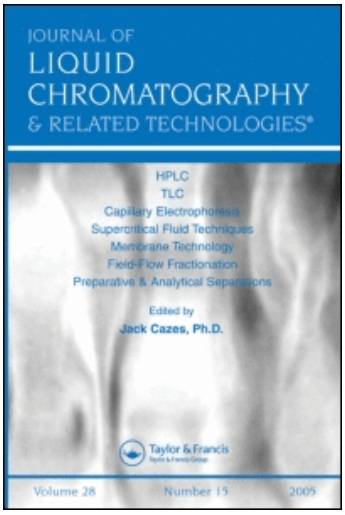

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

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To cite this Article Risner, Charles H.(1993) 'The Quantification of Hydroquinone, Catechol, Phenol, 3-Methylcatechol, Scopoletin, *m p*-Cresol and *o*-Cresol in Indoor Air Samples by High-Performance Liquid Chromatography', Journal of Liquid Chromatography & Related Technologies, 16: 18, 4117 — 4140
To link to this Article: DOI: 10.1080/10826079308019691
URL: http://dx.doi.org/10.1080/10826079308019691

JOURNAL OF LIQUID CHROMATOGRAPHY, 16(18), 4117-4140 (1993)

THE QUANTIFICATION OF HYDROQUINONE, CATECHOL, PHENOL, 3-METHYLCATECHOL, SCOPOLETIN, *m*+*p*-CRESOL AND *o*-CRESOL IN INDOOR AIR SAMPLES BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

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ABSTRACT

A high performance liquid chromatography (HPLC) method was developed for the quantification of the phenolic compounds hydroquinone, catechol, phenol, 3-methylcatechol, scopoletin, *m*+*p*-cresol and *o*-cresol in indoor air samples. Samples are collected on an 0.8 μm pore size mixed cellulose ester membrane (MCEM) followed by a silica gel Sep-Pak (SiOHSP). The MCEM is extracted and the SiOHSP is eluted with 1% acetic acid (HAc). The phenolic compounds were analyzed on a reverse-phase column with fluorescence detection at selected excitation and emission wavelengths specific to the compounds of interest. A mobile phase gradient of 1% HAc and 99% acetonitrile (ACN) + 1% HAc is used. The method is reproducible with percent relative standard deviations (%RSD) ranging from 2.0 to 9.2 for the seven phenolic compounds. Percent recoveries are acceptable (88-98) with the exception of scopoletin (52) and *p*-cresol (52). A comparison of tobacco versus wood smoke show that amounts of these seven phenolic compounds vary widely with their source. A relatively short sampling time (1-4 hours) is required and the procedure is capable of detecting <0.3 μg m^{-3} for all compounds with the exception of 3-methylcatechol with a detection limit of < 4.0 μg m^{-3}.

INTRODUCTION

An earlier method for the determination of phenolic compounds in indoor air, although sufficient for analyte quantification, suffers from limitations due to glass impinger collection (1). The impinger limits the sample flow collection to *ca.* 1 L min^{-1}, thereby limiting the actual sample size processed during 1 h, a time limit that has been imposed in some studies in this laboratory. Under such restrictions, the analytes for many air samples give a small response in the range of the detection limit. The impinger is cumbersome and requires liquid handling; these are not assets to field applications.

Solid phase collection devices have been used previously for the collection of phenolic compounds. NIOSH method 2001 employs silica gel sorbent tubes for collection of *o, m, p*-cresols (2). XAD-7 resin contained in sorbent tubes collects not only these cresols but also phenol (3). Both silica gel and XAD-7 are recommended for use at low sampling flow rates (0.01 to 0.2 L min^{-1} for silica gel and 0.1 L min for the XAD-7) and may not collect a sufficient amount of sample when used for short durations (1-4 h). These commercially available devices are supplied in sealed glass tubes which hinders additional cleanup of the solid phase if it is to be used for the determination of low levels of these compounds.

This paper describes a HPLC method for the determination of hydroquinone, catechol, phenol, 3-methylcatechol, scopoletin, *m*+*p*-cresol and *o*-cresol in indoor

air. The analytes hydroquinone, catechol, 3-methylcatechol and scopoletin are collected on a mixed cellulose ester membrane (MCEM). Phenol and the cresols are collected by means of a pretreated silica cartridge following the MCEM. The MCEM is extracted separately and the silica gel cartridge eluted separately with one percent acetic acid and an aliquot of each is subjected to gradient, reverse-phase chromatography with selective fluorescence detection. To this author's knowledge, this is the first reported procedure for the determination of 3-methylcatechol and scopoletin in indoor air and environmental tobacco smoke (ETS).

MATERIALS AND METHODS

Equipment

HPLC. The HPLC system consisted of two ABI Spectroflow 400 pumps (Applied Biosystems, Inc., Foster City, CA, USA), an 878A autosampler fitted with a 200-μL sample loop and a Perkin-Elmer LS-4 Fluorescence Spectrometer (Perkin-Elmer Corp., Norwalk, CT, USA). The pumps and the autosampler were controlled by a DS650 Data System and data are acquired on VAX MULTICHROM (VG Instruments, Danvers, MA, USA). Separations were accomplished on a Beckman Ultrasphere ODS, 4.6 X 250 mm, 5 μm particle size column (Beckman, San Ramon, CA, USA). A 30 X 4.6 mm Brownlee RP-18 Spheri-5 MPLC guard refill in a Brownlee 3-cm MPLC holder (Brownlee Labs, Inc., Santa Clara, CA, USA) was placed directly before the analytical column.

Sampling. The sampling train consisted of one mixed cellulose ester membrane (MCEM) 37 mm, 0.80 or 0.45 μm pore size (SKC Inc., Eighty Four, PA, USA) secured with a 37-mm gasket (Sloan Valve Co., Franklin Park, IL, USA) in cassettes (SKC Inc., Eighty Four, PA, USA). The MCEM was connected by a nylon adaptor (SKC Inc., Eighty Four, PA, USA) and a 4-cm length of 0.635 cm I.D. tubing to a pretreated silica Sep-Pak (SiOHSP), (Waters Chromatography Division Millipore Corp., Milford, MA, USA). These two coupled devices were connected to a Dawson High Volume Air Sampler (Dawson Assoc., Inc., Lawrenceville, GA, USA) with 0.635 cm I.D. tubing.

Chemicals

Hydroquinone, catechol, phenol, 3-methylcatechol, scopoletin, *p*-cresol and *o*-cresol were obtained from Aldrich (Milwaukee, WI, USA). Acetonitrile (ACN), "distilled in glass", was obtained from Burdick and Jackson (Muskegon, MI, USA). Acetic acid (HAc) was reagent grade (Reagents, Inc., Charlotte, NC, USA).

Water was obtained from a Nanopure system, which consisted of a carbon cartridge, two high-capacity mixed ion exchange cartridges and a 0.45 μm filter (Barnstead Co., Div. of Sybron Corp., Dubuque, IA, USA).

Preparation of Standard Solutions

Stock solutions of the seven phenolic compounds were prepared in 1% HAc and diluted to the appropriate concentration with same.

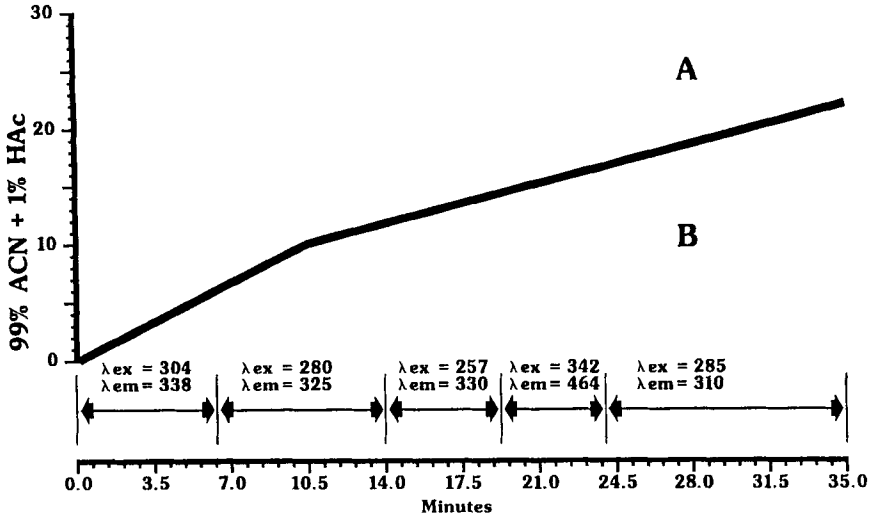

FIGURE 1. Gradient and wavelength-time program: (A) gradient profile; (B) wavelength-time program.

Procedure

All chromatographic separations were performed at room temperature with a mobile phase gradient of one percent HAc and 99 percent ACN plus one percent HAc at a flow rate of 2.0 mL min^{-1}. Figure 1 shows the mobile phase gradient (A) and wavelength-time program (B) used over the chromatographic region of interest. The gradient also includes a five-minute 99 percent ACN plus one percent HAc wash and a 12-minute equilibrium delay prior to the next injection. Results are obtained by means of an external standard procedure and a complete analysis takes *ca.*. one hour.

Silica Sep-Pak Pretreatment. The SiOHSP cartridge was rinsed with 10 ml methylene chloride, dried with helium at 5 L min^{-1}, put in 4-mL vials and stored at -4°C prior to use.

Sampling and Sample Preparation. The air sample collection apparatus was precalibrated with a soap film flow meter (The Gilibrator, Gilian Instrument Corp., Wayne, NJ, USA) at *ca.* 2 L min^{-1} prior to sample collection. After sampling was complete (*ca.* 1 h) the flow was rechecked. Following use, the cassette was disassembled and the side of the MCEM facing the air flow was placed toward the bottom of a 25-ml Erlenmyer flask. One percent HAc (5 ml) was pipetted into the flask which was then stoppered and sonicated 10 min (Ultrasonic Cleaner, Cole-Parmer Instrument Co., Chicago, IL, USA). The SiOHSP was eluted with one percent HAc to a volume of 5 ml.

RESULTS AND DISCUSSION

Devices Evaluated

Table 1 lists the devices evaluated in this work, sampling conditions and the amount in μg m^{-3} of catechol and phenol collected. The results from these devices were compared to those obtained using the original collection system of an MCEM followed by a 25-mL impinger (SKC Inc.,Eighty Four, PA, USA) (1). The following solid phase collection devices were evaluated: Orbo 47 tubes (XAD-7, Supelco Inc.,Bellefonte, PA, USA) and Diol, CN, NH_2 and SiOH Sep-Paks (Waters Chromatography Division, Millipore Corp.,Milford, MA, USA).

Table 1.
Devices Evaluated

Device	Sampling Conditions (38-m^3 room)			$\mu g\ m^{-3}$ Catechol	$\mu g\ m^{-3}$ Phenol
	No. Cigt.	Vol (L)	Flowrate (L min^{-1})	Sample	Sample
MCEM + Impinger	8	60	1.0	1.6	6.2[a]
Orbo 47	8	60	1.0	BDL	0.6
Diol Sep-Pak	8	60	1.0	BDL	4.8
CN Sep-Pak	8	60	1.0	0.6	4.7
NH_2 Sep-Pak	8	59	1.0	BDL	7.4
SiOH Sep-Pak	8	60	1.0	0.5	7.6

BDL = below detection limits at two times signal to noise ratio
[a] - 2.2 $\mu g\ m^{-3}$ from MCEM, 4.0 $\mu g\ m^{-3}$ from impinger

All appeared to collect phenol (see Table 1), but the Orbo 47 tube, CN and Diol Sep-Paks did not collect as much phenol as the other solid phase devices and their use was abandoned. The NH_2 Sep-Pak, although efficient for collection of phenol, appeared to be inefficient or possibly detrimental to catechol collection due to its basicity.

The SiOH Sep-Pak (SiOHSP) collected at least as much phenol as the MCEM and impinger combined and had an affinity for catechol. Since the solid phase device would be placed after the MCEM, which was most efficient in collecting catechol, the SiOHSP was chosen to ensure complete collection of catechol should breakthrough of the MCEM occur.

Excitation and Emission Wavelength Change for Phenol

In many samples, the amount of phenol collected on the SiOHSP was sufficient to saturate the fluorescence detector when the optimal excitation and emission wavelengths for phenol, 274 and 298 nm, respectively were used (1,4). Since the data acquisition program scales to the largest peak, smaller concentration components gave less response. To alleviate this problem, the wavelength program in the region of phenol was changed from its most sensitive setting, to 257 nm excitation, 330 nm emission. This produced a larger response for the lower concentration components by enabling the sensitivity to be increased two fold while still retaining phenol in the concentration range of the samples.

Compounds Evaluated

Upon performing breakthrough experiments using the sampling conditions shown in Table 1, eight cigarettes smoked in a 38-m^3 room over one hour at a sampling rate of 1 L min^{-1}, the results showed that more cigarettes and a longer sampling time had to be used in order to obtain detectable quantities for evaluating breakthrough onto the second device. Using the maximum flow that could be attained with the SiOHSP, 1.7 L min^{-1}, an insufficient amount of analytes is captured from the ETS of eight cigarettes. To remedy this situation, more cigarettes were smoked and a longer sampling time (four hours) was used. Under these extreme conditions additional unknown compounds were observed. Efforts were made to identify these unknown compounds. Table 2 lists the compounds evaluated.

Table 2.
Compounds Evaluated

Compound	Concentration μg/ml	λex, nm	λem, nm	Retention Time, min.
resorcinol	0.230	280	325	8.24
methylhydroquinone	0.52	293	335	12.6
guaiacol	0.26	281	314	21.8
scopoletin	0.035	342	464	23.2
3-methylcatechol	3.02	257	330	18.6
2,6-dimethoxyphenol[a]	4.24	257	330	no response
4-methylcatechol[a]	3.18	257	330	16.6
4-ethylphenol[a]	3.38	257	330	no response
4-ethylcatechol[a]	3.60	257	330	28.4[b]

[a] - reference 5
[b] - retention time of *o*-cresol

Resorcinol, although detected in mainstream tobacco smoke (4), was not observed under these conditions. In addition to the five compounds analyzed in previous work (1), methylhydroquinone, 3-methylcatechol, guaiacol and scopoletin were observed in the air samples collected while smoking 16 cigarettes in a 38-m^3 room with a sampling time of four hours at *ca.* 1.7 L min^{-1}. Guaiacol in air samples gave a response which was less than two times the signal to noise ratio even at its selective wavelengths. For this reason, no effort was made to analyze this compound. Methylhydroquinone, although giving a sufficient signal, appeared to decompose with time in both the sample extract and standard. The

methylhydroquinone standard also contained many impurities. These problems made it impractical to analyze for this compound. The compounds left to analyze under these conditions were: hydroquinone, catechol, phenol, 3-methylcatechol, scopoletin, *m*+*p*-cresol and *o*-cresol. Since 3-methylcatechol was observed in ETS-containing samples and has not been reported previously, standards were obtained from two sources in order to further verify its presence. It was dissolved in both ACN and one percent HAc as a stock standard prior to dilution in one percent HAc to evaluate solubility. In addition, other compounds found in tobacco smoke which have similar retention times according to Ishiguro *et al.* (5) were also evaluated. These compounds are listed in Table 2. These compounds were evaluated at about the same concentration level and wavelengths of detection as 3-methylcatechol. As can be seen in Table 2, none of these compounds under these conditions would be mistaken for 3-methylcatechol. This supports the tentative identification of 3-methylcatechol in ETS.

SiOHSP Preparation and Elution

Another objective of this work, besides the elimination of impinger collection, was to decrease the number of samples by using the eluent of the solid phase device to extract the MCEM. Hydroquinone and catechol deteriorated within one week when the 5 ml eluent of one percent HAc from the SiOHSP was used to extract the MCEM. Since sidestream cigarette smoke is basic, it was decided to prepare the SiOHSP by using one percent HAc instead of methylene chloride. This did not remedy the problem and there was no difference in the amounts of hydroquinone and catechol lost when the eluent of the SiOHSP (after being

pretreated with one percent HAc or methylene chloride) was used to extract the MCEM. Separate extraction of the MCEM and elution of the SiOHSP gave higher results. The cassettes were foil covered in an effort to supress possible photodegradation of hydroquinone and catechol. This did not increase the amount collected. On the basis of these results, it was decided to extract the MCEM and elute the SiOHSP separately. The preparation of the SiOHSP, washing with methylene chloride, a routine cleanup preparation for normal stationary phases, remained the same.

The amount of eluent used to remove the phenolic compounds was evaluated. The use of two milliliters proved insufficient for the elution of phenol and *m*+*p*-cresol from the SiOHSP. Five milliliters completely removed these compounds. A second elution with five milliliters gave results which were below detection limits.

Collection Flow Dependence

Table 3 gives results for the compounds evaluated at two different collection flows. All compounds appear to be independent of flow within the range evaluated, 0.95 to 1.73 L min^{-1}. Comparable values in $\mu g\ m^{-3}$ are obtained.

Breakthrough/Collection Efficiency

Table 4 gives the results obtained in μg when using combinations of the two collection devices. Hydroquinone, catechol, 3-methylcatechol and scopoletin are

Table 3.
Collection Flow Dependence (n=2)[a]

Compound	μg Collected	μg m^{-3}
hydroquinone	0.35 0.78	1.54[b] 1.89[c]
catechol	0.28 0.47	1.22 1.13
phenol	1.46 2.62	6.40 6.31
3-methylcatechol	11.65 20.64	51.0 49.7
scopoletin	0.056 0.098	0.245 0.236
m+*p*-cresol	0.28 0.48	1.22 1.16
o-cresol	0.13 0.24	0.57 0.58

[a] - 16 cigarettes smoked in a 38-m^3 room, sampled over four hours
[b] - 0.95 L min^{-1}
[c] - 1.73 L min^{-1}

collected on the MCEM while phenol, *m*+*p*-cresol and *o*-cresol are collected on both devices, but mainly on the SiOHSP (see Figure 2). These combinations indicate that a single MCEM followed by one SiOHSP is sufficient for the collection of these compounds in indoor air.

Chromatographic Precision

Table 5 shows the chromatographic precision of eight phenolic compounds found in ETS. Methylhydroquinone, being unstable (see Compounds Evaluated), has the highest percent relative standard deviation (%RSD).

Table 4.
Breakthrough/Collection Efficiency[a]

Compound	1st MCEM (% of Total)	2nd MCEM (% of Total)	1st SiOHSP (% of Total)	2nd SiOHSP (% of Total)	**Total**
hydroquinone	0.82	---	BDL	---	0.82
	0.80	BDL	BDL	---	0.80
	0.68	---	BDL	BDL	0.68
catechol	0.52	---	BDL	---	0.52
	0.49	BDL	BDL	---	0.49
	0.52	---	BDL	BDL	0.52
phenol	0.41 (14.4)	---	2.44 (85.6)	---	2.85
	0.36 (13.2)	0.37 (13.6)	2.00 (73.2)	---	2.73
	0.60 (23.2)	---	1.98 (76.7)	BDL	2.58
3-methylcatechol	22.9	---	BDL	---	22.9
	22.8	BDL	BDL	---	22.8
	21.5	---	BDL	BDL	21.5
scopoletin	0.108	---	BDL	---	0.108
	0.096	BDL	BDL	---	0.096
	0.097	---	BDL	BDL	0.097
m+*p*-cresol	0.066 (13.0)	---	0.440 (87.0)	---	0.506
	0.056 (11.3)	0.070 (14.2)	0.368 (74.5)	---	0.494
	0.124 (24.8)	---	0.350 (70.0)	0.025 (5.2)	0.500
o-cresol	0.027 (11.0)	---	0.218 (89.0)	---	0.245
	0.021 (8.2)	0.026 (10.2)	0.208 (81.6)	---	0.255
	0.038 (16.1)	---	0.198 (83.9)	BDL	0.236

[a] - 16 cigarettes smoked in a 38-m^3 room, sampled for four hours, normalized to 408 L (240 min X 1.7 L min^{-1}),both devices extracted/eluted with 4 mL 1% acetic acid, μg
[b] - BDL = below detection limits

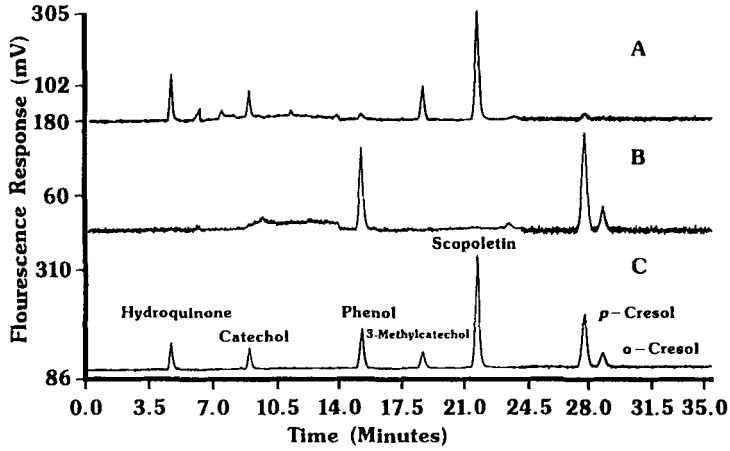

FIGURE 2. Chromatograms of ETS sampled for four hours at 2 L min^{-1} in a 38-m^3 room after smoking 16 cigarettes: (A) MCEM; (B) SiOHSP; (C) standard. Conditions: see text. Concentration of standard (ng mL^{-1}): hydroquinone (129); catechol (94); phenol (616); 3-methylcatechol (3020); scopoletin (33); *p*-cresol (117); *o*-cresol (72).

Table 5.
Chromatographic Precision (n=6)

Compound	Conc. (μg/mL)	$\bar{x}$ (peak ht.)	σ (peak ht.)	% RSD
hydroquinone	0.1288	897	17	1.9
methylhydroquinone	0.0430	67	14	20.8
catechol	0.0940	609	13	2.2
phenol	0.6160	1170	11	0.9
3-methylcatechol	3.0195	456	8	1.8
scopoletin	0.0328	3380	14	0.4
p-cresol	0.1172	1501	7	0.4
o-cresol	0.0720	368	9	2.4

Linearity of the Analytical Method

Table 6 shows all compounds to have a linear response with methylhydroquinone having the worst correlation. All intercepts are within experimental error with the exception of methylhydroquinone due to its instability.

Overall Precision

Table 7 lists the overall precision for seven compounds collected on the two different devices and the minimum detectable quantity (MDQ) by the use of both devices under the conditions specified. Note the rather large MDQ for 3-methylcatechol, a weakly fluorescing compound versus scopoletin, a very strongly fluorescing compound.

Recovery and Standard Addition

Recoveries were determined from samples having the phenolic compounds applied to the collection devices prior to sample collection (Tables 8 and 9). Table 8 shows scopoletin to have a poor recovery. Scopoletin did not dissolve readily in one percent acetic acid and since the concentration of the applied amount was questionable, it was dissolved in ACN and applied to the MCEM prior to sample collection. Upon sample collection the recovery did not improve. Scopoletin from two different sources were prepared in red glass volumetric flasks in ACN and MeOH. The solutions were applied to both an MCEM (ACN) and Teflon membrane (MeOH) and dried in the dark (incubator @ 30°C) for 20 minutes and weighed. They were left in the incubator overnight and again

Table 6.
Linearity of the Analytical Method[a]

Compound	Concentration Range, $\mu g\ m^{-3}$	Sensitivity Range[b], ng	Linear Correlation, R^2	Y-Intercept, $\mu g\ m^{-3}$
hydroquinone[c]	0.32-3.94	5.2-64.4	0.9999	-0.14
methylhydroquinone[c]	0.10-1.32	1.7-21.5	0.9467	-0.20
catechol[c]	0.23-2.88	3.8-47.0	0.9998	-0.02
phenol[d]	1.50-18.88	24.6-308.0	0.9998	+0.005
3-methylcatechol[c]	7.40-92.50	120.8-1510	0.9996	-0.5
scopoletin[c]	0.08-1.00	1.3-16.4	0.9996	-0.002
p-cresol[d]	0.28-3.59	4.7-58.6	0.9998	+0.003
o-cresol[d]	0.18-2.20	2.8-36.0	0.9991	+0.04

[a] - Based on the smoke from 16 cigarettes in a 38-m^3 room sampled for four hours at 1.7 L/min
[b] - 200 μl injection
[c] - Based on 5 mL 1% acetic acid extraction of MCEM
[d] - Based on both the 5 mL extraction of MCEM and 5 mL elution of SiOHSP

Table 7.
Overall Precision(n=6) for the Smoking of 16 Cigarettes in a 38-m^3 Room (μgm^{-3})[a]

Compound	MCEM x,σ,%RSD	SiOHSP x,σ,%RSD	MCEM+SiOHSP x,σ,%RSD	MDQ
hydroquinone	1.96,0.12,6.12	BDL	1.96,0.12,6.12	0.16
catechol	1.18,0.05,4.24	BDL	1.18,0.05,4.25	0.08
phenol	0.98,0.05,5.10	5.49,0.20,3.64	6.47,0.20,3.09	0.29
3-methylcatechol	53.2,4.90,9.21	BDL	53.2,4.90,9.21	3.87
scopoletin	0.24,0.005,2.08	BDL	0.24,0.005,4.38	0.005
m+*p*-cresol	0.14,0.007,5.00	1.00,0.05,5.00	1.14,0.05,4.38	0.10
o-cresol	0.05,0.002,4.00	0.26,0.02,7.69	0.31,0.02,6.45	0.15

[a]- Sampled for four hours at 1.7 L/min
MDQ = Minimum detectable quantity at two times signal to noise ratio
BDL = Below detection limits

Table 8.
MCEM Sample Recovery/Standard Addition[a]

Compound	Amount Added, μg	Amount Recovered, μg	% Recovery
hydroquinone	0.32	0.31	96.8
	0.64	0.62	96.8
			$\bar{x}$ = 96.8
μg: standard addition = 0.82, external standard = 0.88			
catechol	0.47	0.49	104.2
	0.94	0.87	92.5
			$\bar{x}$ = 98.4
μg: standard addition = 0.46, external standard = 0.45			
3-methylcatechol	12.79	11.24	87.8
	24.15	21.06	87.2
			$\bar{x}$ = 87.5
μg: standard addition = 20.7, external standard = 24.1			
scopoletin	0.036	0.020	55.6
	0.068	0.033	48.5
			$\bar{x}$ = 52.0
μg: standard addition = 0.111, external standard = 0.117			

[a] - Amounts added prior to sampling, dried in desiccator overnight, extracted with 5mL 1% acetic acid by sonication for ten minutes, 16 cigarettes smoked over four hours in 38-m^3 room at 1.7 L min^{-1}

Table 9.
SiOHSP Sample Recovery/Standard Addition[a]

Coupound	Amount Added, μg	Amount Recovered, μg	% Recovery
phenol	0.66	0.62	93.9
	1.66	1.62	97.6
			$\bar{x}$ = 95.8
μg: standard addition = 3.07, external standard = 2.96			
p-cresol	0.24	0.14	58.3
	1.12	0.52	46.4
			$\bar{x}$ = 52.4
μg: standard = 0.44, external standard = 0.44			
o-cresol	0.16	0.15	93.8
	0.32	0.31	96.8
			$\bar{x}$ = 95.3
μg: standard addition = 0.46, external standard = 0.48			

[a] - amounts added prior to sampling, helium dried one-half hour at 0.5 L min^{-1}, eluted with 5 mL 1% acetic acid, 16 cigarettes smoked over four hours in 38-m^3 room at 1.7 L min-1

weighed. There was no weight loss after overnight drying from the weight after 20 minutes drying. The membranes spiked with scopoletin were used to sample ETS. The recovery on the Fluoropore membrane was *ca.* 31 percent and that for the MCEM, 45 percent, in the same range as the value in Table 8. It is unknown at this time why the recovery of scopoletin is poor unless the purity of the standard itself is questionable. The reason for the low recovery for *p*-cresol

Table 10.
Percentage Change After Two Weeks Storage of Samples[a] (n=2)

Compound	CONDITIONS				
	Extract/Eluent in Lab[b]	Extract/Eluent, 2°C[b]	MCEM Extract, 2°C[c]	Device in Lab	Device ,- 12°C
hydroquinone					
MCEM	-100.0	-74.0	-2.0	-22.0	-2.3
SiOHSP	d	d	---	d	d
catechol					
MCEM	-100.0	-22.4	-2.3	-45.2	+2.7
SiOHSP	d	d	---	d	d
phenol					
MCEM	-7.9	+10.0	-2.6	-13.4	+2.9
SiOHSP	-1.4	+7.8	---	-13.4	-3.0
3-methyl catechol					
MCEM	-100.0	-49.0	+3.2	-67.2	-10.4
SiOHSP	d	d	---	d	d
scopoletin					
MCEM	-93.6	+6.28	+12.4	-30.0	-5.0
SiOHSP	d	d	---	d	d
p-cresol					
MCEM	-35.4	-12.1	-6.0	-20.0	0.0
SiOHSP	-7.6	-7.9	---	-22.2	-6.9
o-cresol					
MCEM	-4.8	d	d	d	d
SiOHSP	0.0	+16.1	---	-30.0	+5.0

[a] - 16 cigarettes smoked over four hours at 1.7 L min^{-1} in 38-m^3 room, both MCEM and SiOHSP extracted/eluted with 5 mL 1% acetic acid
[b] - MCEM left in extractant two weeks
[c] - MCEM removed immediately after extraction
[d] - not in original sample

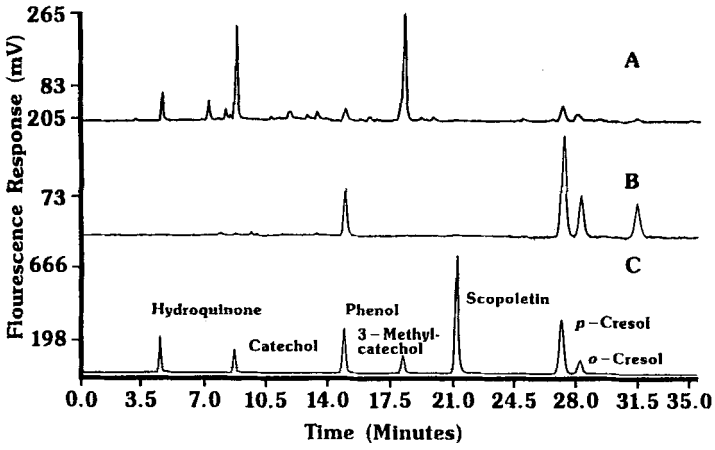

FIGURE 3. Chromatograms of fireplace smoke sampled for one hour at 2 L min^{-1} in a 45-m^3 room: (A) MCEM; (B) SiOHSP; (C) standard. Conditions: see text. Concentration of standard (ng mL^{-1}): hydroquinone (258); catechol (188); phenol (1232); 3-methylcatechol (6039); scopoletin (66); *p*-cresol (234); *o*-cresol (144).

(Table 9) is due to its co-elution with *m*-cresol under the chromatographic conditions used (1,4).

Storage of Samples

Results in percentage change of compound under various storage conditions of each device in Table 10 indicate that room temperature storage of the collection device is detrimental to these compounds over two weeks. Extract storage in the presence of the MCEM at room temperature practically destroys the main compounds that MCEM collects: hydroquinone, catechol, 3-methylcatechol and

Table 11.
Phenolic Compounds from Other Sources ($\mu g\ m^3$)

Compound	Source	
	Fireplace[a] (n=4)	16 Cigarettes[b] (n=6)
hydroquinone	2.20	1.96
catechol	8.36	1.18
phenol	16.18	6.47
3-methylcatechol	423.2	53.2
scopoletin	BDL	0.24
m+*p*-cresol	4.87	1.14
o-cresol	5.04	0.31

BDL = below detection limits
[a] - Sampled in 45-m^3 room, 125 L of air sampled
[b] - Sampled in 38-m^3 room, 408 L of air sampled

scopoletin. A re-extraction of the MCEM did not show that these compounds migrated back to the MCEM. To circumvent these problems, it is recommended that if the MCEM is extracted, its extract be stored at 2°C (refrigerated) with the MCEM removed immediately after sonication if analysis is not to be done within 24 hr. More practically, both devices should be stored at -12°C (freezer) immediately after sampling if analysis is not done within two weeks.

Phenolic Compounds from Fireplace Smoke

To show the usefulness of the procedure, air samples were taken with phenolic compounds present from fireplace smoke (Figure 3 and Table 11).

Note that scopoletin is below detection limits in fireplace smoke but that all other compounds are higher than ETS (Figure 2 and Table 11).

CONCLUSIONS

The above HPLC method was developed for the determination of hydroquinone, catechol, phenol, 3-methylcatechol, *m*+*p*-cresol, *o*-cresol and possibly scopoletin in indoor air. The simplicity of the sampling devices enables better operator control. The use of selective detection enables reliable measurements of these phenolic compounds to be made. Further work will be to improve the recovery of scopoletin and evaluate scopoletin as a particulate marker for cigarette smoke in indoor air.

ACKNOWLEDGEMENTS

This work is dedicated to the late Robert A. Heckman whose advice and encouragement enabled the development of this method and many other methods over the years. The author would like to thank Annette C. Pardue for the preparation of this manuscript.

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Received: March 17, 1993
Accepted: April 9, 1993