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Identification of Cresols and *o*-Dichlorobenzene in Stomach Content, Blood and Urine of a Cadaver by Gas Chromatography/Mass Spectrometry

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Introduction

The application of gas chromatography/mass spectrometry (GC/MS) to forensic toxicology has been an object of growing appreciation, because of its powerful ability to identify and quantitate toxic substances.

Recently, we have experienced, during medical examinations, a death case of a female, suspected to have died of cresol and *o*-dichlorobenzene intoxication. In the present paper, we report that cresol isomers and *o*-dichlorobenzene were successfully identified in stomach content, blood and urine of this cadaver by GC/MS. To our knowledge, this is the first report to identify them by GC/MS for a medicolegal purpose.

Materials and Methods

A 0.2 ml aliquot of stomach content, cardiac blood or urine was extracted with 2.0 ml of petroleum ether after acidification with HCl. After centrifugation at 1,500 g for 5 min, the organic layer was transferred to a test tube, mixed with 0.5 g of Na₂SO₄, and shaken with a Vortex-type mixer in order to secure complete dehydration. A 3.0 μ l aliquot of the organic layer was routinely injected into the port of gas chromatograph of GC/MS.

The GC separation was made on a 2.0 m \times 2 mm glass column packed with PEG-HT + KOH (5+1) % on 60/80 mesh Uniport HP (Gasukuro Kogyo Co., Ltd., Tokyo). The GC conditions were: injection temperature 200°C, column temperature 180°C and helium flow rate 30 ml/min. The analyses were carried out on a JEOL D-300 GC/MS instrument equipped with a computer-controlled data analysis system. The MS conditions were: electron energy 70 eV, separator temperature 200°C and ion source temperature 175°C.

The authentic *o*-, *p*- and *m*-cresol, and *o*-dichlorobenzene were purchased from Tokyo Kasei Kogyo Co., Ltd., Tokyo.

Results and Discussion

A 53-year-old female was found dead by her husband at her house on May 28, 1980. When we performed medical examination, there was a cresol-like odor around the cadaver. Interestingly, no rigor mortis could be observed at any muscle of the body, despite the time-lapse of about 4 h estimated by lividity and rectum temperature. In her open mouth, bubbling saliva was observed. Stomach content, cardiac blood and urine were obtained from the cadaver by drawing with a long needle syringe. Autopsy was not carried out, because no criminality was suspected; there was a letter indicating a suicidal intent. A 500 ml bottle of a disinfectant insecticide, about four-fifth of which

was empty, was discovered later. Its label showed the composition of 7.5% cresol and 72.5% *o*-dichlorobenzene. Therefore, this liquid was strongly suspected to be a poison which caused her death.

In order to identify the suspected materials, the mass spectra were measured for the authentic cresols and *o*-dichlorobenzene. The authentic *o*-, *p*- and *m*-cresol gave similar mass spectra; peaks at m/z 107 and 108 were highest, followed by peaks at m/z 79, 77 and 90. The authentic *o*-dichlorobenzene resulted in characteristic peaks at m/z 75, 111, 113, 146, 148 and 150; the peak at m/z 146 was the base peak. The retention times of the authentic *o*-, *p*-, *m*-cresol and *o*-dichlorobenzene were 4.7, 7.1, 9.0 and 0.6 min, respectively.

On the basis of the above data, mass chromatogram was determined on the extracts of stomach content, blood and urine using ten different ions. The chromatogram obtained from urine extract is presented in Fig. 1; the results obtained from stomach content and blood (data not shown) were essentially the same as those of urine extract. As shown in the figure, the presence of the four compounds in urine extract was apparent.

Mass spectra which correspond to each peak, observed in the mass chromatogram, were measured with the extracts of stomach content, blood and urine. The results on urine extract are presented in Fig. 2. The patterns for stomach content and blood (data not shown) were extremely similar to those for urine; the mass spectra obtained from the three cadaveric samples were almost the same as those of the corresponding authentic samples. However, a few peaks such as that found at m/z 94 shown in Fig. 2C were noted; they are probably derived from impurities. All these spectral data give a substan-

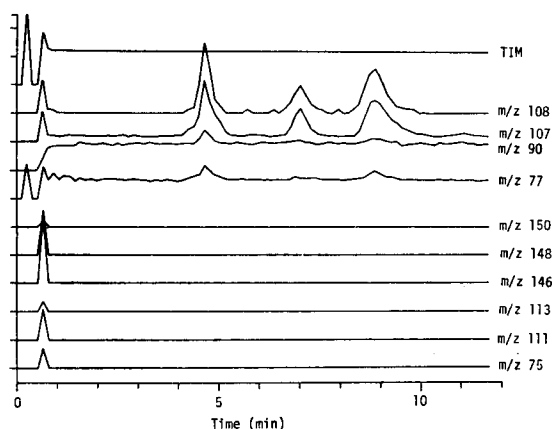


Fig. 1. Mass chromatogram of urine extract obtained from a dead body of a 53-year-old female. The relative sensitivities of ions at m/z 108, 107, 90, 77, 150, 148, 146, 113, 111, and 75 were 1.0, 0.85, 0.56, 0.48, 1.06, 0.64, 0.98, 0.14, 0.43 and 0.28, respectively.

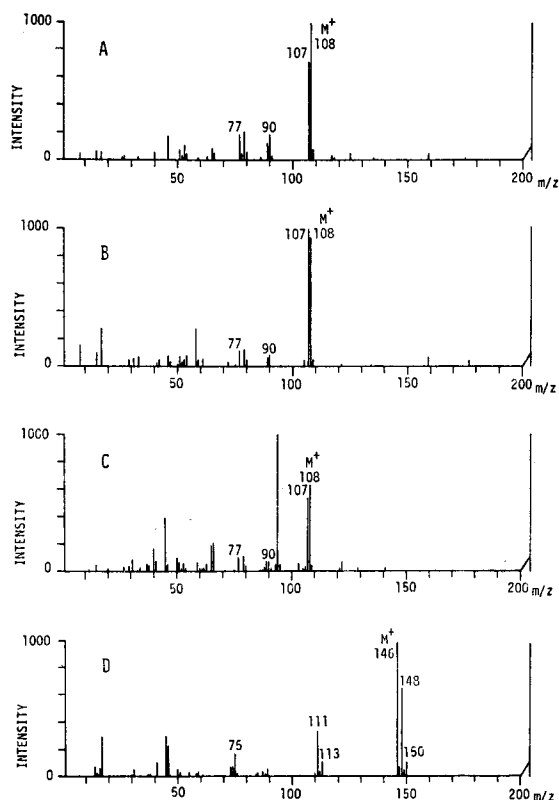


Fig. 2. Mass spectra of *o*-(A), *p*-(B), *m*-cresol (C) and *o*-dichlorobenzene (D) extracted from urine obtained from a dead body of a 53-year-old female.

Table 1. Concentrations of cresols and *o*-dichlorobenzene detected in stomach content, blood and urine sampled from a cadaver of a 53-year-old female

Sample	Concentration ($\mu\text{g/ml}$)			
	<i>o</i> -Cresol	<i>p</i> -Cresol	<i>m</i> -Cresol	<i>o</i> -Dichlorobenzene
Stomach content	861	594	1163	40
Blood	7.0	4.3	7.6	15
Urine	6.5	3.1	6.6	20

tial confirmation of the presence of *o*-, *p*-, *m*-cresol and *o*-dichlorobenzene in stomach content, blood and urine, sampled in the present case of medical examination.

In the mass spectra (Fig. 2) of *o*-, and *m*-cresol, the molecular ion at m/z 108 was the base peak, but in those of *p*-cresol, the peak at m/z 107 ($M-1$) was highest. This peak is probably produced by the release of a proton from the benzyl-position. The peak at m/z 90 ($M-18$) observed for *o*-cresol was higher than the corresponding peaks of *p*- and *m*-cresol. This is due to the "ortho-effect", which induces the subtraction of H_2O from the cresol molecule.

In the spectra of *o*-dichlorobenzene, the peak at m/z 146 is the molecular and base peak (Fig. 2D). Notable peaks were found at m/z 148 ($M+2$) and 150 ($M+4$). The peak height ratio at m/z 146, 148 and 150 was approximately 9/6/1, which is characteristic for the presence of two chloric groups. The peak at m/z 111 ($M-35$) is explained by the release of one chlorine; the isotopic peak was also detected at m/z 113 with the relative peak height ratio of about 0.3. The peak at m/z 75 ($M-71$) is probably formed by the abstraction of two chlorines.

To quantitate cresols and *o*-dichlorobenzene in stomach content, blood and urine, the selected ion monitoring, using ions at m/z 108 for cresols and at m/z 146 for *o*-dichlorobenzene, was performed. The calibration curves were made by injecting known amounts of the authentic samples. The results are shown in Table 1. The concentrations of cresols in stomach content were extremely higher than those in blood or urine, while the levels of *o*-dichlorobenzene in blood and urine were in the same order of magnitude as that in stomach content. These results suggest that the absorption of *o*-dichlorobenzene is quicker than that of cresols.

The LD_{50} of cresols in animals, when administered orally, was reported to be about 0.5 g/kg.¹⁾ According to the Merck Index, when administered orally to human, 8 g or more produces rapid circulatory collapse and death. Although the total amount of stomach content could not be measured, it is easily suspected from the high levels of cresols in stomach content (Table 1) that she ingested a sufficient amount of cresols to die.

The lethal dose of *o*-dichlorobenzene is reported to be 25 g in human.²⁾ Therefore, its toxicity seems comparable to that of cresols. Thus, *o*-dichlorobenzene is also suspected to have competed with cresols for her death.

Summary

We experienced a death case of a 53-year-old woman, suspected to have died of cresol and *o*-dichlorobenzene intoxication. The identification of cresol isomers and *o*-dichlorobenzene in her stomach content, blood and urine was successfully performed by GC/MS.

References

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死体の胃内容物，血液ならびに尿におけるクレゾールと o-ジクロロベンゼンの GC/MS による同定

服部 秀樹・鈴木 修・浅野 稔
はつとり ひでき すずき おさむ あさの みのる

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われわれは，クレゾールならびにo-ジクロロベンゼン
を含有した殺虫消毒剤を服用して自殺したと疑われる一
検死例を経験した。胃内容物，心臓血ならびに尿を採取
し，石油エーテルで抽出し，GC/MS 機を用いマススペ

クトルならびにマスキロマトグラムを測定したところ，
首尾よくクレゾールならびにo-ジクロロベンゼンを同定
することができた。
