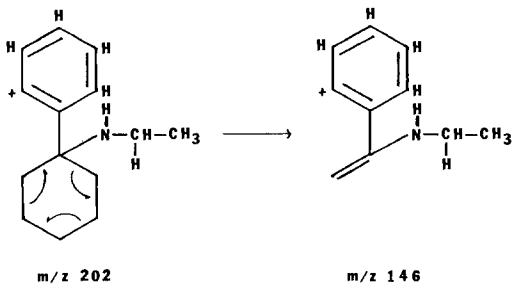

FIG. 11—*Suggested route for the formation of the* m/z *146 fragment in the EI mass spectrum of PCE.*

m/z *117, 115*

Examination of the corresponding fragments in the mass spectra of the deuterium labeled compounds reveals that the PCE *m/z* 117 fragment contains only one of the hydrogens from the *N*-ethyl methylene group, none of the hydrogens from the methyl group, one of the hydrogens located on the cyclohexyl carbons β to the nitrogen, and all five phenyl hydrogens. A proposed structure and mechanism for its formation are shown in Fig. 12. It follows a pathway suggested by Bailey and Legault [*10*] for the formation of the *m/z* 117 fragment in EI mass spectrum of *N*-propyl-1-phenylcyclohexylamine. This open structure seems more reasonable than the phenylcyclopropane radical proposed by Rao and Soni [*11*] for the structure of the *m/z* 117 fragment in the EI mass spectrum of PCP. Although no mechanism was presented for its formation in that paper, the cyclic structure presented nearly precludes the inclusion of the required *N*-methylene hydrogen. However, close examination of the *m/z* 115 to *m/z* 120 cluster in the mass spectrum of the d_2 · PCE gives some indication that the *m/z* 117 PCE fragment may be formed by more than one route. The d_2 · PCE *m/z* 118 fragment is less intense than the PCE *m/z* 117 fragment, and some *m/z* 117 remains in its mass spectrum, indicating that the PCE *m/z* 117 may have a contributor which does not include the *N*-ethyl methylene hydrogen. Papers by Bailey and Legault [*10*] and by Rao and Soni [*11*] both indicate that the *m/z* 115 fragment is formed by the expulsion of two hydrogens from the *m/z* 117 fragment. The mass spectrum of the d_3 · PCE shows that none of the methyl hydrogens are included in the PCE *m/z* 115 fragment. However, the mass spectra of d_2 · PCE, d_4 · PCE, and even d_5 · PCE all clearly indicate that a variety of structures or pathways or both for the formation of the *m/z* 115 fragment are available.

m/z *104*

High-resolution mass spectroscopy shows that the *m/z* 104 fragment in the EI mass spectrum of PCE is composite. One component has an empirical composition of C_7H_6N, the other has an empirical composition of C_8H_8, present in about a 4 : 1 intensity ratio. Examina-

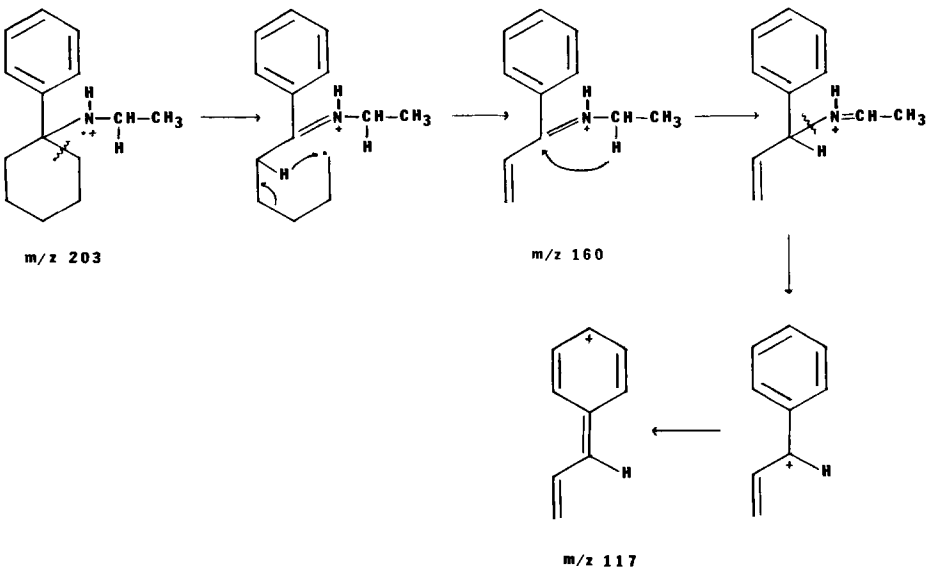

FIG. 12—*Suggested route for the formation of the* m/z *117 fragment in the EI mass spectrum of PCE.*

tion of the *m/z* 104 to *m/z* 109 mass units in the mass spectra of PCE and its deuterium labeled counterparts shows that the major fragment, C_7H_6N, contains no hydrogens from the *N*-ethyl group and no hydrogens from the β cyclohexyl carbons, but does contain all five phenyl hydrogens. Injection into the GC/MS of PCE dissolved in C_2H_5OD produces a prominent *m/z* 105, showing that it is the labile hydrogen on the nitrogen atom which is retained in this fragment. Cooper[2] has suggested the structure for this fragment and the mechanism for its formation, which are shown in Fig. 13.

m/z *91*

This is the well-known tropylium or benzylic ion. Examination of the mass spectra of d_4 · PCE and d_5 · PCE show the fragment to contain two hydrogens from the β carbons of the cyclohexyl ring and five hydrogens from the phenyl ring. The mass spectra of the d_2 · PCE and d_3 · PCE show the fragment to contain no *N*-ethyl hydrogens. The fact that the corresponding fragment appears as a multiplet in the mass spectrum of d_4 · PCE indicates that there may be more than one pathway for its formation. Alternatively, this could also be caused by a deuterium isotope effect discriminating against the transfer of deuteriums from the β carbons if one assumes that β hydrogens, rather than an intact methylene group, are transferred to the aromatic ring.

m/z *77*

In the section of this paper concerning *m/z* 126, it was suggested that *m/z* 77 in the mass spectrum of PCE could be due to simple expulsion of the phenyl ring with the charge remaining on the aromatic portion. The mass spectra of d_2 · PCE and d_3 · PCE substantiate this, but the mass spectrum of d_4 · PCE shows a multiplet of fragments of masses differing by one mass unit in this *m/z* 77 to *m/z* 85 area. In addition, the mass spectrum of d_4 · PCE shows slightly enhanced *m/z* 78 and *m/z* 79. This indicates that at least some of the *m/z* 77 in the mass spectrum of PCE may be related to the *m/z* 91 fragment, in which the phenyl ring has expanded to included a methylene group. The clusters around *m/z* 54 and *m/z* 68 in the mass spectrum of d_5 · PCE also suggest this. Once in the expanded ring, all carbons and hydrogens are treated equally [*12*]. Reverting to the benzylic form and breaking off the phenyl ring would lead to the results observed in the d_4 · PCE and d_5 · PCE mass spectra.

m/z *44*

This fragment is analogous to the *m/z* 84 fragment in the EI mass spectrum of PCP and is thought to be formed by the same process. This suggested mechanism is shown in Fig. 14.

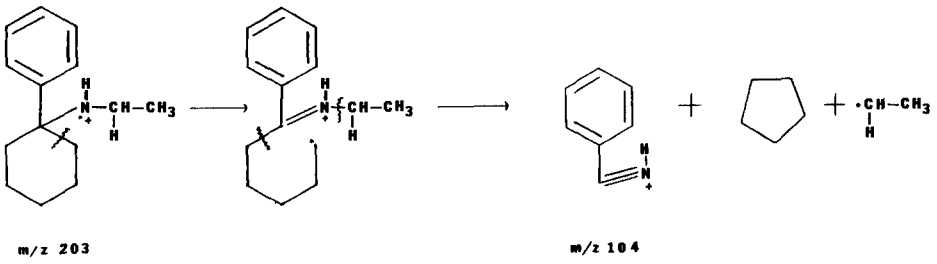

FIG. 13—*Suggested route of formation and structure of the* m/z *104 fragment in the EI mass spectrum of PCE.*

[2]D. A. Cooper, personal communication, July 1985.

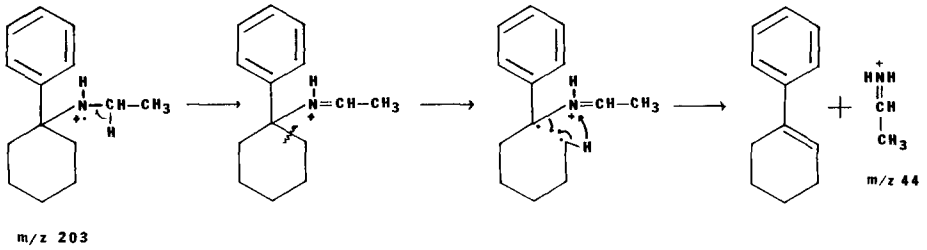

FIG. 14—*Suggested route for the formation of the* m/z *44 fragment in the EI mass spectrum of PCE.*

Observations of the corresponding fragment in the mass spectra of the deuterated analogs show that the fragment contains one hydrogen from the *N*-ethyl methylene group, three hydrogens from the β carbons of the cyclohexyl ring, and no phenyl hydrogens. The proposed structure agrees with these observations.

Since a molecular ion is observed in the EI mass spectrum of PCE, only a compound with the same molecular weight as PCE could reasonably be mistaken for it. Assuming the phenyl group to be present, only a few possibilities exist. An *N*-methylene group could be removed from the *N*-ethyl group and placed on the nitrogen, on the phenyl ring, onto one of the carbon atoms comprising the saturated ring, or included in the saturated ring. All but the first possibility result in the loss of the *m/z* 44 fragment.

The mass spectrum of *N,N*-dimethyl-1-phenylcyclohexylamine is shown in Fig. 15. It bears a close resemblance to that of PCE. However, it is distinguishable from it by major

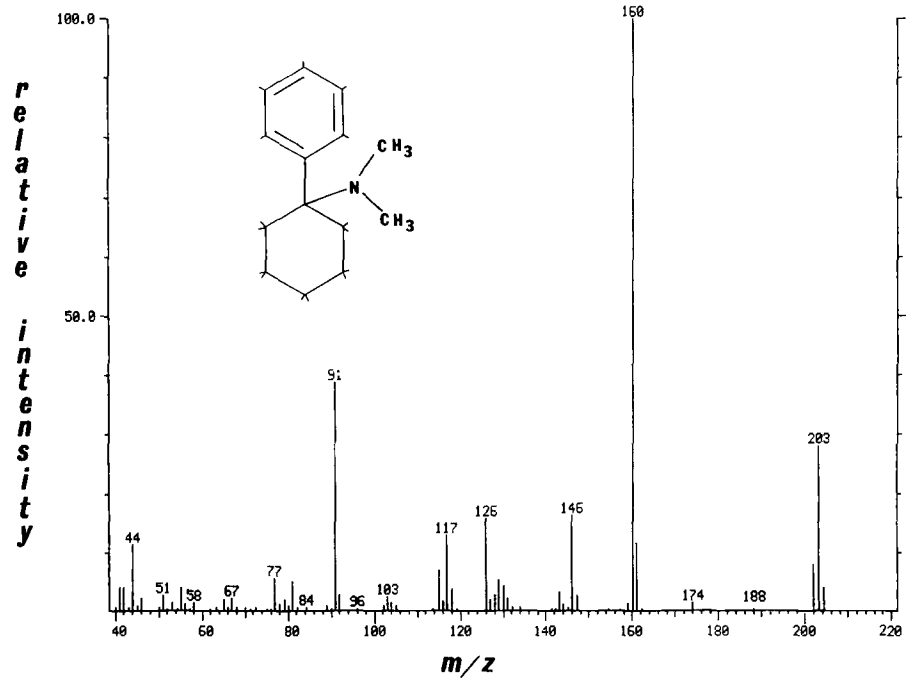

FIG. 15—*EI mass spectrum of* N,N-*dimethyl-1-phenylcyclohexylamine.*

intensity differences at *m/z* 91 and *m/z* 126 and by the fact that the mass spectrum of *N,N*-dimethyl-1-phenylcyclohexylamine contains no appreciable *m/z* 104 fragment, as does that of PCE. If a methylene group from the cyclohexyl ring is removed and placed on the *N*-alkyl group, the phenyl ring, or on one of the four available cyclopentyl carbons, a larger effect on the mass spectrum is produced.

The mass spectrum of *N*-propyl-1-phenylcyclopentylamine is shown in Fig. 16. It is known that the fragments responsible for the base peaks in the EI mass spectra of *N*-ethylcyclopentylamine and *N*-ethylcyclohexylamine have the same structure [7]. Their routes of formation are similar but differ in the size of the fragment lost, $\cdot C_2H_5$ in the former and $\cdot C_3H_7$ in the latter. Similarly, the smaller loss accounts for the base peak of *N*-propyl-1-phenylcyclopentylamine at *m/z* 174, as compared to that of *m/z* 160 for PCE. Figure 17 shows a suggested route for the formation of the base peak of *N*-propyl-1-phenylcyclopentylamine.

Removing a methylene group from the saturated ring and placing it on a β carbon gives rise to two possible routes of formation of the fragment analogous to the PCE *m/z* 160 fragment. These two routes are shown in Fig. 18. Retention in the charged fragment of the methyl-bearing carbon leads to the *m/z* 174 fragment. Loss of this carbon leads to the *m/z* 160 fragment. The EI mass spectrum of *N*-ethyl-1-phenyl-2-methylcyclopentylamine is shown in Fig. 19. The situation is much the same when the methylene group is placed on a γ carbon of the cyclopentyl ring. Figure 20 shows the two possible fragmentation routes for the fragment analogous to the PCE *m/z* 160 fragment. Figure 21 shows the EI mass spectrum of *N*-ethyl-1-phenyl-3-methylcyclopentylamine. The EI mass spectra of both *N*-ethyl-1-phenyl-2-methylcyclopentylamine and *N*-ethyl-1-phenyl-3-methylcyclopentylamine show enhanced *m/z* 174 fragment intensities resulting from retention in the charged fragment of the methyl-bearing carbon atom.

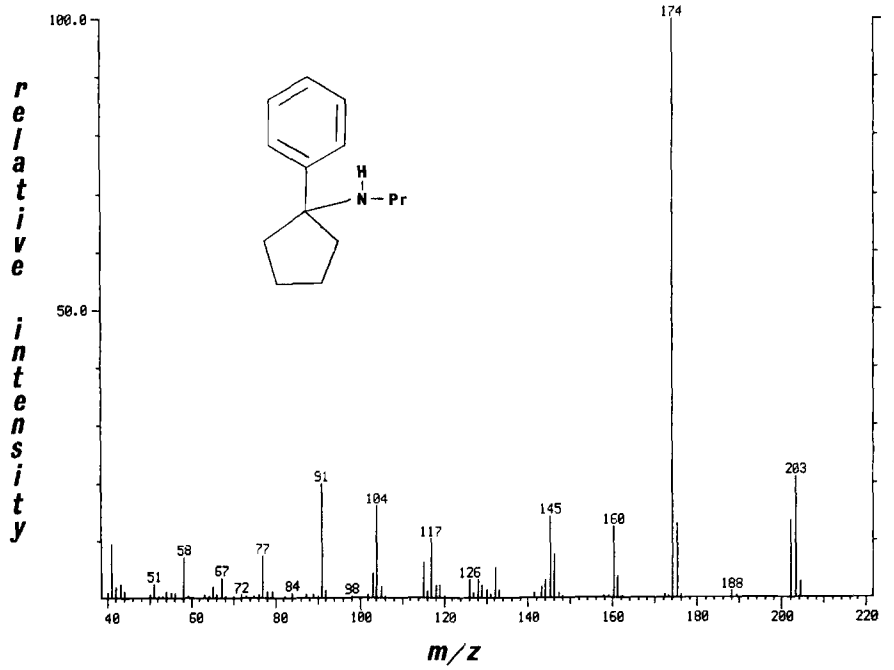

FIG. 16—*EI mass spectrum of* N-*propyl-1-phenylcyclopentylamine.*

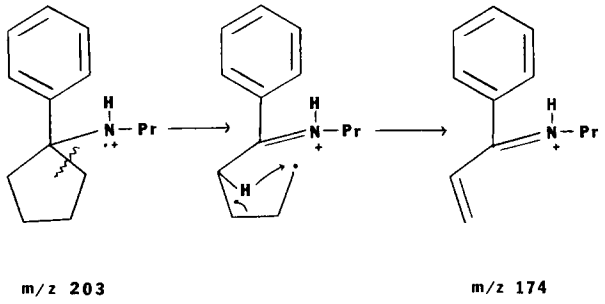

FIG. 17—*Suggested route for the formation of the* m/z *174 fragment in the EI mass spectrum of* N-*propyl-1-phenylcyclopentylamine.*

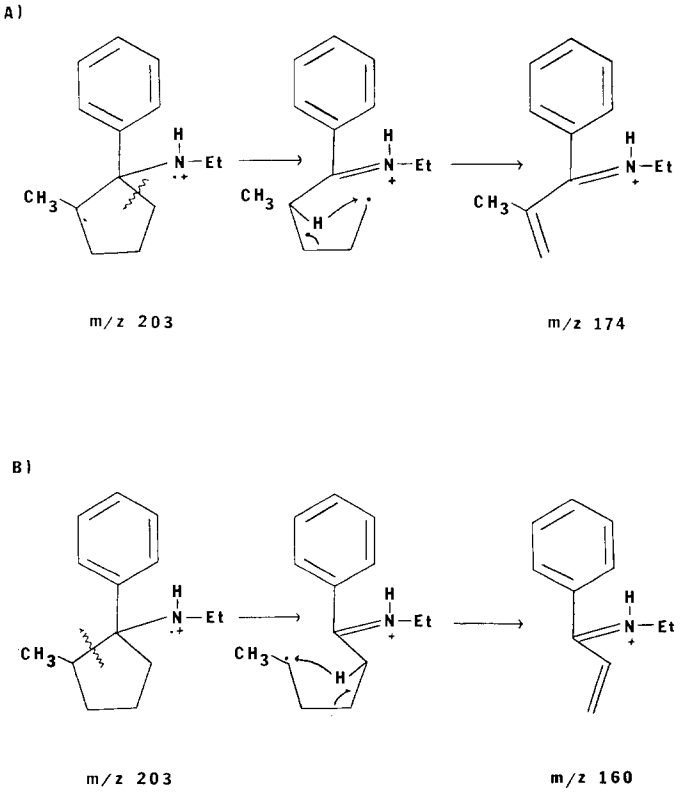

FIG. 18—*Two suggested mechanisms for the decomposition of the cyclopentyl ring of* N-*ethyl-1-phenyl-2-methylcyclopentylamine.*

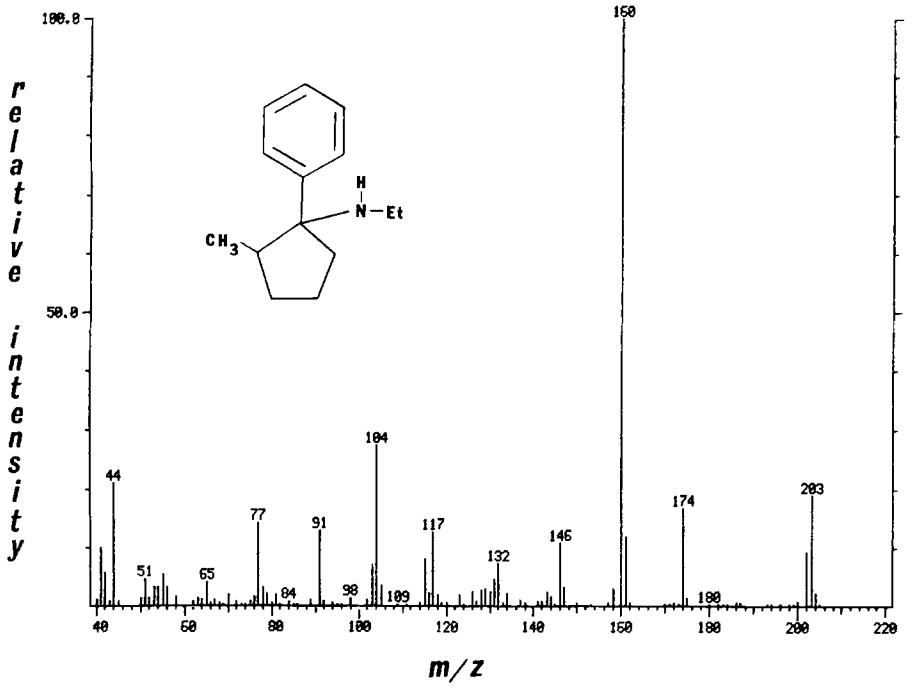

FIG. 19—*EI mass spectrum of* N-*ethyl-1-phenyl-2-methylcyclopentylamine.*

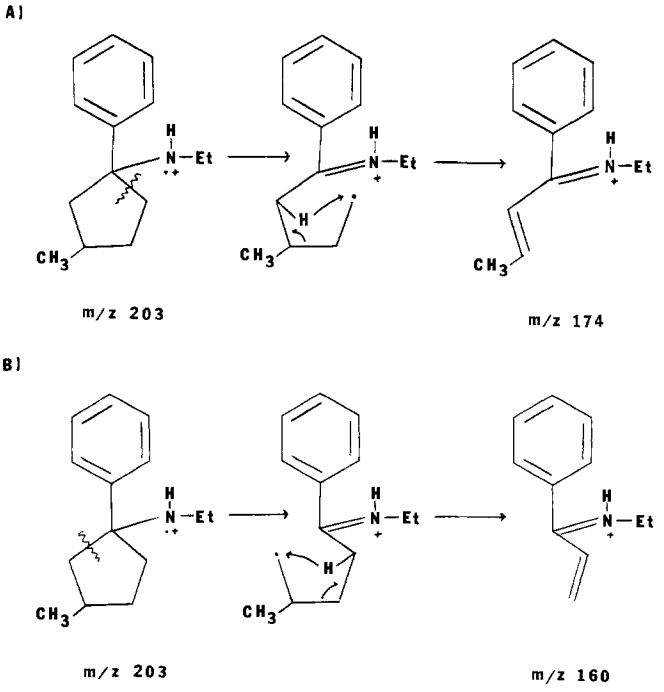

FIG. 20—*Two suggested mechanisms for the decomposition of the cyclopentyl ring of* N-*ethyl-1-phenyl-3-methylcyclopentylamine.*

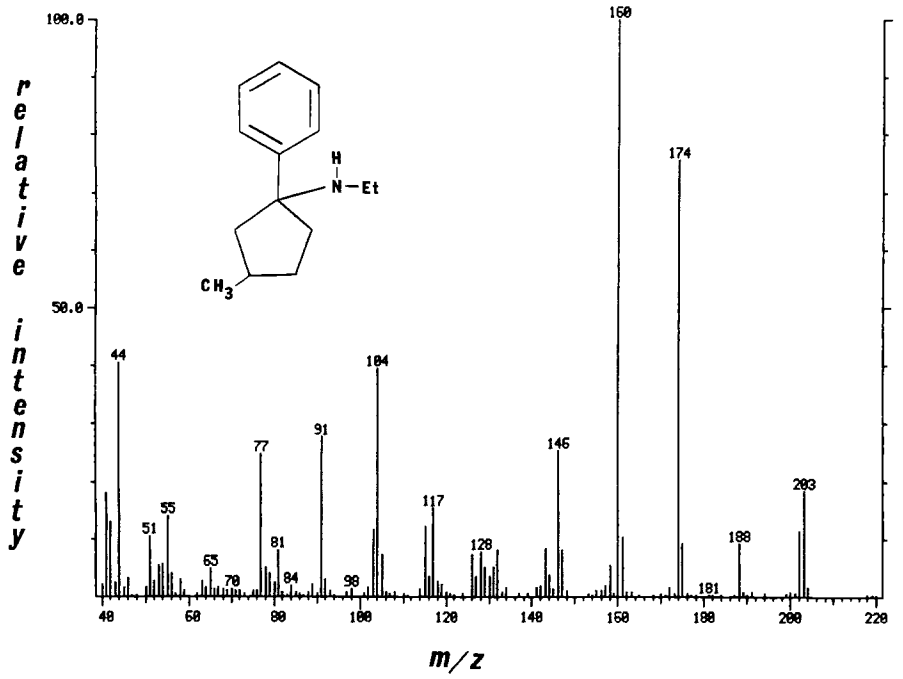

FIG. 21—*EI mass spectrum of* N-*ethyl-1-phenyl-3-methylcyclopentylamine.*

Conclusion

It has been shown in this paper that each of the three major portions of the PCE molecule (phenyl, cyclohexyl, and *N*-ethyl) is found intact in at least one observable fragment. This fact, other data presented in this paper, the multitude of identifiable fragments, and the fact that PCE possesses an observable molecular ion under EI conditions work together to make EI mass spectroscopy a definitive test for the identification of PCE.

Acknowledgment

The author wishes to thank Donald Cooper of the DEA Special Testing and Research Laboratory for providing the high-resolution mass spectroscopic data used in this study and for his constructive comments during the preparation of this manuscript.

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