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Abuse of Smoking Methamphetamine Mixed with Tobacco: II. The Formation Mechanism of Pyrolysis Products

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ABSTRACT: The pyrolysis products of smoking methamphetamine mixed with tobacco were determined by gas chromatography (GC) and GC/mass spectrometry (GC/MS) methods. The mainstream smoke contained methamphetamine (14.5% of the initial methamphetamine), phenylacetone (3.1%), *N*-cyanomethylmethamphetamine (1.9%), *trans*- β -methylstyrene (1.7%), *N*-formylmethamphetamine (1.5%), and other products (each <1%). The amount of each pyrolysis product in the sidestream smoke was less than that in the mainstream smoke by a factor of over 5, except for methamphetamine (10.5%) and *N*-formylmethamphetamine (1.4%). The formation mechanism of these products was investigated, by use of a pyrolyzer, from the standpoint of the material, pyrolysis temperature, and pyrolysis atmosphere. Although several products (for example, dimethylamphetamine and *trans*- β -methylstyrene) were formed by thermal self-decomposition of methamphetamine alone, most of the products, except *N*-cyanomethylmethamphetamine, were formed chiefly by the thermal reaction of methamphetamine with cigarette components. The formation of *N*-cyanomethylmethamphetamine required air and a high pyrolysis temperature. Air and a high pyrolysis temperature generally accelerated the formation of the pyrolysis products.

KEYWORDS: toxicology, methamphetamine, tobacco, pyrolyses, drug abuse by smoking, pyrolysis products, *N*-cyanomethylmethamphetamine

In patterns of drug abuse, smoking a drug produces peculiar influences on abusers in comparison with other drug abuse patterns. The peculiar influences come from the production of unexpected compounds from decomposition produced by burning or heating drugs and from passive inhalation. It has been reported that smoking abuse of cocaine, or crack, has an extremely rapid effect upon the abuser [1]. Moreover, it is presumed that there are many toxicological problems of drug abuse by smoking which have not been solved such as bioavailability of the drug and its products, the peak concentration of the drug in plasma, its half-life in plasma, and its synergism. Various compounds for which there are no toxicological data have been produced by the smoking of many drugs, including heroin [2], cocaine [3], and phencyclidine [4-7]. In a previous paper [8], the authors of this paper reported the production of several acylated and alkylated methamphetamines produced by smoking methamphetamine mixed with tobacco. We have found that *N*-cyanomethylmethamphetamine, a main product of smoking methamphet-

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amine with tobacco, produces a high spontaneous motor activity in mice and rats which is no less than that produced by methamphetamine [9].

This paper describes the determination and the formation mechanism of the products of smoking methamphetamine mixed with tobacco.

Experimental Procedures

Chemicals

Methamphetamine hydrochloride (HCl) was purchased from Dainippon Pharmaceutical Co. (Osaka, Japan). *Trans*- β -methylstyrene, allylbenzene, styrene, *N*-methylphenethylamine, and *N*-ethylbenzylamine were purchased from Tokyo Kasei Kogyo Co. (Tokyo, Japan), and the *cis* and *trans* mixture of β -methylstyrene was obtained from Wako Pure Chemical Industries, Ltd. (Osaka, Japan). Phenylacetone, *N*-formylmethamphetamine, *N*-acetylmethamphetamine, dimethylamphetamine, and *N*-cyanomethylmethamphetamine were synthesized by the method described in the earlier paper [8]. All the reagents used in this study were analytical grade. All solvents were distilled before use.

Instrumentation

A Shimadzu (Osaka, Japan) Model GC-7A gas chromatograph equipped with a flame-ionization detector (FID) and a Shimadzu SPL-9 split injector system were used for determination of the pyrolysis products of methamphetamine. The injector was used at a split ratio of approximately 1:20 and maintained at 240°C. A DB-1 [J & W Scientific Inc. (Folsom, California)] column 30 m long, with a 0.32-mm inner diameter (ID) and a 1.0- μ m film thickness, was used under the temperature program of 5°C/min from 60 to 230°C, with a 1-min hold at 60°C and a 20-min hold at 230°C.

Gas chromatography (GC) mass spectra were obtained in the electron impact (EI) mode by a Finnigan-MAT INCOS-50 GC/mass spectrometer equipped with a Hewlett-Packard Model 5890A GC. A DB-1 column of 30 m, with a 0.25-mm ID and a 0.25- μ m film thickness, was used for GC/mass spectrometry (GC/MS). The temperature program of the injection port and the oven was the same as that for GC analysis. Pyrolysis-gas chromatography (Py-GC) and pyrolysis-GC/MS (Py-GC/MS) were carried out by connecting Japan Analytical Industry Co. (JAI) (Tokyo, Japan) curie point pyrolyzers (Models JHP-2 and JHP-3) to GC and GC/MS.

Precise mass spectra were taken with a Hitachi (Tokyo, Japan) Model M-2000 mass spectrometer in a direct ionization (DI) mode. Infrared (IR) spectra were recorded on a Nicolet (Madison, Wisconsin) Fourier-transform IR (FT-IR) spectrometer. Proton magnetic resonance (PMR) spectra and carbon-13 resonance (CMR) spectra were obtained by a JEOL (Tokyo, Japan) Model JNM-GSX270 nuclear magnetic resonance (NMR) spectrometer.

Smoking Experiments

Figure 1 shows the smoking apparatus, which was made for collecting both mainstream and sidestream smoke. In this experiment, a nonfilter cigarette, "Peace" [with a 70-mm length by 8-mm ID (Japan Tobacco Inc.)], was injected with 0.4 mL of 50-mg/mL methamphetamine HCl in methanol by a syringe at a point within 50 mm of the top of the cigarette. The smoking experiment was carried out in the same manner as in the authors' previous paper [8], using 35-mL puffs for 2 s with 30- or 60-s intervals. The tar from the mainstream smoke was collected on a glass fiber filter [GA 200, 45-mm ID

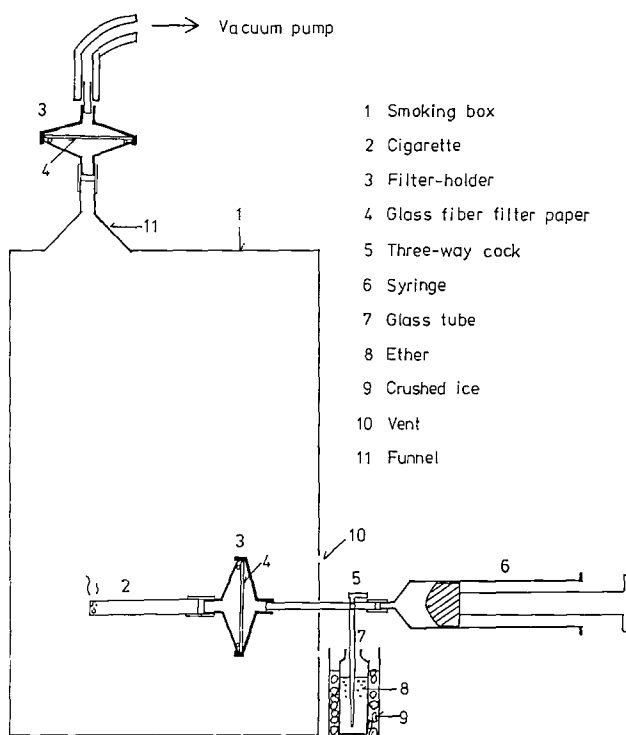


FIG. 1.—Smoking apparatus.

(Toyo Roshi Co., Ltd.) in the lower filter holder, as shown in Fig. 1. The tar from the sidestream smoke was also collected on the filter paper in the upper holder by aspirating the apparatus with a vacuum pump at a flow rate of about 200 mL/s.

The collected tars were extracted successively with 50 and 25 mL of the mixture of dichloromethane (CH_2Cl_2) and methanol (CH_3OH) (9:1) for the mainstream smoke and for the sidestream smoke, respectively. The volatile compound in the mainstream smoke was passed through the glass fiber and was absorbed into precooled ether in the test tube. The butt was extracted with 25 mL of the solvent just described.

In separate experiments, *N*-methylphenethylamine HCl or *N*-ethylbenzylamine HCl was mixed in a cigarette, and the smoke was analyzed using a procedure similar to that described earlier.

Combustion Experiments

The methamphetamine and cigarette components were put into a glass tube (8-mm ID) separately or together, and the sample in the glass tube was heated with a burner. Ten milligrams of methamphetamine HCl, 40 mg of cigarette paper, and 400 mg of tobacco were used in this experiment. The gas produced was collected in manner described in the section on smoking experiments.

Pyrolytic Experiments

To investigate the effect of the pyrolytic temperature on the formation of pyrolysis products, methamphetamine was pyrolyzed in Curie point pyrolyzers at a temperature from 358 to 764°C under a helium stream or in the air. About 10 µg of methamphetamine

HCl was mixed with 80 μg of tobacco for Py-GC, and about 50 μg of methamphetamine HCl was mixed with 500 μg of tobacco for Py-GC/MS. The mixtures were wrapped up in an aluminum foil and further wrapped with a pyro-foil. Two minutes after the sample had been set in the pyrolyzer oven, the temperature was maintained at the Curie point of the pyro-foil for 4 s. The JHP-2 and JHP-3 pyrolyzers were used for the pyrolysis in air or in helium, respectively.

Analytical Procedures

Five millilitres of extract solution from the smoking tar or the butt, 50 μg of biphenyl as an internal standard (IS), and a drop of concentrated HCl were placed in a 5-mL test tube, and the mixture was evaporated to dryness under reduced pressure. The residue was made basic by adding three drops of 28% ammonia solution, after which extraction was performed with 3 mL of *n*-pentane three times. The combined extract was evaporated under a nitrogen stream, and the residue was redissolved in 100 μL of CH_2Cl_2 . The ether solution through which the mainstream smoke had passed was concentrated to a small volume under a nitrogen stream. One microlitre of the solution was injected into GC and GC/MS apparatuses.

Because *N*-formylmethamphetamine and *N*-cyanomethylmethamphetamine could not be perfectly separated on a gas chromatogram, these two compounds could be determined by GC/MS from the area ratios of each base peak in mass spectra, *m/z* 86 and 97, to the IS base peak, *m/z* 154.

Calibration curves for the pyrolysis products were prepared as follows. Standard solutions were made by dissolving the standards at the concentration ranges of 10 to 60 $\mu\text{g}/\text{mL}$ for methamphetamine; 0.75 to 9.0 $\mu\text{g}/\text{mL}$ for phenylacetone; 0.5 to 6.0 $\mu\text{g}/\text{mL}$ for both *N*-formylmethamphetamine and *N*-cyanomethylmethamphetamine; 0.5 to 3.0 $\mu\text{g}/\text{mL}$ for dimethylamphetamine; and 0.25 to 1.5 $\mu\text{g}/\text{mL}$ for *trans*- β -methylstyrene, *N*-acetylmethamphetamine, and *N*-propionylmethamphetamine. These solutions were analyzed as described earlier. The area ratio of each pyrolysis product to IS (A_p/A_{IS}) was plotted as the ordinate and the weight, *W*, of the compound as the abscissa. The calibration curves of the standards were all linear (methamphetamine: $W = 97.18 \times A_p/A_{IS} - 6.71$ [$\gamma = 0.9998$]; *trans*- β -methylstyrene: $W = 154.6 \times A_p/A_{IS} - 0.96$ [$\gamma = 0.9976$]; phenylacetone: $W = 331.61 \times A_p/A_{IS} - 2.34$ [$\gamma = 0.9998$]; dimethylamphetamine: $W = 88.54 \times A_p/A_{IS} - 0.75$ [$\gamma = 0.9999$]; *N*-acetylmethamphetamine: $W = 134.21 \times A_p/A_{IS} - 0.86$ [$\gamma = 0.9991$]; *N*-propionylmethamphetamine: $W = 122.38 \times A_p/A_{IS} - 0.86$ [$\gamma = 0.9983$]; *N*-formylmethamphetamine: $W = 84.4 \times A_p/A_{IS} + 5.02$ [$\gamma = 0.9914$]; *N*-cyanomethylmethamphetamine: $W = 140.3 \times A_p/A_{IS} + 2.93$ [$\gamma = 0.9968$]).

Synthesis of Pyrolysis Products

N-Cyanomethyl-*N*-Methylphenethylamine—This compound was synthesized from 100 mg of *N*-methylphenethylamine by the procedure described in Ref 8. The product was purified with silica gel chromatography to produce 90 mg of yellow oil.

Precise MS (M^+): observed, 174.1160; calculated, 174.1156. EI-MS (*m/z*): 83 ($M^+ - 91$, 100%) and 91 (benzyl, 5%). FT-IR (sandwiched with KBr tablets): 2230 cm^{-1} (cyano). PMR [deuteriochloroform (CDCl_3), δ ppm]: 7.23 (multiplet, phenyl H), 3.54 (singlet, NCH_2CN), 2.75 (singlet, Ar-CH_2 , $\text{N-CH}_2\text{-CH}_2$), 2.41 (singlet, N-CH_3). CMR (CDCl_3 , δ ppm): 139.2, 128.6, 128.5, 126.4 (phenyl C), 114.4 ($-\text{CN}$), 57.3 ($\text{N-CH}_2\text{-CH}_2$), 45.3 (N-CH_3), 42.0 ($\text{N-CH}_2\text{-CN}$), 34.1 (Ar-CH_2).

N-Cyanomethyl-*N*-Ethylbenzylamine—This compound was synthesized from 100 mg of ethylbenzylamine by the method described in Ref 8 to produce 85 mg of pale yellow oil.

Precise MS (M^+): observed, 174.1159; calculated, 174.1156. EI-MS (m/z): 91 (benzyl, 100%), 159 ($M^+ - 15$, 20%), 174 (M^+ , 5%). FT-IR (sandwiched with KBr tablets): 2230 cm^{-1} (cyano). PMR (CDCl_3 , δ ppm): 7.26 (multiplet, phenyl H), 3.59 (singlet, $N\text{-CH}_2\text{CN}$), 3.38 (singlet, Ar-CH_2), 2.60 (quartet, $-\text{CH}_2\text{-CH}_3$), 1.09 (triplet, $-\text{CH}_3$). CMR (CDCl_3 , δ ppm): 137.2, 128.9, 128.5, 127.6 (phenyl C), 114.7 ($-\text{CN}$), 58.1 ($-\text{CH}_2\text{-CH}_3$), 48.2, 40.6 (other CH_2), 12.7 ($-\text{CH}_3$).

Results and Discussion

After the collection of smoke in the smoking apparatus from 35-mL puffs for 2 s with 60- or 30-s intervals, the sample solutions were analyzed as described earlier. Table 1 shows the molar percentages of each pyrolysis product in the tar and butt in relation to the methamphetamine added. The methamphetamine transferred into the mainstream smoke tar was 10.5% for the 60-s interval puffing, whereas it increased to 14.5% for the 30-s interval puffing. But no difference in the methamphetamine amount in the sidestream smoke tar was found between the two types of puffing.

With the 30-s interval puffing, the mainstream smoke tar contained the pyrolysis products from methamphetamine in a range of 0.36 to 3.1% of the methamphetamine added. These products at the 30-s intervals were maximally twice as much as those at the 60-s intervals.

No *trans*- β -methylstyrene and only a trace of phenylacetone were found in the sidestream smoke, and the *N*-cyanomethylmethamphetamine amount was very much lower in the sidestream smoke than in the mainstream smoke.

The pyrolysis products and methamphetamine in the butt were similar in composition to those in the mainstream smoke tar, although the amounts in the butt were substantially smaller than those in the mainstream smoke tar.

The sum totals of the pyrolysis products in the tar and in the butt were 34 and 39% of the methamphetamine added at the 60- and 30-s interval puffings, respectively.

The pyrolysis products which had been passed through filter paper and trapped in ether were examined. The amounts of benzene and toluene detected increased in pro-

TABLE 1—The pyrolysis products of methamphetamine detected in smoke tar and cigarette butts.^a

Fraction	Compound, molar % ^b							
	MA	PhA	CMMA	FrMA	t β MS	AcMA	DMA	PrMA
60-s Interval								
Mainstream smoke	10.52	2.40	0.96	1.00	0.96	0.49	0.51	0.29
Sidestream smoke	10.71	...	0.21	0.54	...	0.50	0.15	0.22
Butt	2.27	0.60	0.36	0.24	0.23	0.09	0.48	0.09
Total	23.50	3.00	1.53	1.78	1.19	1.07	1.14	0.60
30-s Interval								
Mainstream smoke	14.50	3.06	1.94	1.58	1.73	0.73	0.64	0.36
Sidestream smoke	10.21	...	0.13	1.48	...	0.26	0.15	0.19
Butt	2.62	0.70	0.41	0.30	0.16	0.15	0.24	0.07
Total	27.23	3.76	2.48	3.46	1.89	1.14	1.03	0.62

^aThe abbreviations are as follows: MA, methamphetamine; PhA, phenylacetone; CMMA, *N*-cyanomethylmethamphetamine; FrMA, *N*-formylmethamphetamine; t β MS, *trans*- β -methylstyrene; AcMA, *N*-acetylmethamphetamine; DMA, dimethylamphetamine; and PrMA, *N*-propionylmethamphetamine.

^bThe values are the molar percentages of each pyrolysis product in relation to the methamphetamine added into a cigarette.

portion to the addition of methamphetamine into the cigarette, although they were produced solely by smoking the cigarette. Ethylbenzene, styrene, allylbenzene, and *cis*- and *trans*- β -methylstyrene were found in the ether solution as pyrolysis products of methamphetamine.

To investigate the formation of pyrolysis products from methamphetamine, methamphetamine was mixed with cigarette components and these sample were heated in a glass tube. When methamphetamine hydrochloride alone was heated in air, phenylacetone, dimethylamphetamine, dibenzyl, and *N*-formylmethamphetamine were found in the smoked tar (Fig. 2a). The amounts of these products in the tar increased when methamphetamine was heated with cigarette paper, as shown in Fig. 2b. However, no *N*-cyanomethylmethamphetamine was detected in the tar by GC/MS under this condition. Figure 2c shows the gas chromatogram of the tar produced by heating methamphetamine with tobacco. Although, in the chromatogram, *N*-cyanomethylmethamphetamine was not

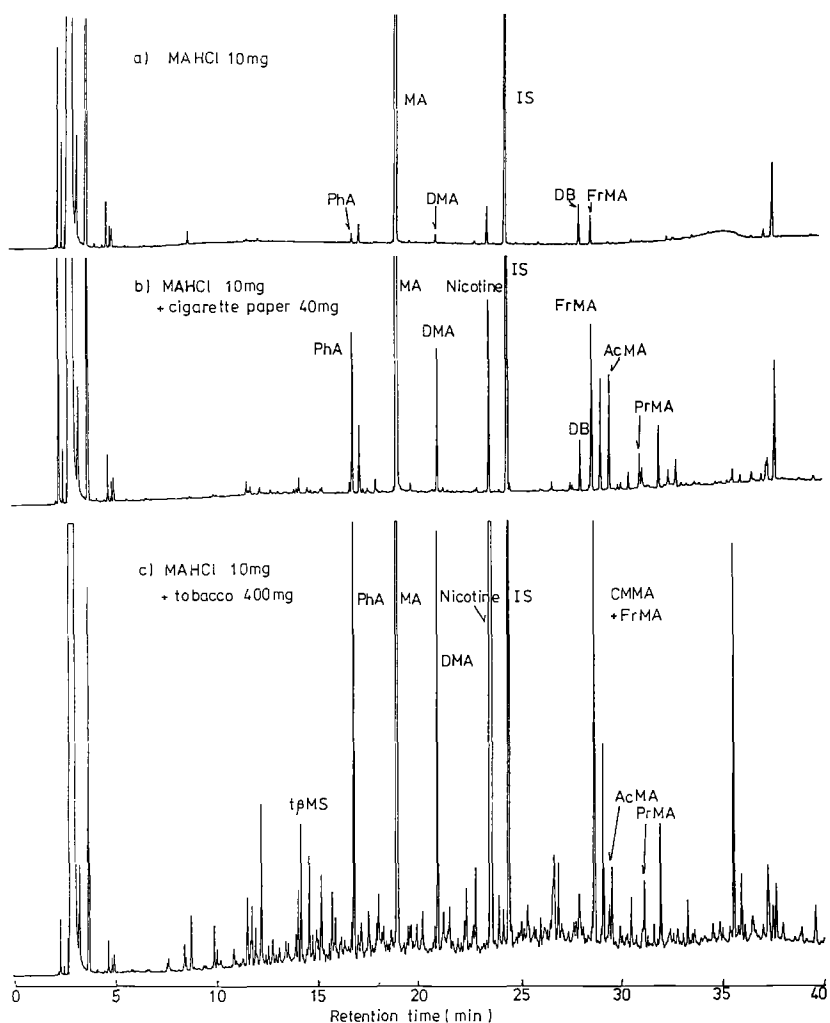


FIG. 2—Gas chromatograms of the tar produced by combustion of methamphetamine combined with cigarette components. (IS indicates biphenyl as the internal standard; DB indicates dibenzyl. The other abbreviations are defined in Footnote a of Table 1.)

separated from *N*-formylmethamphetamine, GC/MS analysis could detect the former in a much greater amount than the latter.

It is known that mainstream smoke results from heating during puffing, which produces a temperature higher than 450°C, while the sidestream smoke is mainly produced when the cigarette is smoldering between puffs and is at a temperature lower than 450°C [10,11].

Using a JHP-3 Curie point pyrolyzer, the formation of the pyrolysis products from methamphetamine was measured at various temperatures.

Figure 3 shows typical pyrograms of methamphetamine at 358, 590, and 764°C under helium. Dimethylamphetamine was formed over the whole range between 358 and 764°C. Benzene, toluene, and styrene were not found in the pyrogram at 358°C but appeared at the higher pyrolysis temperature. *N*-Acetylmethamphetamine and *N*-propionylmethamphetamine were barely produced at 764°C.

The pyrograms of methamphetamine mixed with tobacco at 358, 590, and 764°C under helium are shown in Fig. 4. *N*-Formylmethamphetamine and *N*-acetylmethamphetamine were found even at 358°C, and these compounds were produced more easily when methamphetamine was mixed with tobacco. These results indicate that methamphetamine was acylated chiefly by the products derived from tobacco. Contrary to the results of the smoking experiments, no *N*-cyanomethylmethamphetamine was found in these pyrolysis experiments.

Further pyrolysis experiments were designed so that a JHP-2 pyrolyzer could be used under conditions in which pyrolysis could be carried out in air. As shown in Table 2, air accelerated the decomposition of methamphetamine. The temperatures at the appearance of pyrolysis products were lower in air than in helium. The amounts of products produced in air were all higher, except for dimethylamphetamine.

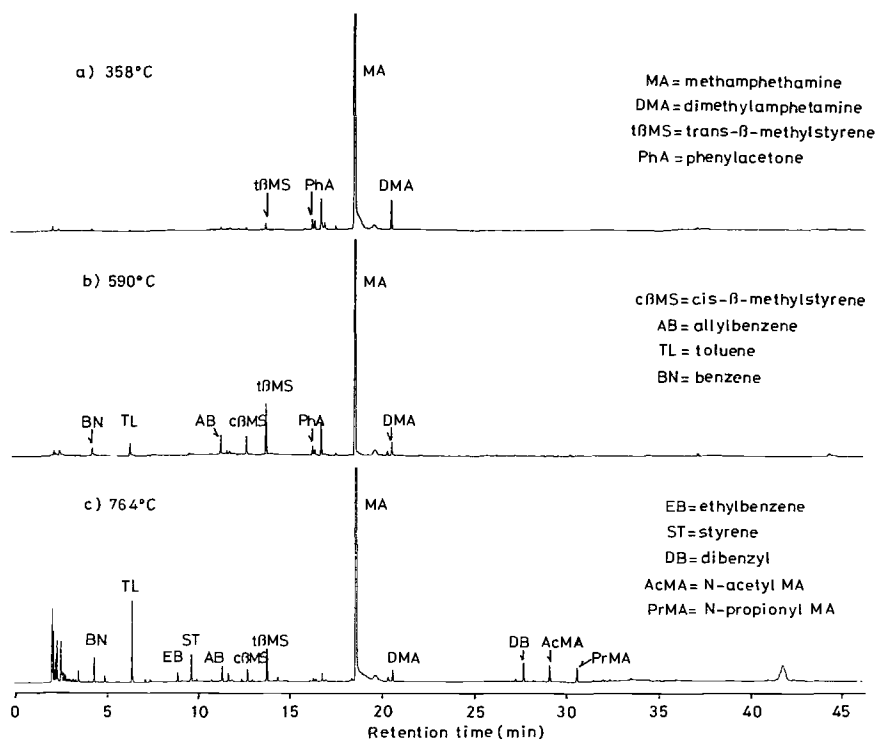


FIG. 3—Typical pyrograms of methamphetamine under helium at a pyrolysis temperature range of 358 to 764°C.

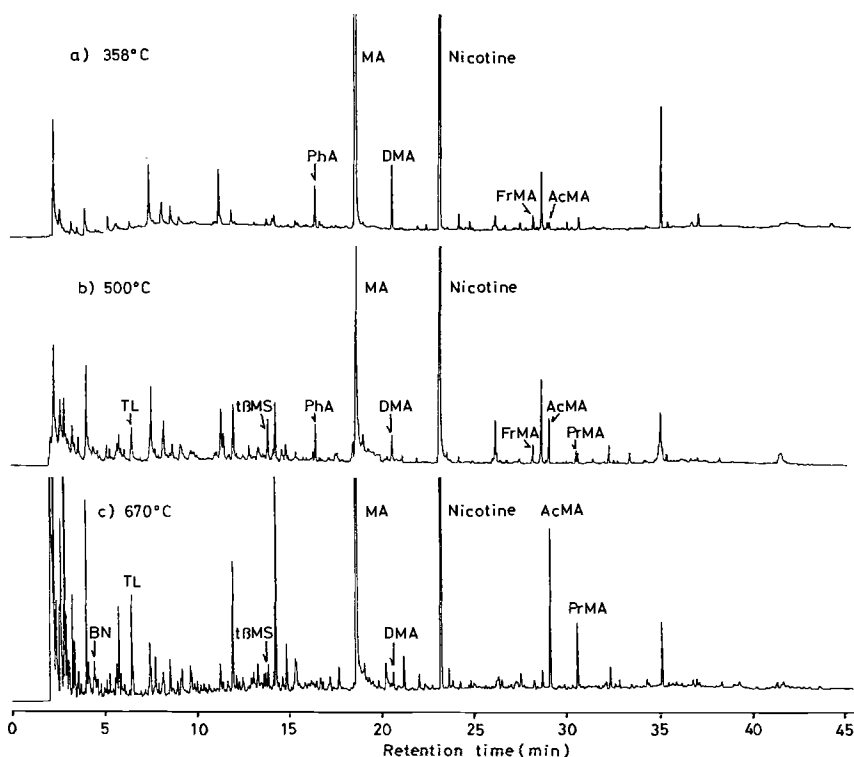


FIG. 4—Typical pyrograms of methamphetamine mixed with tobacco under helium. (The abbreviations are defined in Fig. 3. FrMA stands for *N*-formylmethamphetamine.)

Figure 5a and b shows the pyrograms of methamphetamine combined with tobacco in helium and in air, respectively. Although no *N*-cyanomethylmethamphetamine was found after pyrolysis at 670°C in helium, it was produced by pyrolysis at the same temperature in air (Fig. 5b). Moreover, all peaks of *N*-formylmethamphetamine, *N*-acetylmethamphetamine, and *N*-propionylmethamphetamine in Fig. 5b were appreciably higher than those in Fig. 5a. However, even when pyrolysis was carried out in air, no *N*-cyanomethylmethamphetamine was found at 358°C. These results indicate that tobacco reacts with methamphetamine at a temperature as high as 670°C to yield *N*-cyanomethylmethamphetamine.

All the pyrolysis products, except *N*-cyanomethylmethamphetamine, were formed by the heating of methamphetamine and cigarette components (that is, cigarette paper and tobacco) in the absence of air, whereas air, tobacco, and a high-pyrolysis temperature were required for the formation of *N*-cyanomethylmethamphetamine. This finding was in agreement with the result of the smoking experiment, which showed that *N*-cyanomethylmethamphetamine was found much more often in the tar of mainstream smoke, which formed at a higher temperature, than in the tar of sidestream smoke.

Consequently, *N*-cyanomethylation of methamphetamine seems to be a peculiar reaction occurring during combustion with tobacco, whereas the other reactions, that is, methylation, acylation, and decomposition are produced by heating alone.

To investigate whether *N*-cyanomethylation occurs only when methamphetamine mixed with tobacco is smoked, or also occurs when other drugs are smoked, the smoking experiments were carried out with *N*-methylphenethylamine HCl and *N*-ethylbenzylamine HCl.

From GC/MS analyses of the smoke tars from the tobacco mixed with these amines,

TABLE 2—The relationship between pyrolysis of methamphetamine and temperature.

Pyrolyzed Sample	Atmosphere	Temperature at the Appearance of Each Pyrolysis Product, °C ^a														
		BN	TL	EB	ST	AB	cβMS	tβMS	PhA	MA	DMA	DB	FrMA	CMMA	AcMA	PrMA
Methamphetamine	Helium	500 ↓ ^c	445	670	670	445	445	358	358	358	670	670	670	ND ^b	670	670
	Air	445	358	500	500	445	358	358	358	358	670	670	670	ND	670	670
Methamphetamine and tobacco	Helium	...	445	500	445	358	358	764	358	358	ND	358	445
	Air	445	358	445	445	358	358	358	670	358	358	670	358	445

^aThe abbreviations of the pyrolysis products are the same as Fig. 3. FrMA stands for *N*-formylmethamphetamine and CMMA stands for *N*-cyanomethylmethamphetamine.

^bND means nondetected.

^cThe arrow sign, ↓, indicates an increase of the product in the air in comparison with helium at the same temperature.

^dThe sign ... indicates that the data on the pyrolysis products are not proved because of contaminated tobacco components.

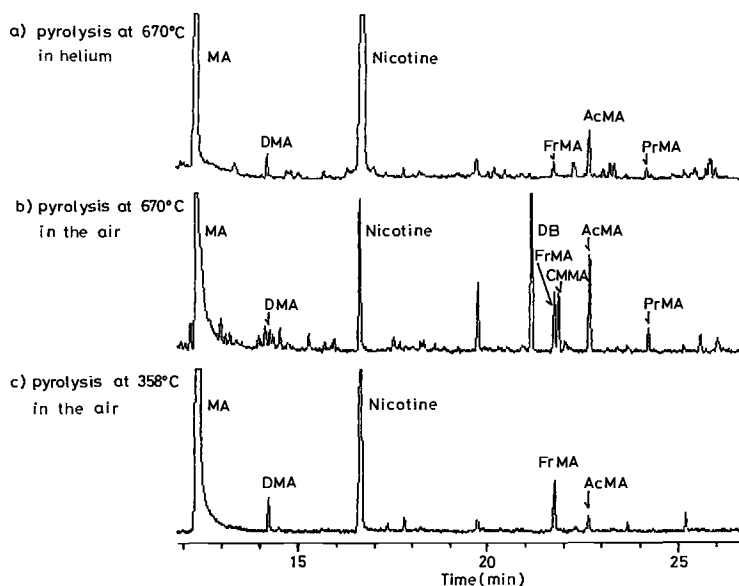


FIG. 5—A comparison of pyrograms of methamphetamine mixed with tobacco measured under various conditions.

their *N*-cyanomethylated amines, *N*-cyanomethyl-*N*-methylphenethylamine and *N*-cyanomethyl-*N*-ethylbenzylamine, were identified as pyrolysis products. Consequently, it was proved that *N*-cyanomethylation could occur from smoking some of these amines mixed with tobacco.

Conclusions

Smoking methamphetamine mixed with a cigarette transferred 10 to 15% of the initial methamphetamine from the cigarette to the mainstream smoke tar and about 10% to the sidestream smoke tar. In the mainstream smoke, phenylacetone was the largest pyrolysis product of smoking methamphetamine, and its amount was 3.1% of the methamphetamine added. *N*-Cyanomethylmethamphetamine was the second largest product formed, a 1.9% yield.

In the sidestream smoke, no *trans*- β -methylstyrene and little phenylacetone were found, and *N*-cyanomethylmethamphetamine was produced in a smaller amount in the sidestream smoke (0.21%) than in the mainstream smoke (2.4%). The total sum of pyrolysis products in the smoke tar and the butt was maximally 39% of the methamphetamine added in the cigarette. Although the other volatile pyrolysis products, benzene, toluene, styrene, and allylbenzene were not trapped on the filter paper, it was confirmed that these compounds were produced by smoking methamphetamine.

From the pyrolysis experiments performed using a pyrolyzer, it became apparent that all products except *N*-cyanomethylmethamphetamine were produced by self-decomposition of methamphetamine, and the amounts of these products were increased by the combination of methamphetamine with cigarette paper and tobacco and were further increased by combustion with these cigarette components. *N*-Cyanomethylmethamphetamine was only produced by combustion of methamphetamine with tobacco at a higher pyrolysis temperature than 690°C.

The smoking experiments on the other secondary amines indicated that *N*-cyanomethylation was not a specific reaction from smoking methamphetamine mixed with tobacco.

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