Table IV. Examples of Low Molecular Weight PAHs Content of Ashes Sampled in Two Different Coal-Operated Power Plants (ppb)[a]

compound	concn sample A, ppb		concn sample B, ppb	
	this method	soxhlet toluene	this method	soxhlet toluene
acenaphthylene	1.4	0.9	1.3	0.9
acenaphthene	2.0	1.7	1.6	1.2
fluorene	2.7	2.2	3.0	2.3
anthracene	3.2	1.4	0.9	0.5
fluoranthene	1.3	0.8	0.8	0.6
pyrene	1.0	0.3	0.5	0.4
benz[a]anthracene	nd[b]	nd	nd	nd

[a] Results obtained by using the direct elution extraction method (toluene at 100 °C) and the toluene Soxhlet extraction. [b] nd = not determined.

lengthy operation of this method, rather than to an actual poor recovery (*10*). Of course the results of Soxhlet extraction related to anthracene could be improved with some precautions to avoid the exposure of the sample to light.

Table III shows that the method described allows for recoveries of the four-ring PAHs of higher than 70% in all cases and 90% in all cases except three, at concentrations ranging between 2 and 60 ng of PAH/g of ash, while only 6–12 mL of solvent are used.

A very important problem arises with five-membered-ring compounds, which show absolutely unsatisfactory recoveries both with Soxhlet and with our method. At low concentrations of benzo[e]pyrene the recovery with Soxhlet by using toluene is 9%, while it is practically absent with the present method. These results are compatible with those obtained in previous work (*4*), so that the question of whether the heavier compounds are present or not in coal fly ashes remains open.

In Table IV the results obtained with the present method for two actual samples of fly ash are reported as an example. The same sample has been analyzed by using Soxhlet with toluene and the results are consistent with those obtained with the spiked samples. The two power plants where sampling was made have analogous characteristics as described. However, it should be noted that an average standard deviation of about 8% (measured on 10 samples) is found for the overall analytical method in the range of concentration between the detection limit (0.1 ppb) and 10 ppb, so that some numerical differences may not be significant.

The advantages of the method described lie essentially in the ease of operation and in the enormous time saving during sample preparation prior to GC/MS analysis. With the present method the sample is ready for analysis within half an hour, while using Soxhlet followed by solvent concentration, at least 24 h is necessary.

ACKNOWLEDGMENT

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Registry No. Acenaphthylene, 208-96-8; acenaphthene, 83-32-9; fluorene, 86-73-7; anthracene, 120-12-7; fluoranthene, 206-44-0; pyrene, 129-00-0; benz[a]anthracene, 56-55-3; chrysene, 218-01-9; toluene, 108-88-3.

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Desorption Characteristics of Four Polyimide Sorbent Materials Using Supercritical Carbon Dioxide and Thermal Methods

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^{14}C-Labeled 1,2,3,4,5,6-hexachlorocyclohexane, hexachlorobiphenyl, anthracene, and parathion were used to study the desorption of four polyimide-based sorbent materials using both supercritical carbon dioxide and thermal methods. Supercritical fluid desorption was found to be superior to thermal desorption. Both types of desorption were more difficult from the polyimides than from Tenax-GC used in previous work. This work helps to define the applicability of supercritical desorption of polyimides. The identities of the compounds desorbed with supercritical CO_2 were verified by using thin-layer chromatography and mass spectrometry. Results were compared to those from Tenax-GC studies.

The identification and quantification of organic compounds in ambient air are problems that are complicated both by the wide range of molecular weights and polarities of these compounds and by the trace levels at which these compounds are present. One of the most useful methods to overcome the problem of low analyte concentration is to use a sorbent such as Tenax-GC (*1*, *2*). In such an analysis, an air stream is drawn through a cartridge packed with the sorbent material and the organic compounds are selectively retained. The trapped compounds are subsequently thermally desorbed and cryogenically focused onto the head of a gas chromatographic column for analysis.

Although such chromatographic preconcentration techniques are very powerful, they do have limitations. One such limitation is the poor retention of certain compounds on the sorbent itself. For example, Tenax-GC retains nonpolar compounds much more efficiently than polar compounds such as methanol or vinyl chloride (*2*). This results in the "breakthrough" of the polar compounds while the nonpolar materials are still being effectively concentrated and the inability to quantify such poorly retained, polar materials. If,

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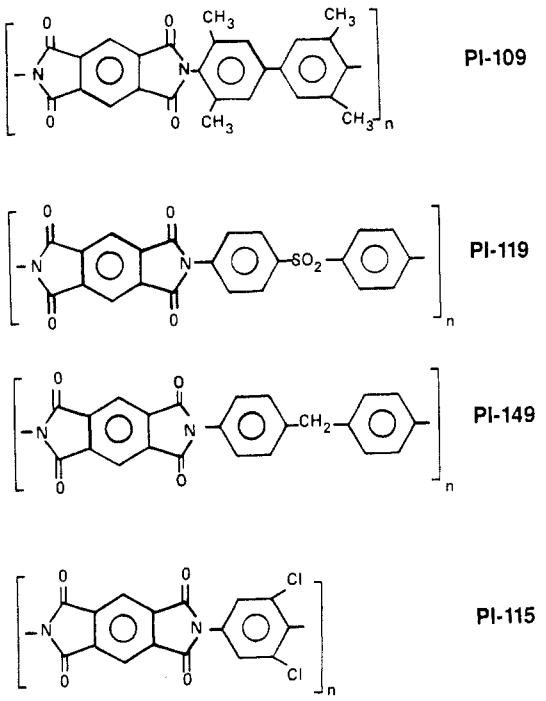

Figure 1. Polyimide (PI) sorbents used in this study.

however, sampling is terminated prior to the breakthrough of such polar compounds, the absolute masses of compounds accumulated may be quite small and poor sensitivity will be observed. The analyst must therefore evaluate sampling goals and choose the target compounds or sampling conditions such that adequate sensitivity is realized and the resulting quantification is valid.

The use of more polar sorbents should increase the breakthrough volumes of compounds such as methanol and thus provide for the analysis of compounds with a broader range of polarities than is possible using Tenax-GC. The development of some polyimide-based sorbents was the goal of a project at Research Triangle Institute (RTI) (*3*). Of the many polymers synthesized, the four shown in Figure 1 were shown to provide good retention of polar compounds, good thermal stability (necessary for use with thermal desorption), and low background. The suitability of these polymers for use in air sampling is currently being studied in detail in our laboratory. Gas chromatographic retention data for a variety of compounds on the four polyimides and Tenax-GC are shown in Table I (*3*).

Another problem associated with this preconcentration technique can occur in the thermal desorption step if an organic compound is strongly adsorbed on the sorbent. Thermal desorption can fail because of the very strong interactions between the analyte and the sorbent since a temperature sufficient to desorb the analyte might also destroy the sorbent, the analyte, or both. If the analyte is thermally unstable, thermal desorption can invalidate quantification and introduce artifacts even if the analyte is only weakly adsorbed.

The use of supercritical carbon dioxide to accomplish the desorption can provide solutions to these last two problems. First, the critical temperature of CO_2 is 31 °C; thus high temperatures are avoided. Second, the relatively high density of supercritical CO_2 can provide for a solvating capability that will desorb compounds that cannot be desorbed thermally. The application of this technique to desorb 1,2,3,4,5,6-hexachlorocyclohexane (γ-BHC), anthracene, 2,3,5,2′,3′,5′-hexachlorobiphenyl, and parathion from Tenax-GC with good recoveries has recently been demonstrated (*4*). Wright et al. (*5*) have also demonstrated the supercritical fluid extraction of XAD-2 resin, polyurethane foam (PUF), Spherocarb, and urban dust. More and more researchers are investigating the use of supercritical fluids for extraction and desorption as an attractive alternative to solvent and thermal approaches. The present work was carried out to see if supercritical CO_2 desorption is equally applicable to the more highly retentive polyimides as it is appliable to Tenax-GC for the specified compounds. ^{14}C-Labeled compounds were used for the recovery determinations.

EXPERIMENTAL SECTION

Materials. Solutions of ^{14}C-labeled γ-BHC, anthracene, parathion, and 2,3,5,2′,3′,5′-hexachlorobiphenyl were prepared and the radiochemical purities were determined by using thin-layer chromatography (TLC) as previously described (*4*). The polyimide materials, previously synthesized at RTI (*3*), were sieved, and the 40–60 mesh fraction for each polymer was used to fill a sorbent cartridge with a bed volume of 0.8 mL as for our earlier work with Tenxa-GC (*4*).

Procedures. Aliquots of the radiolabeled analytes were loaded onto the polyimide sorbent and desorbed into scintillation cocktail, using supercritical carbon dioxide as previously described for Tenax-GC (*4*). An exception is that up to 14 mL of CO_2, as measured at room temperature and 3000 psi, was used to examine the recovery as a function of CO_2 volume. For each polyimide, the sample was loaded and the supercritical carbon dioxide desorption was carried out by using a fresh vial of scintillation cocktail to trap analyte from the effluent, for every 2 mL of liquid used in order to determine the CO_2 volume needed for desorption. Two milliliters of liquid CO_2 is roughly equivalent to 10 column volumes at supercritical conditions. The desorption volumes thus determined were used in the actual recovery determinations where each desorption was performed in duplicate. The reproducibility of the method was generally within 10% and allowed for meaningful comparisons of the recoveries as well as the elucidation of retention trends.

We felt that it was very important to verify the nature of the materials eluted from each of the four sorbents in terms of the radiochemical purity and chemical structure of the main component. Because earlier work (*3*) did not suggest any analyte transformation by the sorbents themselves, any structural changes seen in the present work would be the result of aspects of the procedure unrelated to the particular sorbent in use. For this purpose, polyimide 119 (sulfone) was used as a representative sorbent. Thin-layer chromatographic and mass spectral methods were as previously described (*4*).

Thermal desorption recoveries for γ-BHC, parathion, and hexachlorobiphenyl were obtained as for Tenax-GC (*4*) except that helium volumes of 12 mL ("low flow", 50 column volumes) and 120 mL ("high flow", 500 column volumes) were used. The low flow experiments used a helium volume that corresponds the same number of column volumes used for the thermal desorption of the larger Tenax-GC cartridges currently used for field sampling (*2*).

RESULTS AND DISCUSSION

Before the present work on the desorption of the polyimides was begun, we ranked the sorbents by using the gas chromatographic retention volumes, shown in Table I, and efficiency data (theoretical plates, see ref 3) that each test compound demonstrated on each polymer. For each polymer, the average of all of the retention volumes ($\bar{V}$) and the average of all of the efficiencies ($\bar{N}$) were determined to allow a one number comparison for each of the two parameters. The ideal sorbent will have a large retention volume, i.e., large volumes of air may be sampled without breakthrough of the organic compound of interest, and high demonstrated efficiencies for each test compound, i.e., the test compounds are "well trapped" and breakthrough, when it does occur, happens quickly. Such a treatment here indicated the following trends:

$$\bar{V}: \text{ Tenax-GC} < 115 = 119 = 149 < 109$$

$$\bar{N}: 115 = 149 < \text{Tenax-GC} < 119 < 109$$

When both of these factors are taken into consideration, the polyimides can be listed in decreasing priority as follows: (1)

Table I. Gas Chromatographic Retention Data for Polyimides and Tenax-GC (3)

	retention volume in mL[a]				
test compound	Tenax-GC	PI-109	PI-119	PI-149	PI-115
heptane	270	430	310	420	490
octane	320	490	390	NS[b]	NS
nonane	360	540	430	NS	NS
decane	400	590	470	NS	NS
benzene	230	450	350	420	380
ethanol	77	330	300	300	270
2-butanone	200	450	370	410	380
nitromethane	160	430	640	420	370
pyridine	280	560	500	520	510
acetonitrile	110	380	320	380	340
dichloroethane	220	450	350	430	380
vinyl chloride	49	210	130	190	180
water[c]	2.5	36	15	33	70

[a] Retention data for equal volumes of sorbent (column 0.914 m × 2 mm i.d.). Temperature program from 50 to 275 °C at 8 °C/min was used with helium carrier at 20–25 mL/min. [b] Not separated from each other. [c] Water was analyzed isothermally at 120 °C.

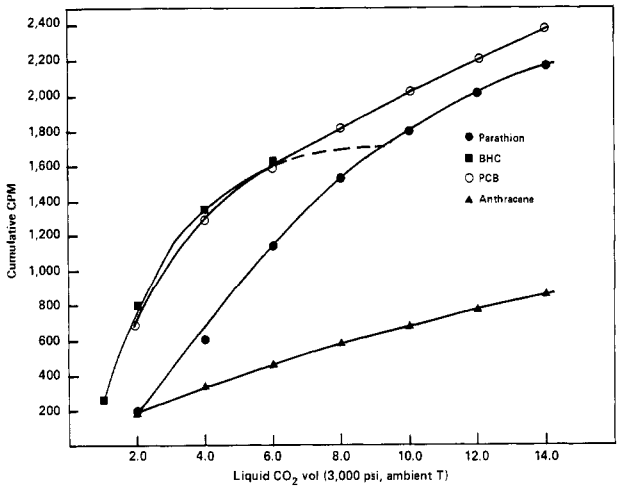

Figure 2. Radioactivity recovery as a function of CO_2 volume for PI-109.

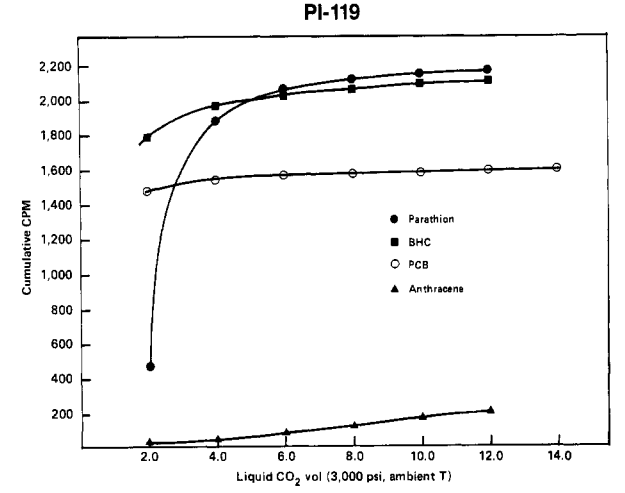

Figure 3. Radioactivity recovery as a function of CO_2 volume for PI-119.

109, (2) 119, (3) 149, and (4) 115. Tenax-GC is included as a substance for comparison because it is in such widespread use for environmental sampling.

Supercritical CO_2 Desorption. Plots of the cumulative counts per minute vs. liquid carbon dioxide volume for the four test compounds on each of the four polymers are shown in Figures 2–5. The first thing we learned after generating these data was that these polymers are indeed much more retentive than Tenax-GC where 14 column volumes, or approximately 2.8 mL of liquid CO_2, were sufficient to give very good recoveries (4). In addition, the general patterns of retentions support our predictions based on the gas chromatographic data discussed above; i.e., PI-109 and PI-115 are the most and least retentive sorbents, respectively. It was also noted that anthracene is very poorly desorbed from all of the polymers, indicating a rather high energy of adsorption. Because of this, anthracene was not included in the following recovery experiments except in one case where its recovery was determined simply to quantify the poor desorption character.

After the desorption volumes were determined, the recovery capability was examined more closely. The quantities of radioactivity that could be recovered from the polyimides by using supercritical CO_2 are shown in Table II. For this experiment each desorption was done at least twice and reproducibility was almost always within 10%. The recoveries shown are with respect to the counts obtained from a known quantity of radiolabeled analyte added directly to scintillation cocktail. All of the analytes, with the exception of parathion on PI-109 and anthracene appear to be well recovered. The retention trends suggested by Figures 2–5 are supported by these data.

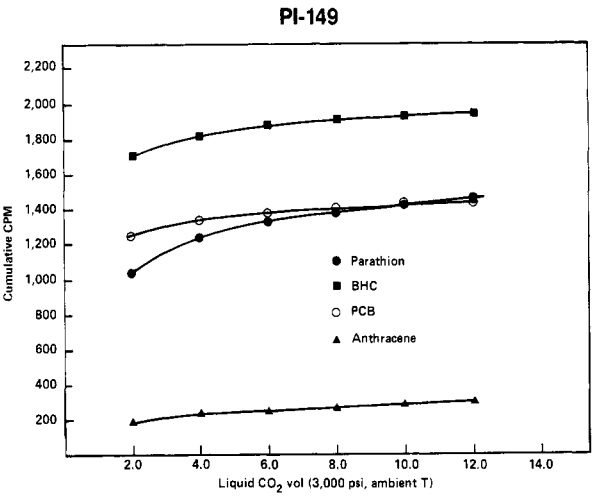

Figure 4. Radioactivity recovery as a function of CO_2 volume for PI-149.

Table II. Percent Radioactivity Recovered by Supercritical CO_2 Desorption

	sorbent			
compound	PI-109 (a)	PI-119 (a)	PI-149 (a)	PI-115 (a)
γ-BHC	100 (45)	86 (30)	92 (30)	99 (30)
hexachlorobiphenyl	81 (60)	89 (30)	87 (30)	95 (30)
parathion	67 (101)	85 (45)	87 (45)	88 (45)
anthracene	*b*	*b*	26 (45)	*b*

[a] Approximate number of column volumes of CO_2 used at 3000 psi and 40 °C. [b] Recovery not determined.

Table III. Purities of Analytes from TLC before and after Supercritical CO_2 Desorption from Polyimide 119

compound	control	after desorption
γ-BHC	92%	99%
hexachlorobiphenyl	90%	79%
parathion	97%	84%

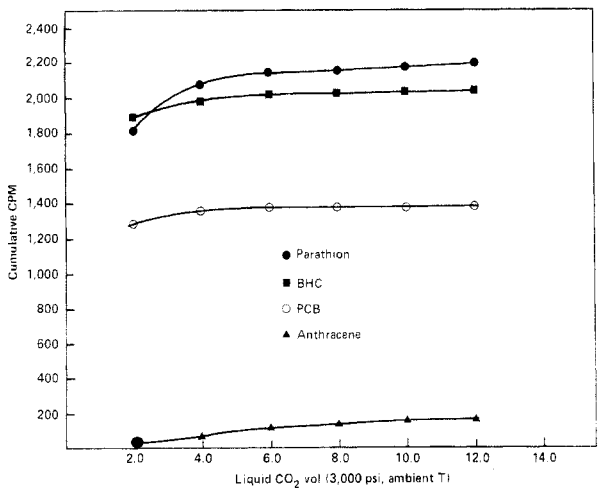

Figure 5. Radioactivity recovery as a function of CO_2 volume for PI-115.

Table IV. Percent Compound Recovered by Supercritical CO_2 Desorption Corrected for TLC Purity

compound	PI-109	PI-119	PI-149	PI-115
γ-BHC	100	86	92	99
hexachlorobiphenyl	72	79	77	85
parathion	58	74	76	77
anthracene	–[a]	–	–	–

[a] Not determined, see text.

Before firm conclusions can be made based on the above recoveries, the radiochemical purities of the compounds after desorption must be examined. Purity data based on single analyses are shown in Table III and indicate decreases for both parathion and hexachlorobiphenyl. The decrease in the purity for the biphenyl was a surprise since such compounds are notoriously stable. Such a decrease in purity was also produced in the desorption of hexachlorobiphenyl from Tenax-GC (*4*), so is most likely real. However, an apparent decrease in the purity could be caused by a higher recovery from the sorbent of the initial impurity as compared to the hexachlorobiphenyl. Such a situation mandates that data of Table II be corrected to account for this. Compound recoveries from Table II corrected for any decreases in purity after desorption shown by TLC experiments are shown in Table IV. The mass spectrum of the major component from each TLC plate compared well to the reference spectrum of the corresponding parent compound, thus serving to verify the structures of the desorbed compounds. These data are shown in Table V. When reference parathion was separated by TLC alongside the desorbed compound, only one spot in the developed plate was seen for each sample spot applied. The spot from the reference material had the same retention as the spot from the desorbed parathion sample. The differences between the desorbed and reference parathion mass spectra are most likely the result of a much lower mass present in the desorbed sample. The molecular ion observed for both the reference and desorbed parathion samples was that expected for the unchanged compound.

The corrected recovery data of Table IV indicate that supercritical carbon dioxide can be successfully used to desorb three of the four test analytes from the polyimides. Again, recoveries do improve as polymers of lower overall retention are used. Recoveries lower than about 80% make quantification more difficult because as the recoveries decrease, the variations in the measured results increase. Here, the marginal recoveries simply indicate that we have approached the limit of applicability of supercritical carbon dioxide desorption of the polyimide sorbents. Based on data in Table I, the advantages of these new sorbents will be in the sampling of polar, volatile organic compounds. Supercritical desorption of compounds having lower molecular weights than the most difficult compounds used in this study could most likely be used with the polyimide materials. This desorption method could easily be used to extend the useful analyte molecular weight range over that achievable by using thermal desorption of sorbent materials. Earlier work with Tenax-GC (*4*) demonstrated the supercritical CO_2 desorption of hexachlorobiphenyl and parathion and suggested that other methods of air sampling, such as those using polyurethane foam (PUF) plugs, might not be needed. A wide spectrum of materials could be assayed by using only Tenax-GC. Similar molecular weight extensions should be achievable with supercritical CO_2 desorption of the polyimides as compared to thermal desorption of the same materials.

Thermal Desorption. Thermal desorption data for the sorbents are presented in Table VI. With the low desorption volumes, recoveries from all of the sorbents were very poor although the general trends in recovery as sorbent tenacities decrease are maintained. The use of larger desorption volumes improved the recoveries for γ-BHC to acceptable levels for polyimides 119, 149, and 115. The recoveries for hexachlorobiphenyl improved but were still not very good. One of the most interesting comparisons of the polyimides to Tenax-GC can be made based on data in this table. Anthracene could be thermally desorbed from Tenax-GC while

Table V. Significant Mass Spectral Ions and Percent Relative Abundance (RA) Data for Desorbed and Standard Compounds

γ-BHC (mol wt 288)			hexachlorobiphenyl (mol wt 358)			parathion (mol wt 291)		
ion	% RA desorbed	% RA std	ion	% RA desorbed	% RA std (*6*)	ion	% RA desorbed	% RA std
109	59	58	218	28	30	97	86	67
111	56	58	288	48	65	109	100	73
181	100	100	290	67	82	123	*a*	25
183	100	93	292	38	40	125	*a*	33
217	70	61	323	14	10	137	*a*	49
219	88	84	325	19	15	139	*a*	45
221	44	37	327	14	10	155	43	41
252	17	16	358	48	50	186	14	23
254	32	26	360	100	100	218	*a*	12
256	20	16	362	71	80	235	14	23
288	6	6	364	38	32	263	14	19
290	12	10				291	86	100
262	9	8						
294	3	4						

[a] Not detected.

Table VI. Percent Recovery Using Thermal Desorption at 250 °C

compound	PI-109 50[a]	PI-109 500[a]	PI-119 50	PI-119 500	PI-149 50	PI-149 500	PI-115 50	PI-115 500
γ-BHC	1	50	6	88	9	83	22	94
hexachlorobiphenyl	1	14	16	21	17	57	32	59
parathion	8	3	3	9	8	25	12	36
anthracene	8	3	8	6	11	26	14	18

[a] These values indicate the number of column volumes of helium used for the desorption.

hexachlorobiphenyl and parathion could not, indicating the different selectivities of the sorbent materials. The polyimides, on the other hand, begin to release hexachlorobiphenyl and parathion, especially evident for PI-115, under high flow thermal desorption conditions while anthracene cannot be desorbed even when using supercritical carbon dioxide. Such selectivities are the result of different sorptive sites on the polymeric materials. The practical implications of such different sites on the polyimides are currently being studied in our laboratory.

Retention data for water on the various sorbent materials are shown in Table I. As sorbents become more polar in an effort to more efficiently trap polar, volatile materials, water is retained to much higher degrees than on Tenax-GC. Consequently, an analytical method which uses a cryogenic focusing step after desorption of the polymer must take this water into account, otherwise substantial quantities of ice might form in the trap and obstruct flow through the system. We are currently evaluating various means to accomplish this water removal so that the polar nature of these polyimide sorbents can be used to full advantage.

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Indirect Determination of Cyanide Compounds by Ion Chromatography with Conductivity Measurement

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Ion chromatography (IC) is a suitable analytical technique for the determination of anions. The cyanide is not detected by the conductivity detector of the ion chromatograph due to its low dissociation constant (pK = 9.2). This paper describes an IC procedure for the determination of free cyanide and metal cyanide complexes that uses a conductivity detector. It is based on the oxidation of cyanide ion by sodium hypochlorite to cyanate ion (pK = 3.66). Therefore, cyanide ion can now be measured indirectly by the conductivity detector. In this procedure, optimum operating conditions were examined. In addition, the interferences from anions and reducing agents were investigated. The method was applied to the determination of metal cyanide complexes. The coefficients of variation (%) for CN^- (1.05 mg/L), $Zn(CN)_4^{2-}$ (CN^-, 0.80 mg/L), and $Ni(CN)_4^{2-}$ (CN^-, 0.96 mg/L) were 1.1%, 1.5%, and 0.5%, respectively. The proposed method proved to be useful for the determination of cyanide compounds in natural water and wastewater.

Many methods have been developed for the determination of cyanide compounds in water, wastewater (*1*, *2*), and plating solutions (*3*). These methods make use of titrimetry, colorimetry, cyanide-selective electrodes (*1*, *2*), coulometry (*4*, *5*), gas chromatography (*6*), high-performance liquid chromatography (*7*), and so on.

Usually, ion chromatography (IC) is the most suitable technique for the separation and determination of both anions and cations (*8*). However, hydrogen cyanide cannot be directly detected by the conductivity detector of the ion chromatograph due to its low dissociation constant (pK = 9.2).

Girard reported the use of controlled potential coulometry as a detection method for cyanide ion (*9*). An IC with an amperometric detector was also used for the cyanide and/or metal cyanide determination by Bond et al. (*10*) and Rocklin et al. (*11*). Wang et al. reported a potentiometric measurement obtained by using an ion-selective electrode for the determination of cyanide and sulfide (*12*). These electrochemical methods are sensitive and selective for cyanide ion, but such detectors are too expensive and not familiar.

Another IC method without an electrochemical detector (ECD) has also been reported. Pinschmidt developed a procedure for the determination of weak acid ions including cyanide ion (*13*). Dolzine et al. proposed a procedure for the determination of hydrogen cyanide in air by converting it to sodium formate followed by its measurement (*14*). DuVal et al. used the reaction between cyanide ion and iodine (*15*). However, these methods are time consuming and/or troublesome. Some metal cyanide complexes such as iron, gold, and cobalt can be measured using a MPIC-NS1 separator column with a conductivity detector (*16*). Silinger reported a method that uses sodium hypochlorite to oxidize free cyanide ion (CN^-) to cyanate ion (CNO^-) before chromatographic

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