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Extraction of Semivolatile Organic Compounds from High-Efficiency Particulate Air (HEPA) Filters by Supercritical Carbon Dioxide

by J. B. Schilling

Chemical Technology Division
Argonne National Laboratory, Argonne, Illinois 60439
operated by The University of Chicago
for the United States Department of Energy under Contract W-31-109-Eng-38

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EXTRACTION OF SEMIVOLATILE ORGANIC COMPOUNDS FROM HIGH-EFFICIENCY PARTICULATE AIR (HEPA) FILTERS BY SUPERCRITICAL CARBON DIOXIDE

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September 1997

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ABSTRACT

Supercritical fluid extraction (SFE) using unmodified carbon dioxide has been explored as an alternative method for the extraction of semivolatile organic compounds from high-efficiency particulate air (HEPA) filters. HEPA filters provide the final stage of containment on many exhaust systems in U.S. Department of Energy (DOE) facilities by preventing the escape of chemical and radioactive materials entrained in the exhausted air. The efficiency of the filters is tested by the manufacturer and DOE using dioctylphthalate (DOP), a substance regulated by the U.S. Environmental Protection Agency under the Resource Conservation and Recovery Act. Therefore, the filters must be analyzed for semivolatile organics before disposal.

Ninety-eight acid, base, and neutral semivolatile organics were spiked onto blank HEPA material and extracted using SFE, Soxhlet, automated Soxhlet, and sonication techniques. The SFE conditions were optimized using a Dionex SFE-703 instrument. Average recoveries for the 98 semivolatile compounds are 82.7% for Soxhlet, 74.0% for sonication, 70.2% for SFE, and 62.9% for Soxtec. Supercritical fluid extraction reduces the extraction solvent volume to 10-15 mL, a factor of 20-30 less than Soxhlet and more than 5 times less than Soxtec and sonication. Extraction times of 30-45 min are used compared to 16-18 h for Soxhlet extraction.

I. INTRODUCTION

High-efficiency particulate air (HEPA) filters provide the final stage of containment on many exhaust systems in U.S. Department of Energy (DOE) facilities by preventing the escape of chemical and radioactive materials entrained in the exhausted air. The trapping efficiency of the filters is tested, using bis(2-ethylhexylphthalate) (normally referred to as dioctylphthalate or DOP), by both the manufacturer and by DOE, at one of three HEPA test facilities as well as in the installed duct work at various operating laboratories or production facilities.[1] Dioctylphthalate is a substance regulated by the U.S. Environmental Protection Agency (EPA) under the Resource Conservation and Recovery Act (RCRA). It is considered a hazardous chemical under the State of Washington's Dangerous Waste Regulations, which impacts the acceptance criteria for solid waste sent to the Hanford disposal site. Therefore, the filters must be analyzed for semivolatile organic compounds before disposal.

Traditional extraction techniques for semivolatile organic compounds (SVOCs) include Soxhlet, automated Soxhlet (Soxtec), and sonication.[2] These methods use between 70 and 300 mL of hazardous solvents such as methylene chloride and thus produce large amounts of secondary waste. Waste minimization and pollution prevention have not usually played a role in development of procedures for waste characterization, but new procedures are needed that address these concerns.

A promising technique in this regard is supercritical fluid extraction (SFE), which has been used in many studies for the extraction of organic compounds from environmental solid samples.[3-20] Carbon dioxide is particularly advantageous when used as the supercritical fluid since the extraction solvent can be vented directly to the atmosphere. Organic solvents are needed only in small quantities for collection of the extracted analytes or to modify the

supercritical fluid. In many instances, modifiers are needed to increase the extraction efficiency of SFE for certain matrices or analytes,[21,22] especially for extracting polar materials from solids and sediments that exhibit a wide variety of matrix effects. HEPA filters are made of glass fibers held together with a binder and should be much more uniform. They would not be expected to show the wide variety of matrix effects seen in soils and sediments so modification of the supercritical fluid should not be required.

The Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) is assisting the Waste Management Department of DOE's Environmental and Waste Management Programs in the characterization of historical HEPA filter waste to show that it meets the Hanford waste acceptance criteria.[23] The ACL has been using sonication as the extraction method to remove SVOCs from the HEPA filters for analysis.

However, a method based on SFE has been developed for extraction of SVOCs from HEPA filters. The method was developed by working with spiked filter material. Results for the supercritical fluid extraction of 98 acid, base, and neutral SVOCs analyzed under the EPA's SW-846 Method 8270 are compared to extraction and analysis using the traditional extraction techniques.

II. EXPERIMENTAL

A. Standards

All SW-846 Method 8270 internal standards, base/neutral and acid surrogates, and the decafluorotriphenylphosphine (DFTPP) gas chromatography/mass spectrometry (GC/MS) tuning standard were purchased from ULTRA Scientific (North Kingstown, RI). The semivolatile base/neutral and acid analytes (BNAs) were also purchased from ULTRA Scientific as eight separate mixtures in methylene chloride. The mixtures were combined with the acid and base/neutral surrogates to give a spiking solution with a concentration of 167 µg/mL for each of the 92 BNAs and the three base/neutral surrogates and 333 µg/mL for each of the three acid surrogates. Calibration solutions for GC/MS were made by preparing a separate stock standard solution from the eight BNA mixtures. The stock solution was diluted to the appropriate concentrations and combined with the internal standards and surrogates. Table 1 lists the 98 compounds in the spiking solution grouped into arbitrary compound classes based on functional groups.

B. Solvents

The organic solvents used in this study (methylene chloride, acetone, and hexane) were capillary GC/GC-MS grade solvents from Burdick & Jackson. Soxhlet and sonication extractions were performed using methylene chloride. For Soxtec extraction either 1:1 hexane:acetone or methylene chloride was used. The solvents studied for use as SFE collection solvents were methylene chloride, 1:1 methylene chloride:acetone, and 1:1 hexane:acetone.

C. HEPA Filter Spiking

Sheets of HEPA filter material were obtained from Flanders Filters, Inc. (Washington, NC). The sheets were cut into small pieces approximately 1 cm^2 in size. This material had not

been tested with DOP and subsequent extraction of the material revealed no significant quantities of semivolatile target analytes.

For each sample, a 1.0 g quantity of HEPA filter material was placed in a 2-oz. (~60 mL) glass bottle. A sample weight of 1.0 g was chosen to simulate the amount normally used in HEPA filter analysis at ANL. Next, 600 μL of the methylene chloride spiking solution described above was added dropwise to the material. Thus, each sample was spiked with 100 μg of each of the BNA analytes and base/neutral surrogate standards and 200 μg of each of the acid surrogate standards. The solvent was allowed to evaporate, and the samples were sealed in the bottles and allowed to sit for approximately 1 h. After the spiked filter material was transferred to the extraction apparatus, the bottles used for spiking were triple rinsed with methylene chloride, and the rinsate was concentrated and analyzed for semivolatile compounds. BNA concentrations found in the bottle rinsate were used to correct the total spike amounts.

D. Extraction of Samples Using Supercritical Fluid Extraction

All supercritical fluid extractions were carried out by using a Dionex SFE-703 instrument (Dionex Corporation, Sunnyvale, CA). Figure 1 shows a schematic diagram of the SFE extractor. Up to eight samples may be extracted at one time. The supercritical CO_2 is depressurized through heated, wafer-style restrictors and bubbled through a vial of a collection solvent. The instrument was equipped with 10-mL stainless steel cells (hand-tight 14 x 60-mm vessels from Keystone Scientific, Bellefonte, PA) and 250 mL/min heated restrictors. The cells are capable of operating at up to 10,000 psi (6.9×10^4 kPa). The carbon dioxide used was SFC/SFE-grade CO_2 with helium head space at 2000 psi (1.38×10^4 kPa) (Air Products and Chemicals, Lehigh Valley, PA). Although the system is capable of extracting up to eight samples simultaneously, in general, samples were extracted in duplicate. The spiked filter material was loaded into the

SFE cells between layers of Ottawa sand standard (Fisher Scientific, Pittsburgh, PA). Unless otherwise noted, the extractions were carried out dynamically for 30 min, the SFE restrictors were held at 150°C, and the analytes were collected in 15 mL of methylene chloride held at 0°C. After the extractions were complete, the collection solvent was concentrated to 1 mL by using a Kuderna-Danish (K-D) concentrator. The specifics of the optimization process and experimental conditions are given in Section III.

E. Extraction of HEPA Filters Using Traditional Methods

Soxhlet extraction of spiked HEPA filter samples was carried out using a standard extraction apparatus. First, 1.0 g of spiked filter material was placed in a Soxhlet thimble and covered with sand. Next, 300 mL of methylene chloride was added to the apparatus and the extraction was carried out for approximately 16 h. The final extract was concentrated to 1 mL using a K-D concentrator. For this method, 12 extractions (three sets of four) were run over the course of several months.

Automated Soxhlet (Soxtec) extractions were carried out with a Tector Soxtec System HT2 extraction unit. Two sets of four samples were extracted, one set with 75 mL of methylene chloride, one with 75 mL of 1:1 hexane:acetone. The samples were extracted in the boiling solvent for 1 h followed by 1 h of rinsing with the sample suspended above the solvent. The extraction solvent was concentrated to approximately 10 mL in the Soxtec apparatus and then to 1 mL in a K-D concentrator.

Sonication extraction followed an extraction procedure that was slightly modified from the one normally used at ANL for HEPA filters. First, 25 mL of methylene chloride was added to the 1.0 g of the spiked solid. The solution was sonicated for two minutes using a Heat Systems-Ultrasonics, Inc. Sonicator unit. The extraction solvent was decanted through a pipet

filled with glass wool. After one sonication, it was obvious from the color of the filter material that not all of the semivolatiles had been removed. Therefore, the sonication was repeated twice more with fresh solvent. The standard procedure uses only one sonication step. The three resulting liquid extracts were combined and concentrated to 1 mL in a K-D concentrator. Six samples were extracted by using this process.

F. Extract Analysis

All SFE, sonication, Soxtec, and Soxhlet extracts were analyzed by GC/MS according to SW-846 Method 8270.[2] The analyses were run on a Hewlett-Packard 5890 Series II gas chromatograph (equipped with a 5973A automatic liquid sampler) interfaced to a 5989A quadrupole mass spectrometer. A splitless injection of 1 μL of sample was made onto a J&W DB-5MS column (30-m length x 0.32-mm i.d. x 0.25 μm film thickness). Helium was used as the carrier gas, with an approximate flow of 1.5 mL/min through the column and a split flow of 50 mL/min. The injector purge valve was opened 45 seconds after injection. The injector temperature was held at 280°C. The initial oven temperature of 35°C was held for 2 min, ramped to 200°C at 6°C/min, ramped to 280°C at 10°C/min, and held at 280°C for 17.5 min. The GC/MS interface temperature was held at 280°C with an ion source temperature of 270°C. Compounds were ionized by using 70 eV electron impact ionization, and spectral scan data were taken over a mass range of 35-500 amu.

III. RESULTS AND DISCUSSION

A. Control Tests and Supercritical Fluid Extraction Method Development

To determine any possible contamination of the sand and HEPA filter material, 10-mL cells full of the Ottawa sand standard or 1.0 g blank HEPA filter material (plus sand to fill the cell) were subjected to a 30-min dynamic extraction with supercritical CO_2. Any extracted material was collected in methylene chloride and run through the normal concentration process and GC/MS analysis. Extraction of sand showed no semivolatile organic compounds. Extraction of the HEPA filter material gave very small amounts of three phthalates (< 2 μg) and several polynuclear aromatic hydrocarbons (PAHs) (< 0.5 μg). Compared to the spike amounts and the errors associated with the extraction and analysis, these values are not significant.

Several extractions were run on blank sand with the semivolatiles spiked into the collection solvent. This allows the determination of possible volatilization losses resulting from bubbling the CO_2 through the solvent. In four determinations, the average recovery for all 98 spike compounds was greater than 95%.

To determine the effect of the collection solvent on recovery, sand was spiked with the semivolatile compounds and extracted by using three different solvents and two different solvent volumes. The three solvents are standard solvents used for semivolatile organic extraction: 1:1 hexane:acetone; 1:1 methylene chloride:acetone; and methylene chloride. Six extractions were done using each solvent; four with 15 mL of solvent and two with 10 mL. Figure 2 compares the recoveries for the three solvents (15 mL) by compound class, and Table 2 gives the average recoveries for the 98 compounds by solvent and volume. Average recoveries for individual compounds and compound classes show somewhat larger variation, but, overall, there is little difference between the three solvents and the two solvent volumes used (the maximum difference

is about 5%). Methylene chloride was chosen for most of the experimental work. It requires no solvent mixing and does not have the drawbacks of the acetone-containing solvents, which always show large acetone aldol condensation product peaks in the GC/MS analysis.

To optimize the extraction of semivolatile organic compounds from HEPA filter material, extractions were carried out at temperatures of 40, 50, 75, 100 and 125°C and pressures of 300, 400, 500, and 600 atm (30.4, 40.5, 50.7, and 60.8 MPa). Representative data for average recoveries (by compound class) are shown in Figs. 3 and 4. Figure 3 shows the recovery variation with supercritical fluid pressure at an extraction temperature of 50°C. The compound recoveries rise between 300 and 400 atm (30.4 and 40.5 MPa) and then drop off as the pressure is increased further. Figure 4 shows the temperature dependence of the semivolatile recoveries at an extraction pressure of 400 atm (40.5 MPa). The recoveries rise between 40 and 50°C and then decrease as the temperature increases. Overall, the highest recoveries were obtained at 50°C and 400 atm (40.5 MPa).

Duplicate extractions were made with dynamic extraction times of 5, 10, 20, 30, 45, and 60 min. The extraction temperature and pressure were held at 50°C and 400 atm (40.5 MPa). Figure 5 shows the variation of the average percent recovery across all 98 compounds versus the extraction time. The recovery increases rapidly at short extraction times and then levels off. The figure shows that extraction is essentially complete after about 30 min.

Final SFE extraction data were obtained by running three sets of eight spiked samples over the course of several weeks. The method differed slightly for each set of samples. Two sets were extracted for 45 min, one using hexane/acetone collection solvent and one using methylene chloride. The third set was extracted for 30 min using methylene chloride. Table 3 presents a comparison of the average results for each of the 98 semivolatile organic compounds

for the three sets of data. In general, differences seen among the three sets of data are no larger than what is normally expected in semivolatile extraction and analysis. The only major difference appears to be the lower recoveries of aromatic amines when hexane/acetone is used compared to when methylene chloride is used. Average percent recoveries across all 98 compounds are 70.1% (hexane/acetone, 45 minutes), 71.0% (methylene chloride, 30 minutes), and 67.4% (methylene chloride, 45 minutes).

B. Comparison of Results from Supercritical Fluid Extraction and Traditional Extraction Methods

Table 4 compares the SFE recoveries with those from Soxhlet, Soxtec, and sonication. The SFE results are the average of the 24 final extractions just discussed. The Soxhlet numbers are the average of 12 extractions (three sets of four) run over the course of several months. Soxtec recoveries are the average of two sets of four samples, one set extracted with hexane/acetone and one with methylene chloride. Sonication results are from extraction of a set of six spiked samples. The lower percent relative standard deviation (%RSD) values for sonication are due in part to the fact that all six samples were processed as a group, while the other techniques involved running several groups of samples over an extended period of time.

Sonication and SFE are compared in Figures 6-12. Figures 6-11 plot average percent recovery for each compound. The error bars indicate plus and minus one standard deviation from the mean. Figure 12 gives average percent recovery values by compound class. Overall, for the 98 compounds, the average recovery for sonication is 74.0% and for SFE, 70.2%. For 58 compounds, average recoveries are higher for sonication, while the remaining 40 compounds have higher values with SFE. In general, there is good agreement between the sonication and SFE results for many of the compound classes. Excellent agreement is seen with the phenols, except for 4-nitrophenol and 2,4-dinitrophenol, where the sonication recoveries are higher. Some

differences are observed with the polynuclear aromatic hydrocarbons (PAHs). Agreement is seen with many of the smaller PAHs (except 2-methylnaphthalene), but SFE appears to be more efficient for many of the larger PAHs, especially chrysene, 7,12-dimethylbenz(a)anthracene, and 3-methylcholanthrene. Other compounds with larger discrepancies (higher sonication yields) include α,α-dimethylphenethylamine, 4-chloroaniline, 4-aminobiphenyl, benzidine, and dibenz(a,j)acridine. Sonication is the method used at ANL for extraction of HEPA filters. However, SFE gives comparable extraction yields and would require less extraction solvent and produce less waste. In the current experiments, 75 mL of methylene chloride was used in the sonication extraction but only 15 mL for SFE. The ANL sonication method for extraction of waste HEPA filters generally uses 25 mL of solvent for a 1-g sample. There is thus a small reduction in solvent usage. In SFE, the analyte/collection solvent mixture is physically separated from the sample during extraction; thus, no filtration or sample rinsing is necessary following the extraction step.

A comparison of Soxhlet and SFE results is given in Figs. 13-18 for individual compounds and Fig. 19 for compound classes. Average recoveries for the two techniques are 82.7% and 70.2%, respectively. This difference is borne out by the data for the individual compounds. The average recovery for 88 out of the 98 compounds is higher with Soxhlet extraction. As mentioned above, SFE tends to be fairly efficient at extracting the higher molecular weight PAHs. In five of the 10 cases in which SFE yields are higher, the analyte is a PAH.

Soxhlet extraction is obviously more efficient at extracting semivolatile organic compounds from HEPA filters than SFE with unmodified CO_2. However, SFE reduces the extraction time by a factor of about 20-30 (30-45 min versus 16 h) and the extraction solvent

volume by a factor of 20-30 (10-15 mL versus 300 mL); these efficiencies would significantly reduce sample analysis costs.

Figures 20-25 compare the Soxtec and SFE recoveries for all 98 semivolatile compounds. Figure 26 compares the average recovery values for the compound classes. Average recoveries for SFE are higher for 87 out of the 98 compounds. The average recovery for all compounds is 62.9% for Soxtec and 70.2% for SFE. The only two compounds where Soxtec yields significantly higher recoveries are di-n-butylphthalate and bis(2-ethylhexyl)phthalate. It is quite likely that this is due to extract contamination rather than an increase in extraction efficiency. Significant differences are especially evident with the PAHs, starting with pyrene and continuing through the higher molecular weight species.

In addition to providing higher extraction yields, SFE also uses less solvent than automated Soxhlet extractions. The Soxtec extractions reported here used a total of 75 mL of solvent, five times more than SFE. Another advantage is that SFE is also faster. The extraction itself is 30-45 min versus 2 h for Soxtec, which also requires additional time to evaporate the excess solvent.

IV. CONCLUSIONS

The extraction of SW-846 Method 8270 acid, base, and neutral semivolatile organic compounds spiked onto blank HEPA filter material has been carried out using supercritical fluid extraction with unmodified CO_2. Optimum conditions have been determined for extraction with a Dionex SFE-703 instrument: the extraction is carried out at a temperature of 50°C and a pressure of 400 atm (40.5 MPa). The instrument has 250 mL/min restrictors that are held at a temperature of 150°C. The extraction is carried out dynamically for 30-45 min. The extracted compounds are collected by bubbling the CO_2 through 10-15 mL of collection solvent (methylene chloride, 1:1 hexane/acetone, and 1:1 methylene chloride/acetone all work equally well) held at 0°C.

Supercritical fluid extraction of the semivolatile organics has been compared to the conventional extraction techniques of Soxhlet, automated Soxhlet (Soxtec), and sonication. The SFE technique is seen to give higher extraction yields than Soxtec and is comparable to sonication. Soxhlet extraction gives the highest recoveries of all of the techniques. Average recoveries for the 98 semivolatile compounds are 82.7% for Soxhlet, 74.0% for sonication, 70.2% for SFE, and 62.9% for Soxtec.

The SFE approach reduces the volume of hazardous solvents used and thus the amount of waste produced. In the current study, 75 mL of solvent was necessary for both sonication and Soxtec extraction, while 300 mL of solvent was used for the Soxhlet extractions. If 15 mL of solvent is used for analyte collection, SFE reduces the solvent usage by a factor of between 5 and 20. This factor can be increased if the amount of solvent is reduced to 10 mL.

In terms of extraction times, SFE has a clear advantage over Soxhlet (30-45 min versus 16-18 h). It is also faster than Soxtec, which requires 2 h for the extraction. Sonication sample

extraction times are fairly comparable if only one extraction is done at a time. Because the instrument used here can perform eight extractions at a time, SFE would be more efficient than sonication.

ACKNOWLEDGMENTS

The author wishes to thank Yifen Tsai for her contribution to this work. Thanks also go to Jimmy Harris of Flanders Filters, Inc., who supplied the blank HEPA filter material. This work was performed for the U. S. Department of Energy, Office of Pollution Prevention (EM-77) under Contract W-31-109-Eng-38.

REFERENCES

1. K. A. Gablin, *Solutions for Dioctyl Phthalate (DOP) Tested High Efficiency Particulate Air (HEPA) Filters Destined for Disposal at Hanford, Washington*, Argonne National Laboratory Report ANL/EWM/CP--78035 (Nov. 1992).

2. *Test Methods for Evaluating Solid Waste*, U.S. Environmental Protection Agency, Document SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC (Sept. 1994).

3. V. Lopez-Avila and N. S. Dodhiwala, *Method for the Supercritical Fluid Extraction of Soils/Sediments*, U.S. Environmental Protection Agency Report EPA/600/4-90/026, Washington, DC (1990).

4. V. Lopez-Avila, W. F. Beckert, and S. Billets, "Supercritical Fluid Extraction and Its Application to Environmental Analysis," *Waste Testing and Quality Assurance: Third Volume,* ASTM STP 1075, D. Friedman, Ed., American Society for Testing and Materials, Philadelphia, PA (1991).

5. H. Engelhardt, J. Zapp, and P. Kolla, "Sample Preparation by Supercritical Fluid Extraction in Environmental, Food, and Polymer Analysis," Chromatographia 32, 527-537, (1991).

6. V. Lopez-Avila and W. F. Beckert, "Supercritical Fluid Extraction in Environmental Analysis," *Supercritical Fluid Technology: Theoretical and Applied Approaches to Analytical Chemistry*, F. V. Bright and M. E. P. McNally, Eds., ACS Symposium Series 488, American Chemical Society, Washington, DC (1992).

7. L. J. D. Myer, J. H. Damian, P. B. Liescheski, and J. Tehrani, "Supercritical Fluid Extraction versus Soxhlet Sample Preparation: A Comparative Study for Extraction and Analysis of Organics in Solid Matrices," *Supercritical Fluid Technology: Theoretical and Applied Approaches to Analytical Chemistry*, F. V. Bright and M. E. P. McNally, Eds., ACS Symposium Series 488, American Chemical Society, Washington, DC (1992).

8. S. B. Hawthorne, D. J. Miller, J. J. Langenfeld, and M. D. Burford, "Analytical-Scale Extraction of Environmental Samples Using Supercritical Fluids," *Pollution Prevention in Industrial Processes: The Role of Process Analytical Chemistry*, J. J. Breen and M. J. Dellarco, Eds., ACS Symposium Series 508, American Chemical Society, Washington, DC (1992).

9. T. S. Oostdyk, R. L. Grob, J. L. Snyder, and M. E. McNally, "Study of Sonication and Supercritical Fluid Extraction of Primary Aromatic Amines," Anal. Chem. 65, 596-600, (1993).

10. M. D. Burford, S. B. Hawthorne, and D. J. Miller, "Extraction Rates of Spiked versus Native PAHs from Heterogeneous Environmental Samples Using Supercritical Fluid Extraction and Sonication in Methylene Chloride," Anal. Chem. 65, 1497-1505, (1993).

11. V. Camel, A. Tambuté, and M. Caude, "Analytical-Scale Supercritical Fluid Extraction: A Promising Technique for the Determination of Pollutants in Environmental Matrices," J. Chromatogr. 642, 263-281, (1993).

12. V. Janda, K. D. Bartle, and A. A. Clifford, "Supercritical Fluid Extraction in Environmental Analysis," J. Chromatogr. 642, 283-299, (1993).

13. S. B. Hawthorne, D. J. Miller, M. D. Burford, J. J. Langenfeld, S. Eckert-Tilotta, and P. K. Louie, "Factors Controlling Quantitative Supercritical Fluid Extraction of Environmental Samples," J. Chromatogr. 642, 301-317, (1993).

14. M. I. L. Bicking, T. G. Hayes, J. C. Kiley, and S. N. Deming, "An Experimental Design Approach to the Optimization of Supercritical Fluid Extraction for the Determination of Oil and Grease in Soil," J. Chromatogr. Sci. 31, 170-176, (1993).

15. T. S. Oostdyk, R. L. Grob, J. L. Snyder, and M. E. McNally, "Optimization of the Supercritical Fluid Extraction of Primary Aromatic Amines," J. Chromatogr. Sci. 31, 177-182, (1993).

16. D. R. Gere, C. R. Knipe, P. Castelli, J. Hedrick, L. G. Randall Frank, H. Schulenberg-Schell, R. Schuster, L. Doherty, J. Orolin, and H. B. Lee, "Bridging the Automation Gap between Sample Preparation and Analysis: An Overview of SFE, GC-MS, and HPLC Applied to Environmental Samples," J. Chromatogr. Sci. 31, 246-258, (1993).

17. T. L. Chester, J. D. Pinkston, and D. E. Raynie, "Supercritical Fluid Chromatography and Extraction," Anal. Chem. 66, 106R-130R, (1994).

18. S. Reindl and F. Höfler, "Optimization of the Parameters in Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons from Soil Samples," Anal. Chem. 66, 1808-1816, (1994).

19. S. B. Hawthorne, Y. Yang, and D. J. Miller, "Extraction of Organic Pollutants from Environmental Solids with Sub- and Supercritical Water," Anal. Chem. 66, 2912-2920, (1994).

20. S. B. Hawthorne and D. J. Miller, "Direct Comparison of Soxhlet and Low- and High-Temperature Supercritical CO_2 Extraction Efficiencies of Organics from Environmental Solids," Anal. Chem. 66, 4005-4012, (1994).

21. J. J. Langenfeld, S. B. Hawthorne, D. J. Miller, and J. Pawliszyn, "Role of Modifiers for Analytical-Scale Supercritical Fluid Extraction of Environmental Samples," Anal. Chem. 66, 909-916, (1994).

22. Y. Yang, A. Gharaibeh, S. B. Hawthorne, and D. J. Miller, "Combined Temperature/Modifier Effects on Supercritical CO_2 Extraction Efficiencies of Polycyclic Aromatic Hydrocarbons from Environmental Samples," Anal. Chem. 67, 641-646, (1995).

23. A. M. Essling, L. E. Ross, F. P. Smith, I. M. Fox, E. A. Huff, D. R. Huff, P. Melnikov, S. D. Kent, K. J. Parish, Y. Tsai, L. L. Chromizky, P. D. Postlethwait, E. Vinokour, L. B. TenKate, T. TenKate, J. S. Yaeger, L. L. Smith, F. Markun, D. L. Bowers, A. S. Boparai, and D. G. Graczyk, "Characterization of Used HEPA Filters for Disposal," *Analytical Chemistry Laboratory Progress Report for FY 1994*, Argonne National Laboratory Report ANL/ACL-94/4 (Dec. 1994).

Table 1. RCRA Method 8270 Base/Neutral/Acid Analytes Used in This Study

Alkylphenols	
Phenol	4-Methylphenol
Phenol-d_5[a]	2,4-Dimethylphenol
2-Methylphenol	
Halophenols	
2-Fluorophenol[a]	2,4,5-Trichlorophenol
2-Chlorophenol	2,4,6-Trichlorophenol
4-Chloro-3-methylphenol	2,3,4,6-Tetrachlorophenol
2,4-Dichlorophenol	Pentachlorophenol
2,6-Dichlorophenol	2,4,6-Tribromophenol
Nitrophenols	
2-Nitrophenol	2,4-Dinitrophenol
4-Nitrophenol	4,6-Dinitro-2-methylphenol
Carboxylic acid	
Benzoic acid	
Alcohol	
Benzyl alcohol	
Ketones	
Acetophenone	Isophorone
Ethers	
bis(2-Chloroethyl)ether	Dibenzofuran
bis(2-Chloroisopropyl)ether	4-Chlorophenyl phenyl ether
bis(2-Chloroethoxy)methane	4-Bromophenyl phenyl ether
Sulfonates	
Methyl methanesulfonate	Ethyl methanesulfonate

Table 1 (contd)

PAHs	
Acenaphthene	7,12-Dimethylbenz(a)anthracene
Acenaphthylene	Fluoranthene
Anthracene	Fluorene
Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	3-Methylcholanthrene
Benzo(k)fluoranthene	2-Methylnaphthalene
Benzo(g,h,i)perylene	Naphthalene
Benzo(a)pyrene	Phenanthrene
Chrysene	Pyrene
Dibenzo(a,h)anthracene	Terphenyl-d_{14}[a]
Phthalates	
Dimethylphthalate	Butylbenzylphthalate
Diethylphthalate	bis(2-Ethylhexyl)phthalate
Di-n-butylphthalate	Di-n-octylphthalate
Halocarbons	
2-Fluorobiphenyl[a]	1,2,4,5-Tetrachlorobenzene
1-Chloronaphthalene	Pentachlorobenzene
2-Chloronaphthalene	Hexachlorobenzene
1,2-Dichlorobenzene	Hexachloroethane
1,3-Dichlorobenzene	Hexachlorobutadiene
1,4-Dichlorobenzene	Hexachlorocyclopentadiene
1,2,4-Trichlorobenzene	
Amides	
Phenacetin	Pronamide
Pyridines	
2-Picoline	Dibenz(a,j)acridine

Table 1 (contd)

Aromatic Amines	
4-Aminobiphenyl	Diphenylamine
Aniline	1-Naphthylamine
Benzidine	2-Naphthylamine
4-Chloroaniline	2-Nitroaniline
3,3'-Dichlorobenzidine	3-Nitroaniline
α,α-Dimethylphenethylamine	4-Nitroaniline
Nitrosamines	
N-Nitrosodimethylamine	N-Nitrosodiphenylamine
N-Nitrosodi-n-propylamine	N-Nitrosopiperidine
N-Nitrosodi-n-butylamine	
Hydrazine	
1,2-Diphenylhydrazine	
Azoamine	
p-Dimethylaminoazobenzene	
Nitrobenzenes	
Nitrobenzene	2,6-Dinitrotoluene
Nitrobenzene-d_5[a]	Pentachloronitrobenzene
2,4-Dinitrotoluene	

[a]Surrogate spiking standards.

Table 2. Average Semivolatile Recoveries versus Collection Solvent and Solvent Volume for SFE of Spiked Sand

Solvent	Average Percent Recovery	
	10 mL Solvent	15 mL Solvent
1:1 Hexane:acetone	64.7	64.8
1:1 Methylene chloride:acetone	59.6	62.6
Methylene chloride	63.4	63.0

Table 3. Comparison of Average SFE Recoveries for 30- and 45-min Extractions with Methylene Chloride and Hexane/Acetone Extraction Solvents

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
Alkylphenols						
Phenol-d_5	69	6	72	9	72	18
Phenol	68	5	74	5	67	17
2-Methylphenol	65	6	71	8	66	15
4-Methylphenol	67	4	73	10	71	10
2,4-Dimethylphenol	68	6	74	8	68	11
Halophenols						
2-Fluorophenol	60	8	59	12	54	40
2-Chlorophenol	59	6	69	9	57	30
2,4-Dichlorophenol	73	5	84	8	76	9
2,6-Dichlorophenol	75	6	78	9	74	9
4-Chloro-3-methylphenol	80	4	81	10	78	10
2,4,6-Trichlorophenol	76	5	85	13	82	8
2,4,5-Trichlorophenol	79	4	92	7	87	5
2,3,4,6-Tetrachlorophenol	73	3	95	6	89	8
2,4,6-Tribromophenol	76	3	93	11	92	4
Pentachlorophenol	74	5	93	6	90	9

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
Nitrophenols						
2-Nitrophenol	65	8	68	14	58	25
2,4-Dinitrophenol	70	6	109	17	112	18
4-Nitrophenol	77	5	75	19	87	9
4,6-Dinitro-2-methylphenol	80	3	107	19	119	16
Phthalates						
Dimethylphthalate	81	5	84	7	81	10
Diethylphthalate	83	4	82	12	81	7
Di-n-butylphthalate	85	3	80	18	82	10
Butylbenzylphthalate	98	3	83	16	86	3
bis(2-Ethylhexyl)phthalate	102	5	82	19	85	3
Di-n-octylphthalate	103	6	89	13	89	8
Ketones						
Acetophenone	62	4	57	18	49	28
Isophorone	73	5	67	10	61	19
Sulfonates						
Methyl methanesulfonate	47	5	47	21	36	39
Ethyl methanesulfonate	60	5	52	17	45	30

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
PAHs						
Naphthalene	70	5	61	14	54	26
2-Methylnaphthalene	73	4	69	11	62	17
Acenaphthylene	80	5	75	9	77	7
Acenaphthene	79	5	78	8	77	7
Fluorene	72	4	82	8	79	8
Phenanthrene	82	4	82	11	85	4
Anthracene	81	3	81	10	82	7
Fluoranthene	85	3	87	10	85	8
Pyrene	94	5	85	12	82	5
Terphenyl-d_{14}	93	4	89	9	88	8
Benzo(a)anthracene	91	2	93	7	92	3
Chrysene	91	2	89	10	92	3
Benzo(b)fluoranthene	93	3	96	14	97	15
7,12-Dimethylbenz(a)anthracene	87	3	100	23	97	26
Benzo(k)fluoranthene	90	3	95	8	95	7
Benzo(a)pyrene	90	2	96	11	96	10
3-Methylcholanthrene	79	3	92	12	90	5

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
PAHs (contd)						
Indeno(1,2,3-cd)pyrene	96	3	94	18	91	13
Dibenzo(a,h)anthracene	95	3	95	16	91	10
Benzo(g,h,i)perylene	94	5	88	18	88	12
Halocarbons						
1,3-Dichlorobenzene	26	20	26	65	15	75
1,4-Dichlorobenzene	30	18	29	58	17	71
1,2-Dichlorobenzene	37	14	34	48	22	63
Hexachloroethane	30	18	27	63	18	70
1,2,4-Trichlorobenzene	58	7	60	24	45	36
Hexachlorobutadiene	44	8	50	35	35	47
1,2,4,5-Tetrachlorobenzene	38	11	57	21	40	31
Hexachlorocyclopentadiene	69	4	79	14	68	17
2-Fluorobiphenyl	80	5	71	11	78	11
2-Chloronaphthalene	77	7	79	19	70	12
1-Chloronaphthalene	78	6	68	8	75	14
Pentachlorobenzene	70	4	87	11	81	6
Hexachlorobenzene	73	3	85	4	88	6

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
Ethers						
bis(2-Chloroethyl)ether	52	10	40	31	32	47
bis(2-Chloroisopropyl)ether	49	11	37	30	31	47
bis(2-Chloroethoxy)methane	66	5	63	11	57	19
Dibenzofuran	78	4	78	7	78	6
4-Chlorophenyl phenyl ether	74	5	83	8	77	7
4-Bromophenyl phenyl ether	73	5	81	6	84	6
Carboxylic acid						
Benzoic acid	68	9	76	20	75	25
Alcohol						
Benzyl alcohol	70	4	70	10	64	20
Nitrosamines						
N-Nitrosodimethylamine	26	22	26	63	15	64
N-Nitroso-di-n-propylamine	63	5	56	14	51	25
N-Nitrosopiperidine	76	3	71	10	63	19
N-Nitroso-di-n-butylamine	77	5	73	10	69	14
N-Nitrosodiphenylamine	79	3	77	16	84	10

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
Aromatic amines						
Aniline	45	6	53	17	46	32
α,α-Dimethylphenethylamine	35	21	26	37	35	24
4-Chloroaniline	30	11	76	6	70	8
2-Nitroaniline	83	4	81	17	84	11
3-Nitroaniline	63	4	89	9	87	8
2-Naphthylamine	44	5	77	5	78	8
1-Naphthylamine	42	6	84	7	94	6
4-Nitroaniline	79	4	91	8	91	6
Diphenylamine	79	3	77	16	84	10
4-Aminobiphenyl	37	4	82	8	86	5
Benzidine	20	28	54	20	67	24
3,3'-Dichlorobenzidine	41	10	89	4	91	2
Azoamine						
p-Dimethylaminoazobenzene	95	3	86	11	84	6
Amides						
Phenacetin	88	2	78	18	87	10
Pronamide	80	4	82	14	86	9

Table 3 (contd)

Compound	45 min, Hex/Ace[a]		30 min, MC[b]		45 min, MC[b]	
	% Recovery	% RSD[c]	% Recovery	% RSD[c]	% Recovery	% RSD[c]
Pyridines						
2-Picoline	36	14	35	44	23	50
Dibenz(a,j)acridine	55	27	48	23	58	19
Hydrazine						
1,2-Diphenylhydrazine	78	6	72	22	79	7
Nitrobenzenes						
Nitrobenzene-d_5	65	6	41	38	52	31
Nitrobenzene	62	6	58	21	49	30
2,6-Dinitrotoluene	83	4	87	10	90	11
2,4-Dinitrotoluene	91	3	95	9	92	10
Pentachloronitrobenzene	83	3	87	11	92	12

[a]Hex/Ace = 1:1 hexane/acetone.
[b]MC = methylene chloride.
[c]RSD = relative standard deviation

Table 4. Comparison of Compound Recoveries between SFE and Traditional Extraction Techniques

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
Alkylphenols								
Phenol-d_5	71	12	78	36	66	14	79	4
Phenol	69	11	76	21	56	20	67	7
2-Methylphenol	67	11	75	10	58	12	66	4
4-Methylphenol	71	9	74	9	69	20	71	5
2,4-Dimethylphenol	70	9	52	47	65	12	60	16
Halophenols								
2-Fluorophenol	57	24	75	23	48	56	63	12
2-Chlorophenol	61	20	79	18	48	34	61	7
2,4-Dichlorophenol	78	10	95	5	71	14	76	3
2,6-Dichlorophenol	76	8	81	36	75	16	79	9
4-Chloro-3-methylphenol	80	8	91	8	81	15	88	3
2,4,6-Trichlorophenol	81	10	105	13	76	12	89	9
2,4,5-Trichlorophenol	86	8	103	12	85	13	83	3
2,3,4,6-Tetrachlorophenol	86	12	99	14	82	12	88	4
2,4,6-Tribromophenol	88	11	103	18	95	11	100	5
Pentachlorophenol	86	12	97	20	84	16	92	6

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
Nitrophenols								
2-Nitrophenol	64	18	76	29	57	29	75	5
2,4-Dinitrophenol	98	25	106	15	74	64	126	5
4-Nitrophenol	80	13	98	15	93	20	110	4
4,6-Dinitro-2-methylphenol	103	22	101	8	79	35	107	4
Phthalates								
Dimethylphthalate	82	8	96	5	80	11	81	4
Diethylphthalate	82	8	93	11	80	10	82	4
Di-n-butylphthalate	82	11	100	19	108	18	88	6
Butylbenzylphthalate	89	11	95	17	84	15	82	6
bis(2-Ethylhexyl)phthalate	89	14	116	19	121	11	81	5
Di-n-octylphthalate	93	11	89	18	76	12	86	5
Ketones								
Acetophenone	55	21	72	14	46	41	62	8
Isophorone	66	14	84	4	59	23	75	5
Sulfonates								
Methyl methanesulfonate	43	26	65	29	42	59	56	15
Ethyl methanesulfonate	51	22	72	21	46	40	59	7

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
PAHs								
Naphthalene	61	19	79	15	46	18	65	9
2-Methylnaphthalene	68	13	90	9	70	15	87	4
Acenaphthylene	77	7	89	6	65	6	71	1
Acenaphthene	78	6	91	7	74	8	83	5
Fluorene	78	8	93	9	78	7	82	6
Phenanthrene	83	7	92	11	73	6	79	6
Anthracene	82	7	90	11	80	7	84	2
Fluoranthene	85	7	93	8	83	4	87	4
Pyrene	87	9	90	11	74	9	74	4
Terphenyl-d_{14}	90	7	97	5	78	10	78	4
Benzo(a)anthracene	92	5	101	4	79	9	81	4
Chrysene	91	6	98	2	71	9	72	5
Benzo(b)fluoranthene	95	12	88	9	66	9	80	5
7,12-Dimethylbenz(a)anthracene	95	22	83	14	60	6	63	7
Benzo(k)fluoranthene	93	7	91	7	66	10	79	8
Benzo(a)pyrene	94	9	92	3	69	12	78	7
3-Methylcholanthrene	87	10	72	10	57	12	55	6

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
PAHs (contd)								
Indeno(1,2,3-cd)pyrene	94	13	100	8	75	14	90	7
Dibenzo(a,h)anthracene	94	11	97	8	76	14	96	6
Benzo(g,h,i)perylene	90	13	95	8	72	12	85	5
Halocarbons								
1,3-Dichlorobenzene	22	59	37	93	12	109	18	46
1,4-Dichlorobenzene	25	54	39	88	13	108	19	41
1,2-Dichlorobenzene	30	46	45	75	17	105	26	36
Hexachloroethane	25	54	37	91	17	108	24	42
1,2,4-Trichlorobenzene	54	27	71	27	36	57	53	12
Hexachlorobutadiene	42	36	61	36	32	82	45	17
1,2,4,5-Tetrachlorobenzene	45	29	36	49	39	31	53	12
Hexachlorocyclopentadiene	72	14	94	8	64	19	80	6
2-Fluorobiphenyl	77	10	98	5	73	14	82	8
2-Chloronaphthalene	75	14	99	13	76	12	91	8
1-Chloronaphthalene	74	11	82	14	60	18	63	10
Pentachlorobenzene	79	11	97	14	72	10	79	6
Hexachlorobenzene	82	9	92	11	73	10	78	3

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
Ethers								
bis(2-Chloroethyl)ether	41	35	57	47	28	80	43	20
bis(2-Chloroisopropyl)ether	38	35	58	48	28	82	41	20
bis(2-Chloroethoxy)methane	62	14	82	7	57	25	68	5
Dibenzofuran	78	6	84	6	71	10	76	3
4-Chlorophenyl phenyl ether	78	8	89	9	74	9	78	5
4-Bromophenyl phenyl ether	80	8	91	9	72	6	72	8
Carboxylic acid								
Benzoic acid	73	20	89	14	49	53	86	7
Alcohol								
Benzyl alcohol	68	14	84	13	62	17	70	2
Nitrosamines								
N-Nitrosodimethylamine	22	55	44	78	25	102	20	51
N-Nitroso-di-n-propylamine	56	18	72	15	51	39	63	7
N-Nitrosopiperidine	70	14	82	10	59	26	71	5
N-Nitroso-di-n-butylamine	73	11	86	9	68	17	74	5
N-Nitrosodiphenylamine	80	11	89	19	78	7	82	5

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]	% Recovery	% RSD[a]
Aromatic amines								
Aniline	48	22	93	43	31	23	56	11
α,α-Dimethylphenethylamine	32	28	53	28	34	53	70	6
4-Chloroaniline	60	35	90	3	55	36	81	4
2-Nitroaniline	83	12	93	12	80	10	85	4
3-Nitroaniline	80	16	98	6	66	22	84	2
2-Naphthylamine	67	25	85	16	49	62	72	6
1-Naphthylamine	75	31	94	13	60	44	83	5
4-Nitroaniline	87	9	97	6	76	21	91	4
Diphenylamine	80	11	87	19	78	7	82	5
4-Aminobiphenyl	70	33	82	7	66	34	88	6
Benzidine	48	48	56	38	49	82	71	8
3,3'-Dichlorobenzidine	75	31	98	23	66	17	79	5
Azoamine								
p-Dimethylaminoazobenzene	88	9	96	4	75	14	74	3
Amides:								
Phenacetin	85	12	92	22	82	11	91	5
Pronamide	83	10	95	14	80	8	83	3

Table 4 (contd)

Compound	SFE		Soxhlet		Soxtec		Sonication	
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Pyridines								
2-Picoline	31	41	31	56	22	86	38	22
Dibenz(a,j)acridine	54	23	81	10	68	16	86	6
Hydrazine								
1,2-Diphenylhydrazine	77	13	86	21	76	11	79	13
Nitrobenzenes								
Nitrobenzene-d_5	53	30	75	22	46	45	62	8
Nitrobenzene	56	22	69	23	44	44	59	9
2,6-Dinitrotoluene	87	9	93	6	77	13	80	4
2,4-Dinitrotoluene	93	8	92	6	78	14	85	4
Pentachloronitrobenzene	88	10	103	13	80	8	83	5

[a]RSD = relative standard deviation

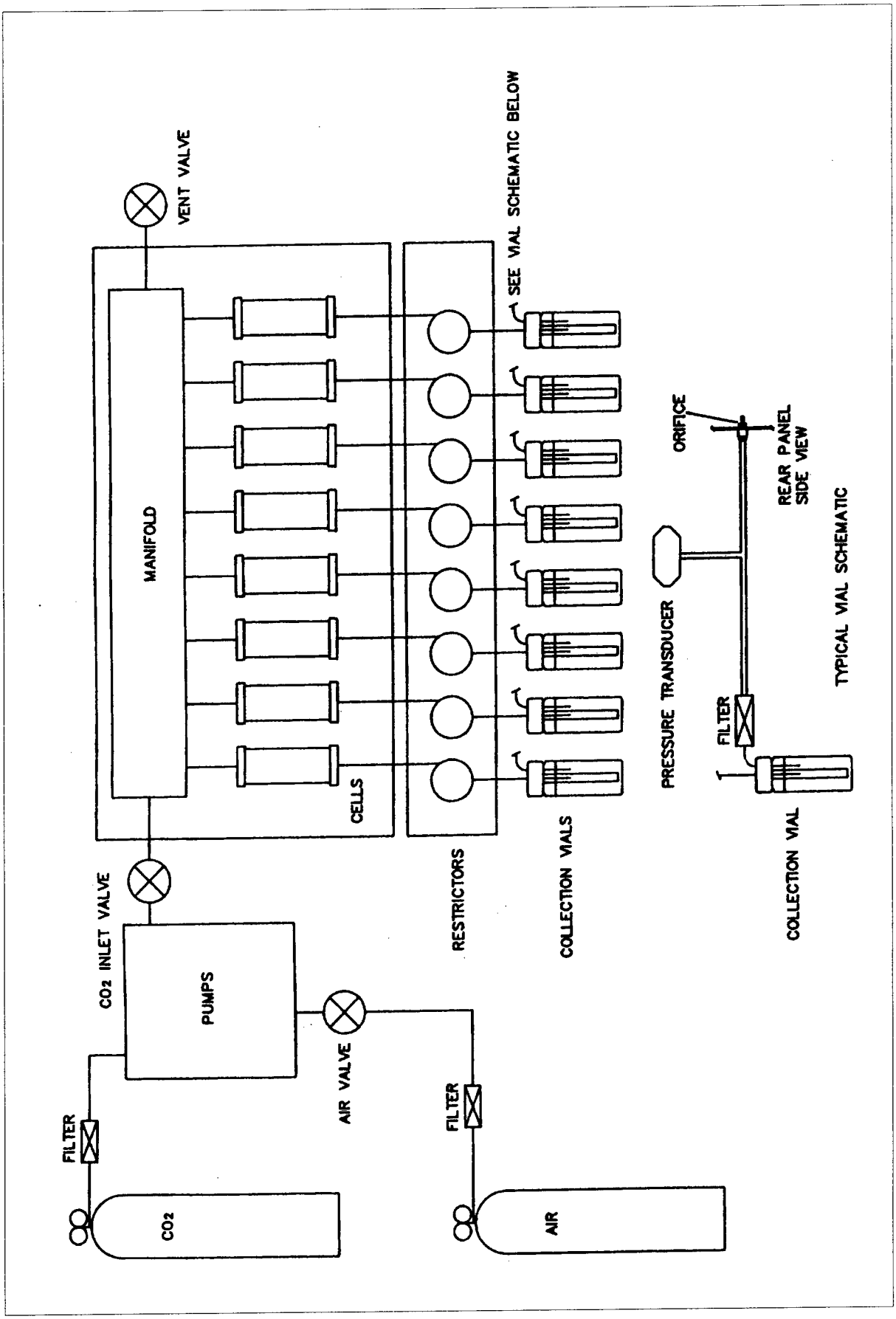

Fig. 1. Schematic Diagram of the Dionex SFE-703 Supercritical Fluid Extractor

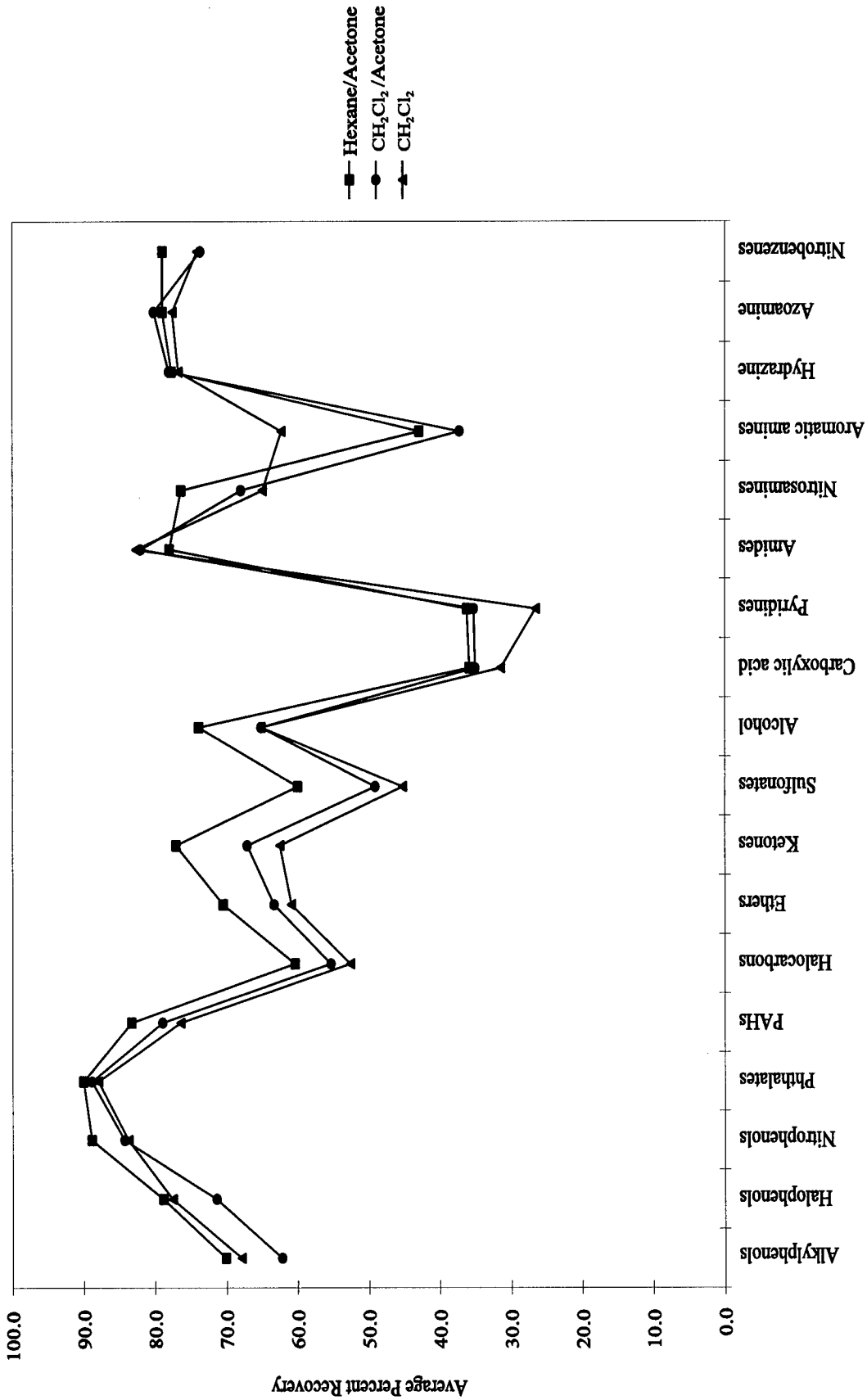

Fig. 2. Average Percent Recoveries Using 15 mL of Hexane/Acetone, Methylene Chloride/Acetone, or Methylene Chloride as the SFE Collection Solvent

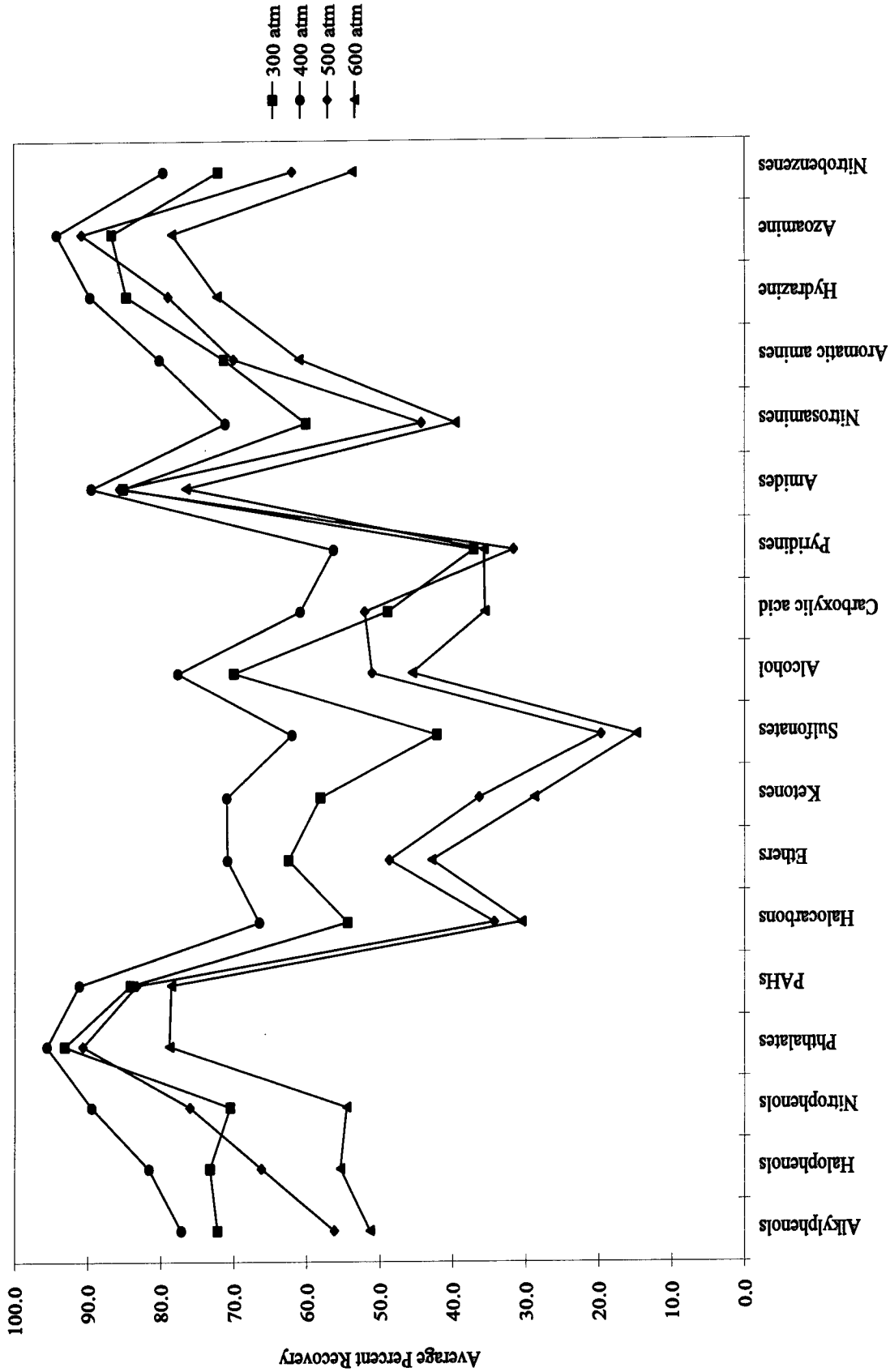

Fig. 3. Average Percent Recoveries versus Pressure for the Extraction of Semivolatiles from HEPA Filters Using SFE at 50°C

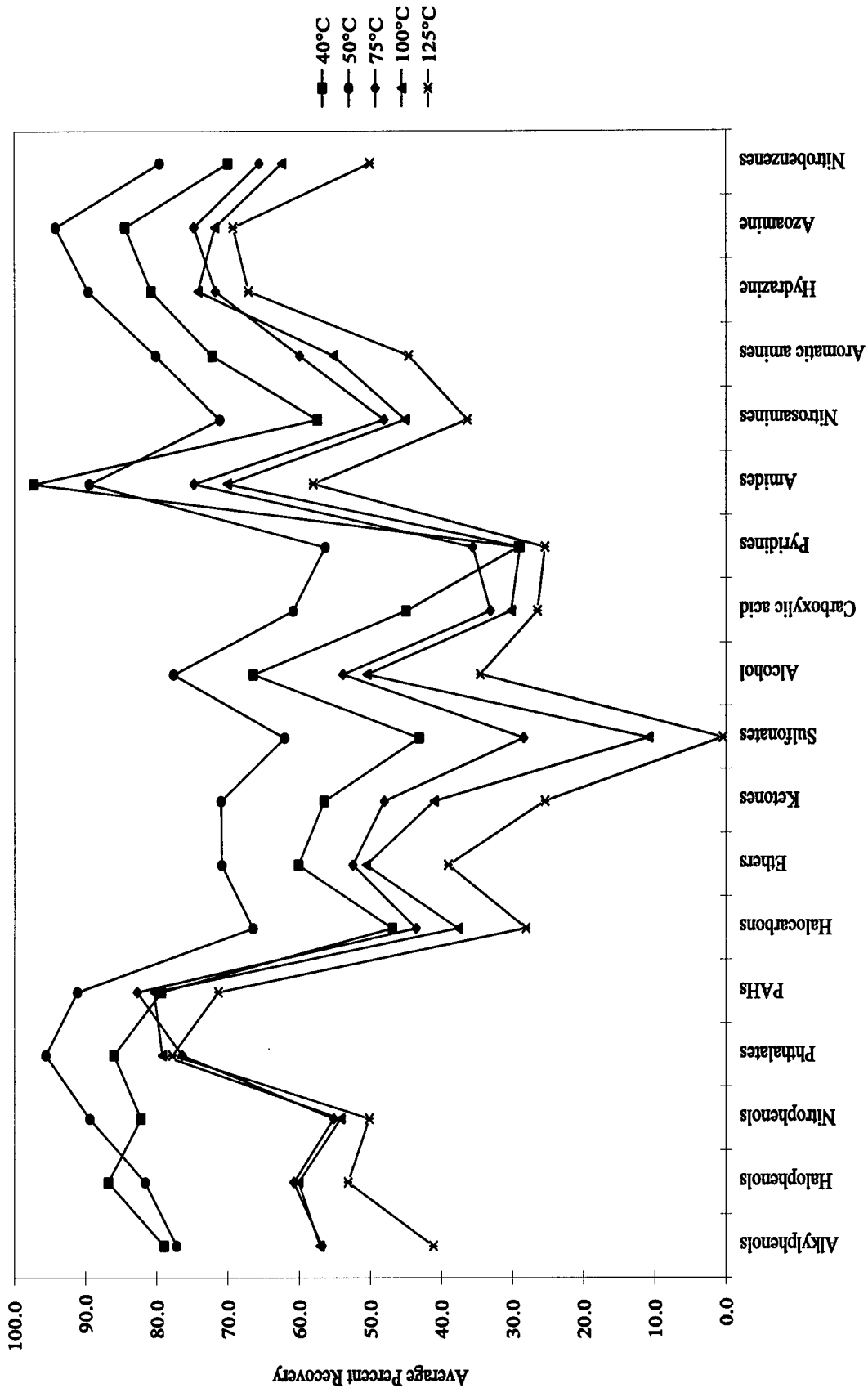

Fig. 4. Comparison of Average Recoveries versus Temperature for the Extraction of Semivolatiles from HEPA Filters Using SFE at 400 atm

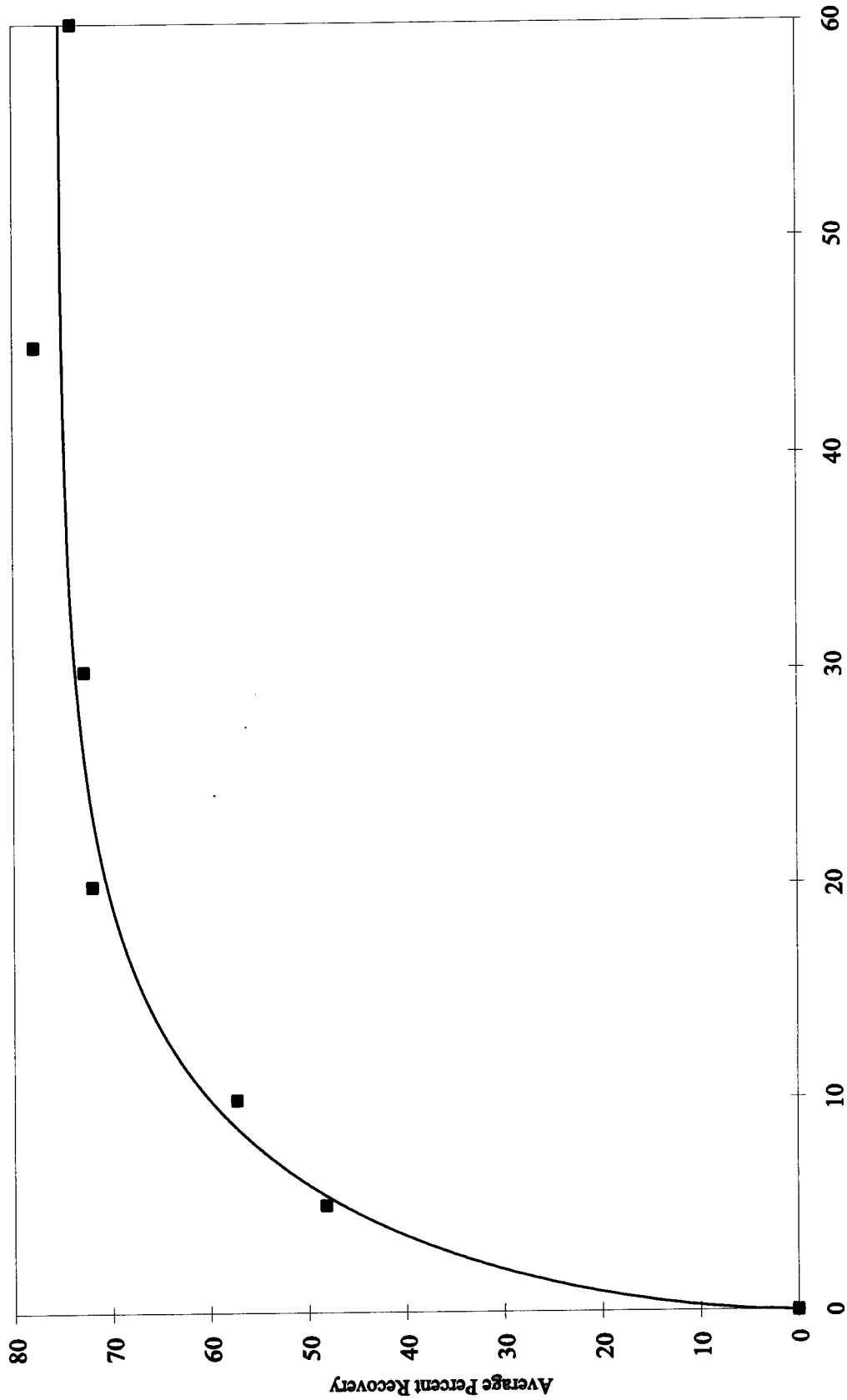

Fig. 5. Variation of Average Percent Recovery across All Compounds with SFE Extraction Time

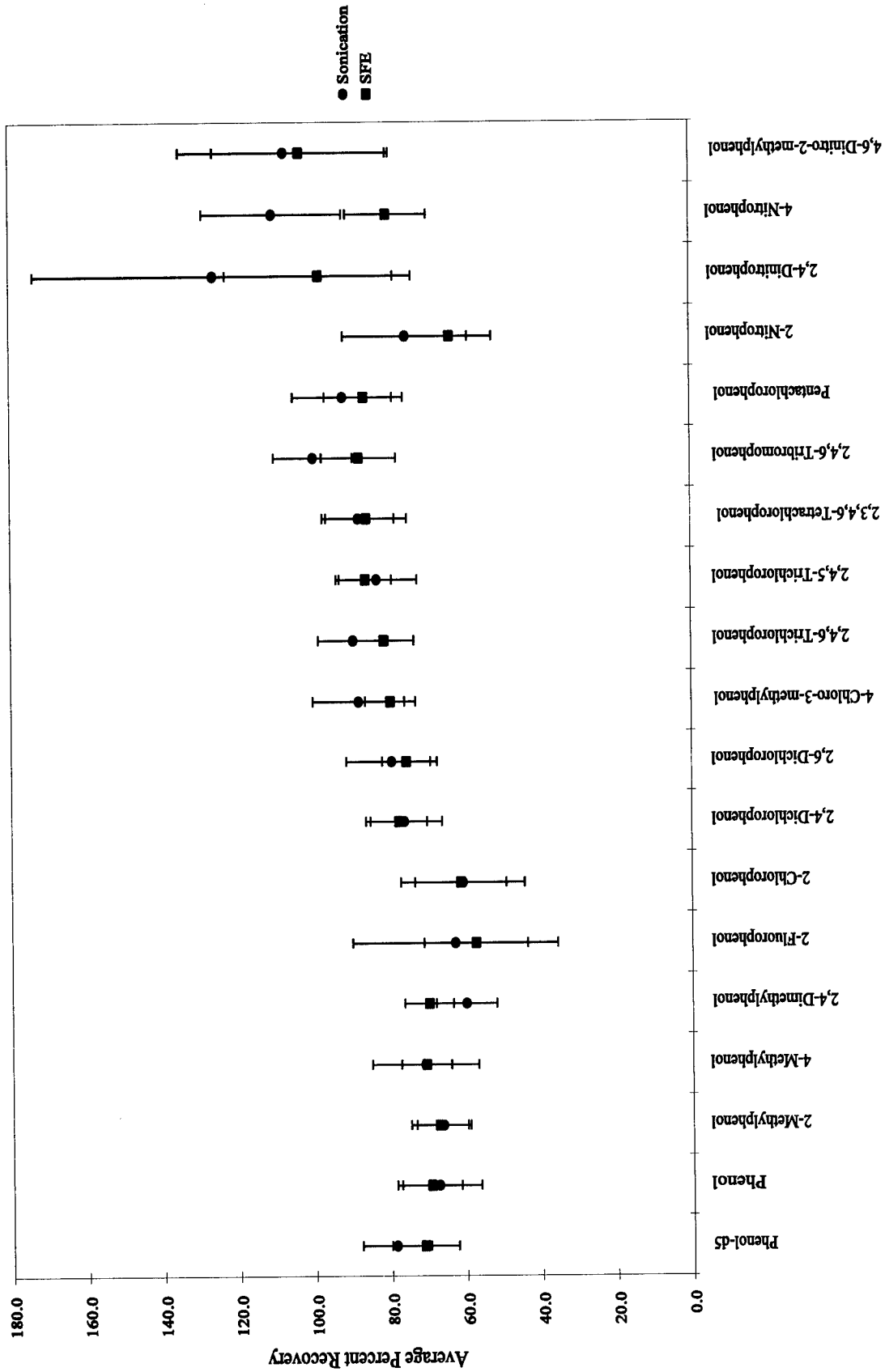

Fig. 6. Comparison between Sonication and SFE for the Extraction of Phenols from HEPA Filters

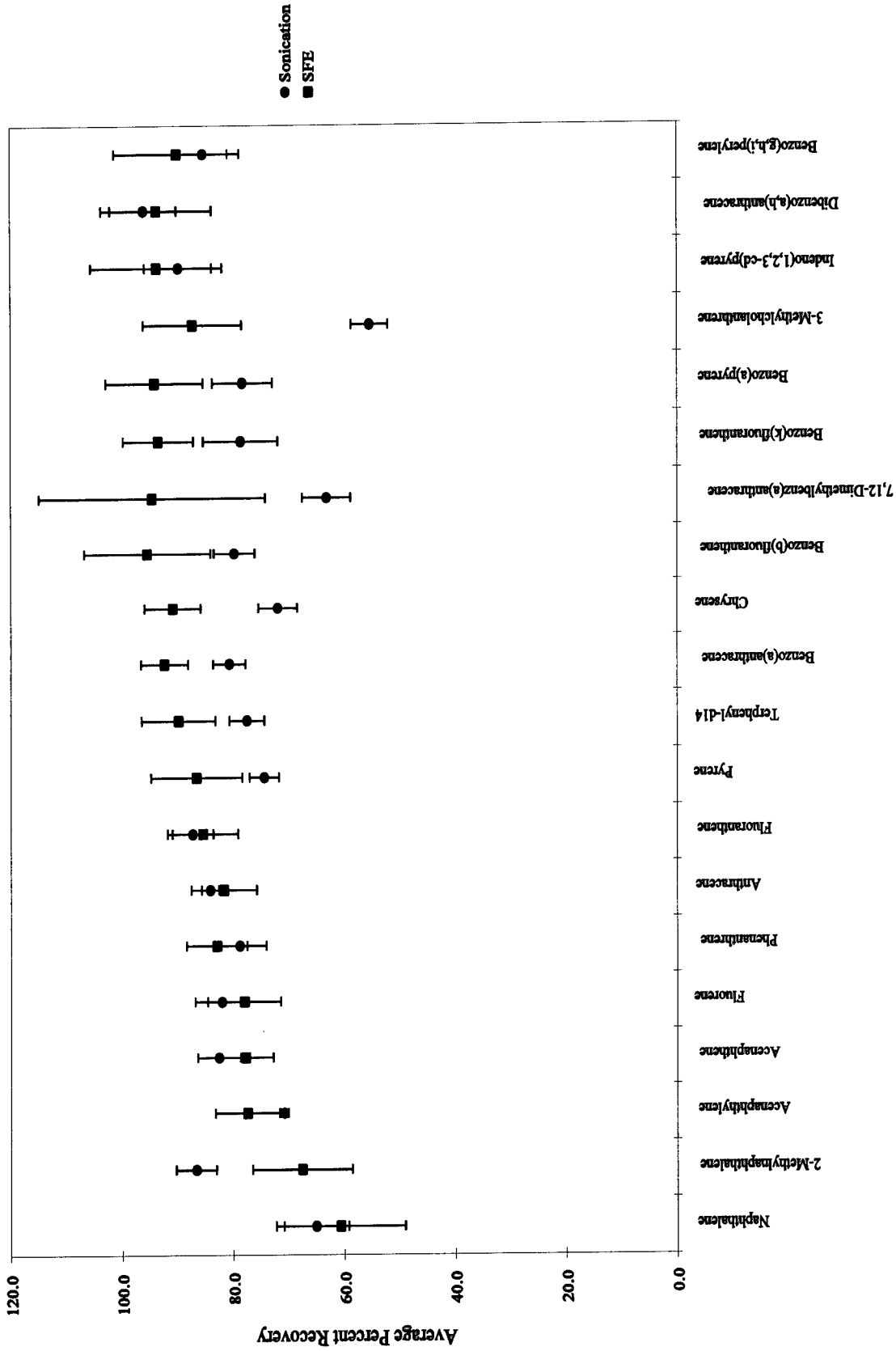

Fig. 7. Comparison between Sonication and SFE for the Extraction of PAHs from HEPA Filters

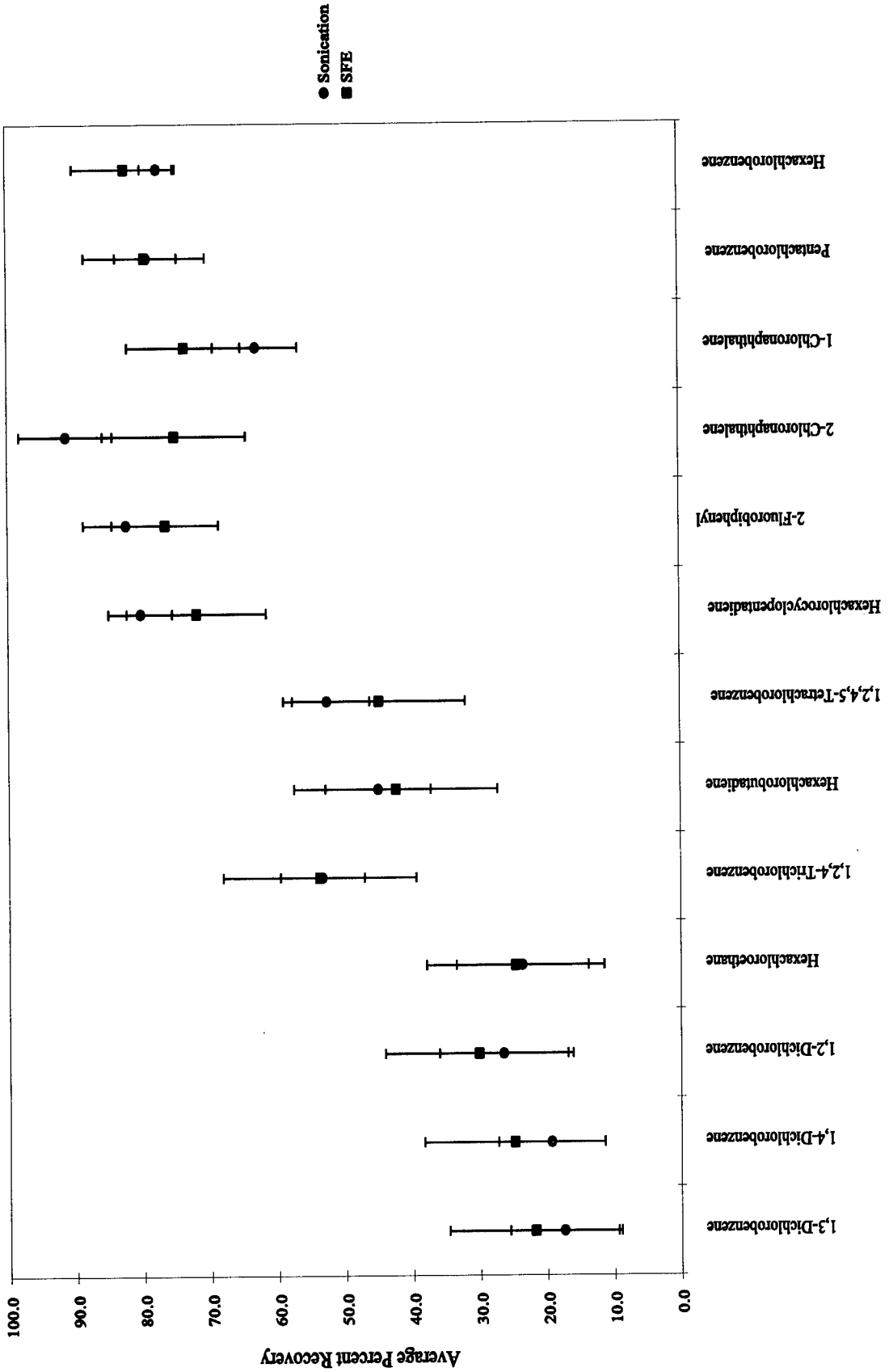

Fig. 8. Comparison between Sonication and SFE for the Extraction of Halocarbons from HEPA Filters

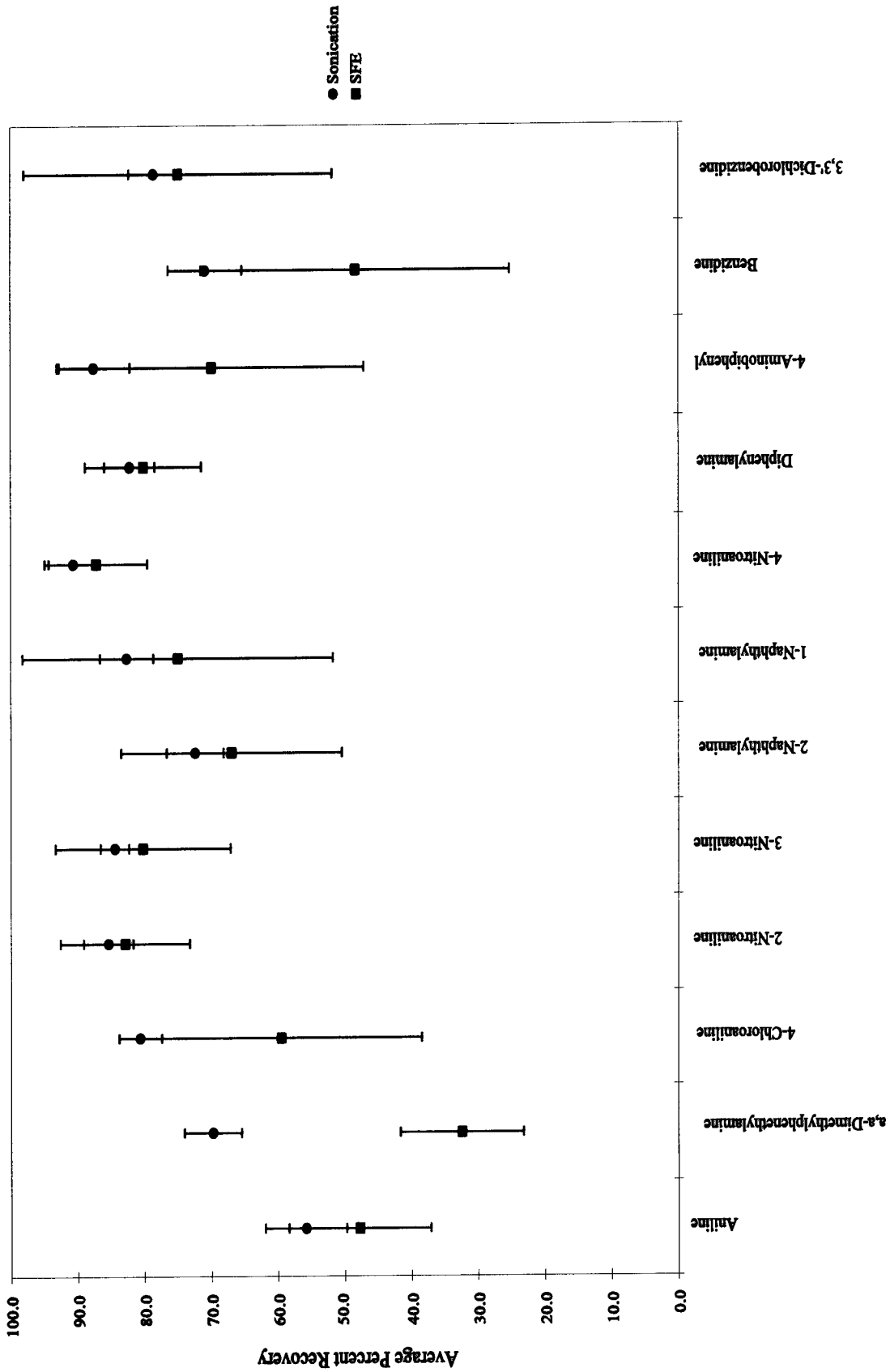

Fig. 9. Comparison between Sonication and SFE for the Extraction of Aromatic Amines from HEPA Filters

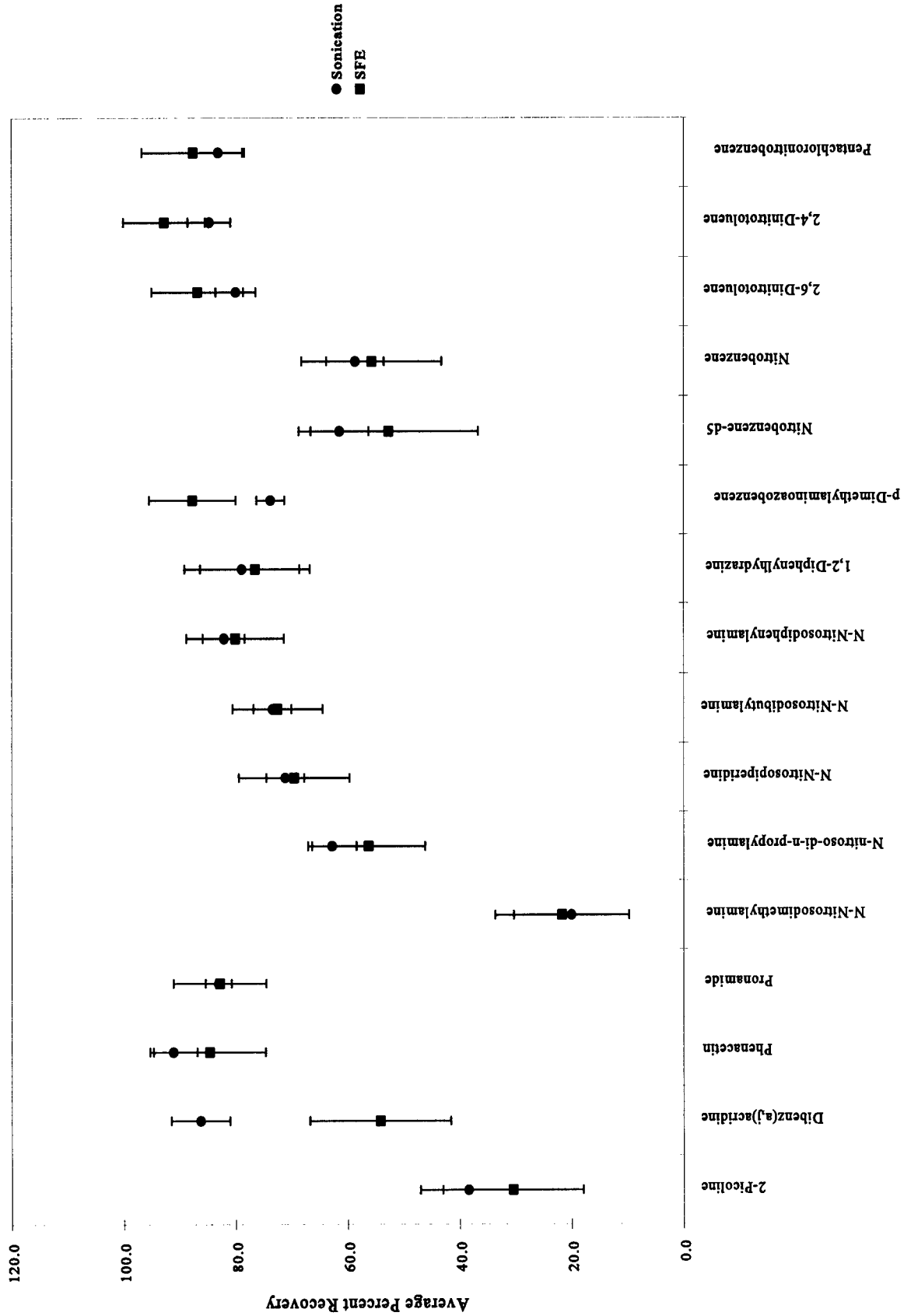

Fig. 10. Comparison between Sonication and SFE for the Extraction of Miscellaneous Nitrogen Compounds from HEPA Filters

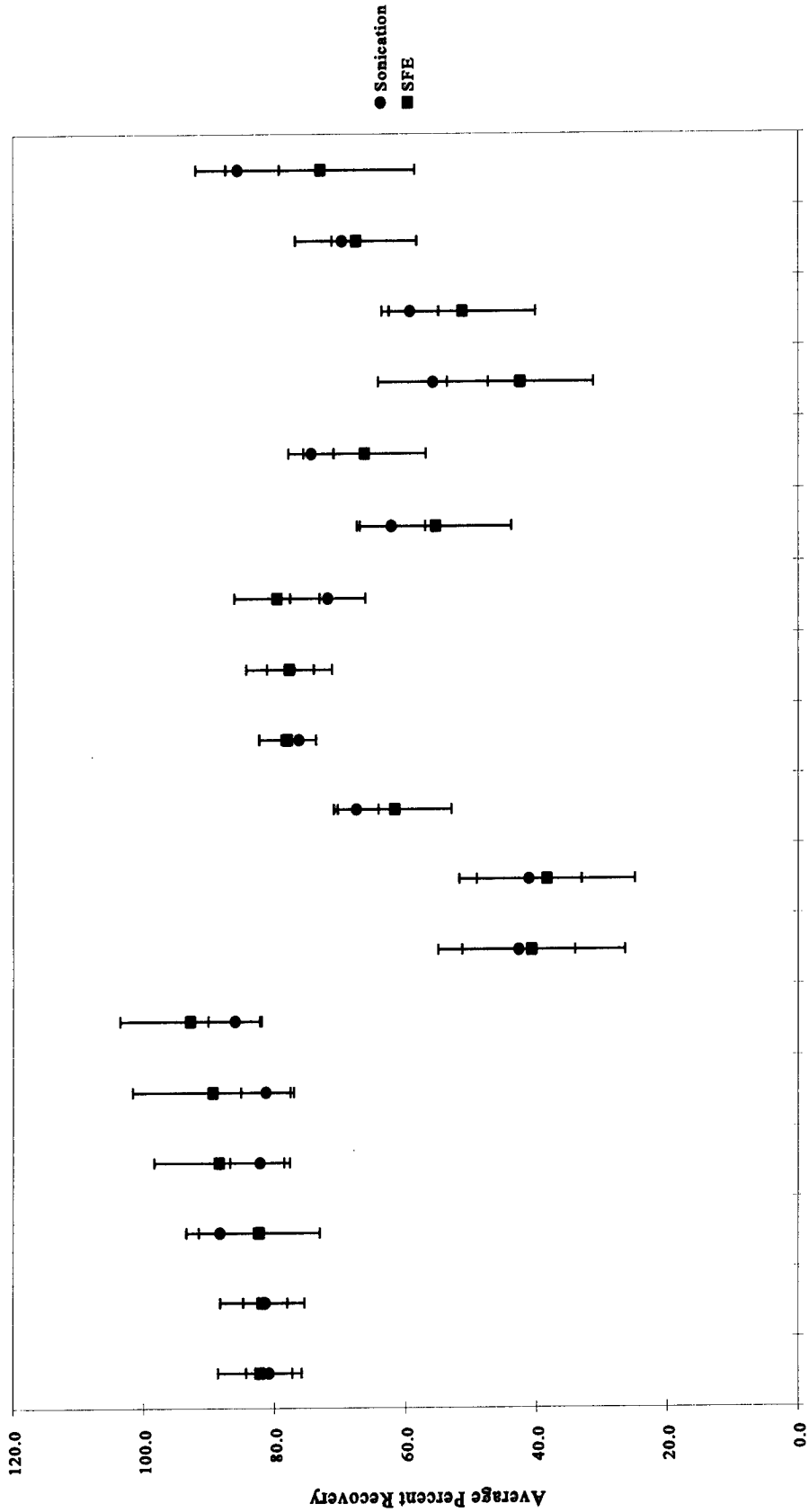

Fig. 11. Comparison between Sonication and SFE for the Extraction of Miscellaneous Compounds from HEPA Filters

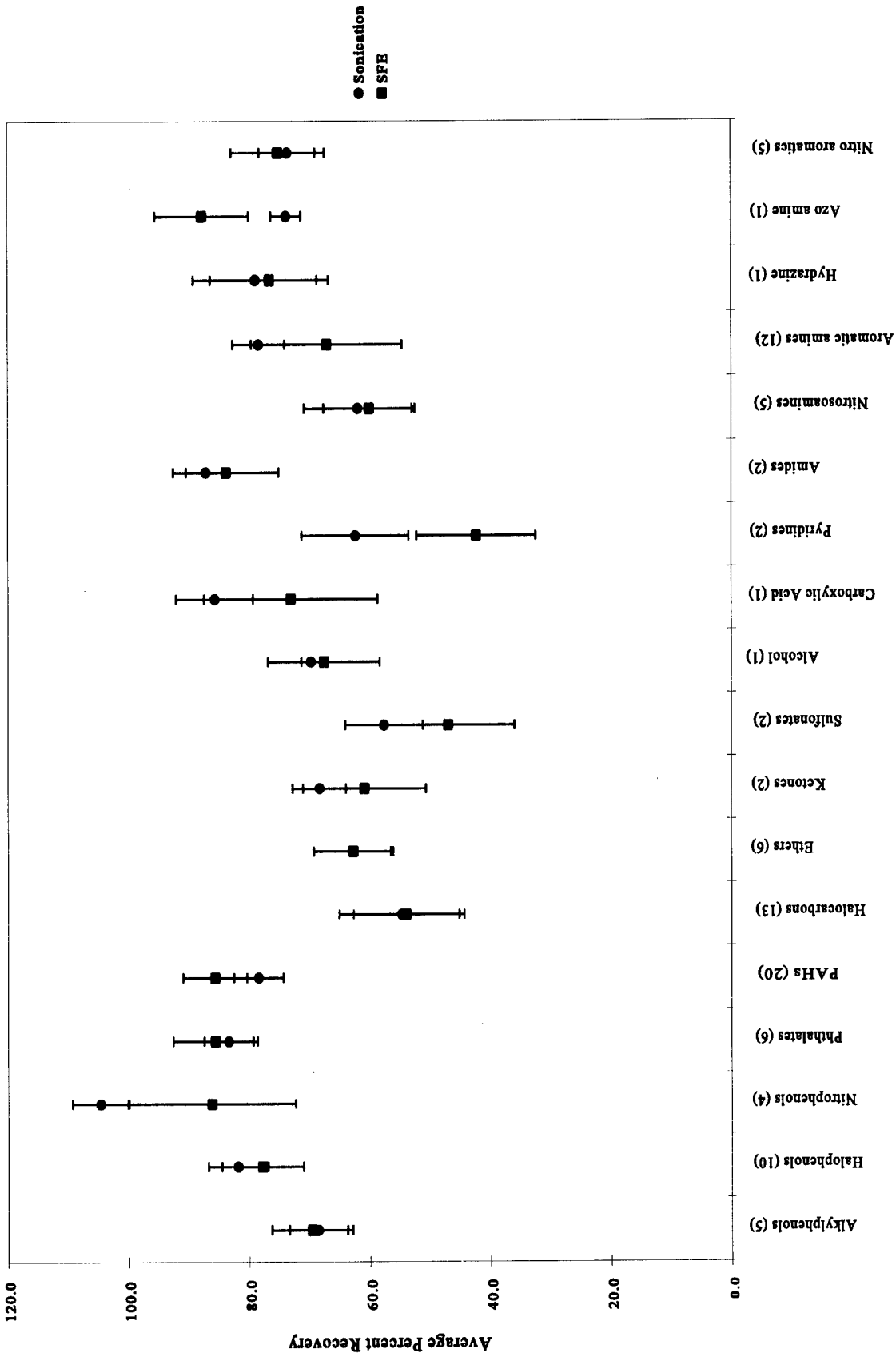

Fig. 12. Comparison between Sonication and SFE for the Extraction of Semivolatile Organic Compounds from HEPA Filters (Average Recoveries for Compound Classes)

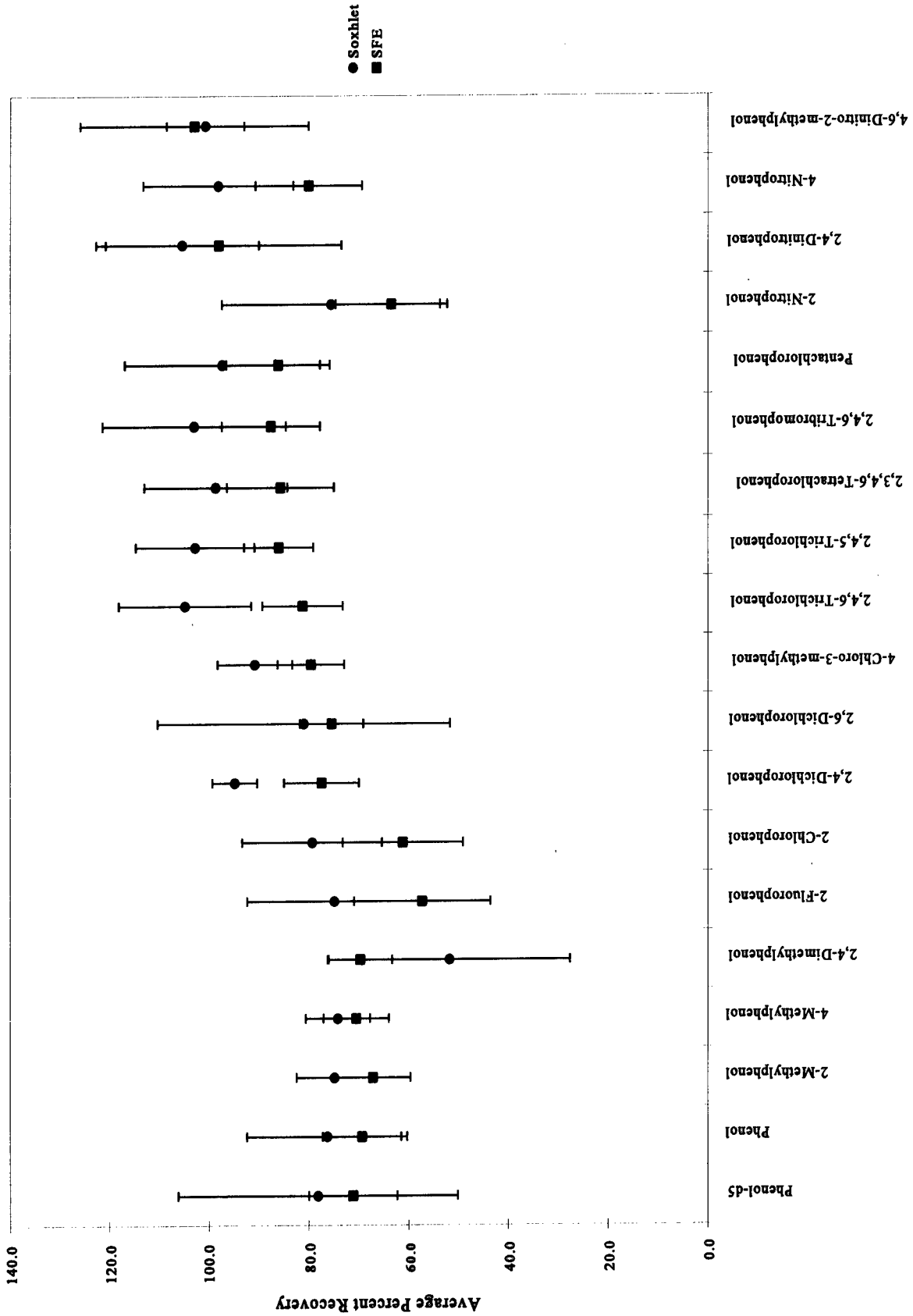

Fig. 13. Comparison between Soxhlet and SFE for the Extraction of Phenols from HEPA Filters

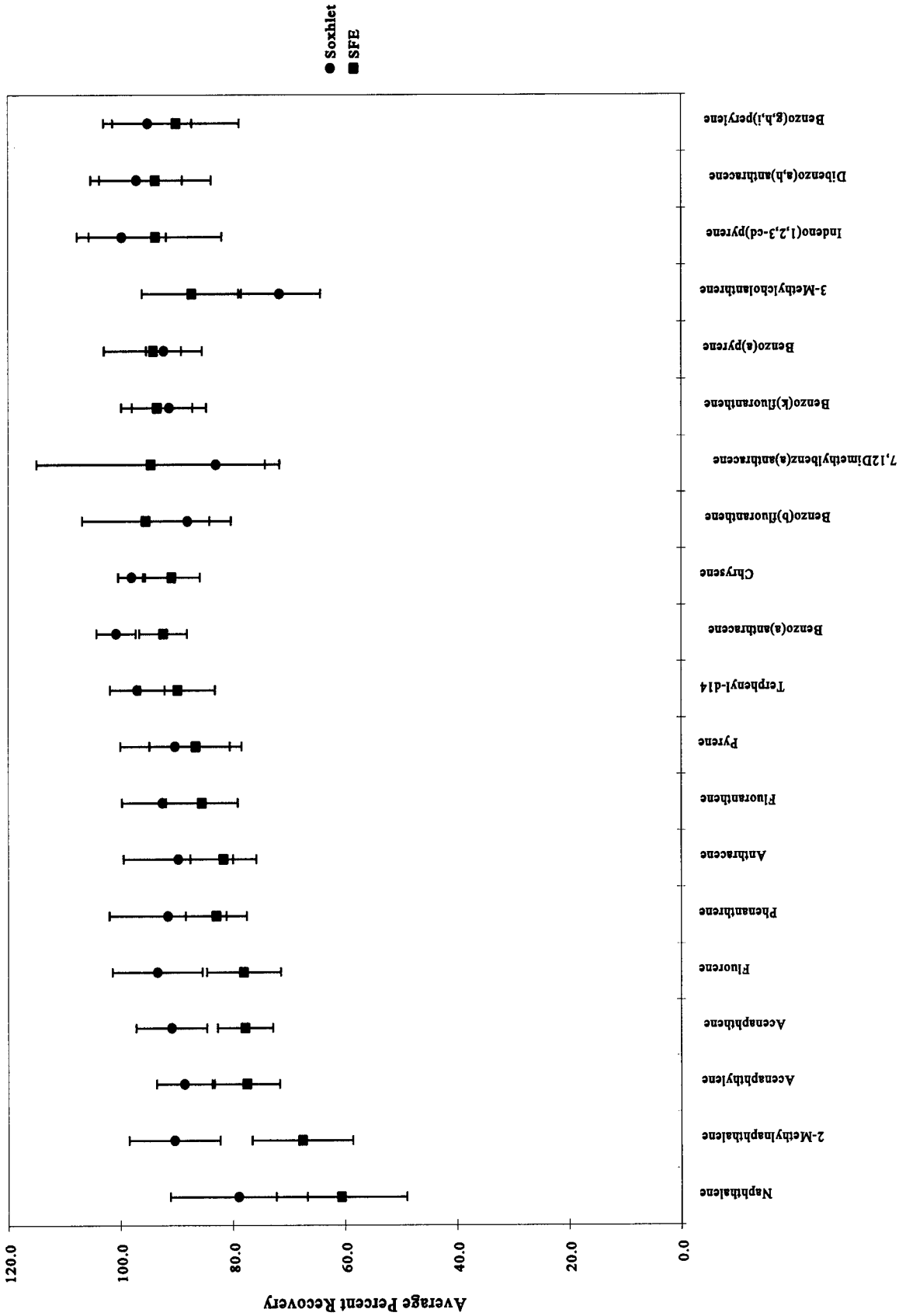

Fig. 14. Comparison between Soxhlet and SFE for the Extraction of PAHs from HEPA Filters

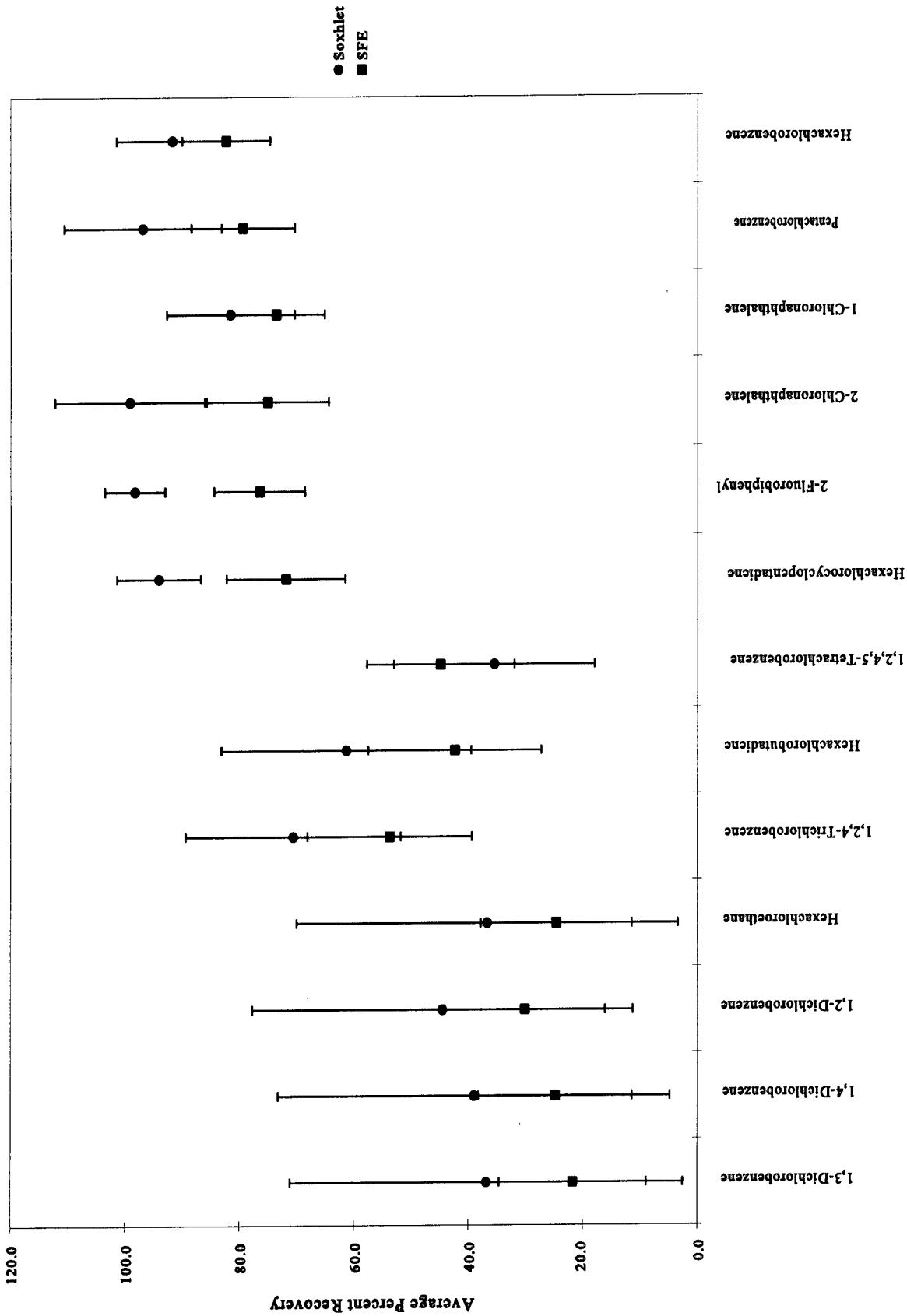

Fig. 15. Comparison between Soxhlet and SFE for the Extraction of Halocarbons from HEPA Filters

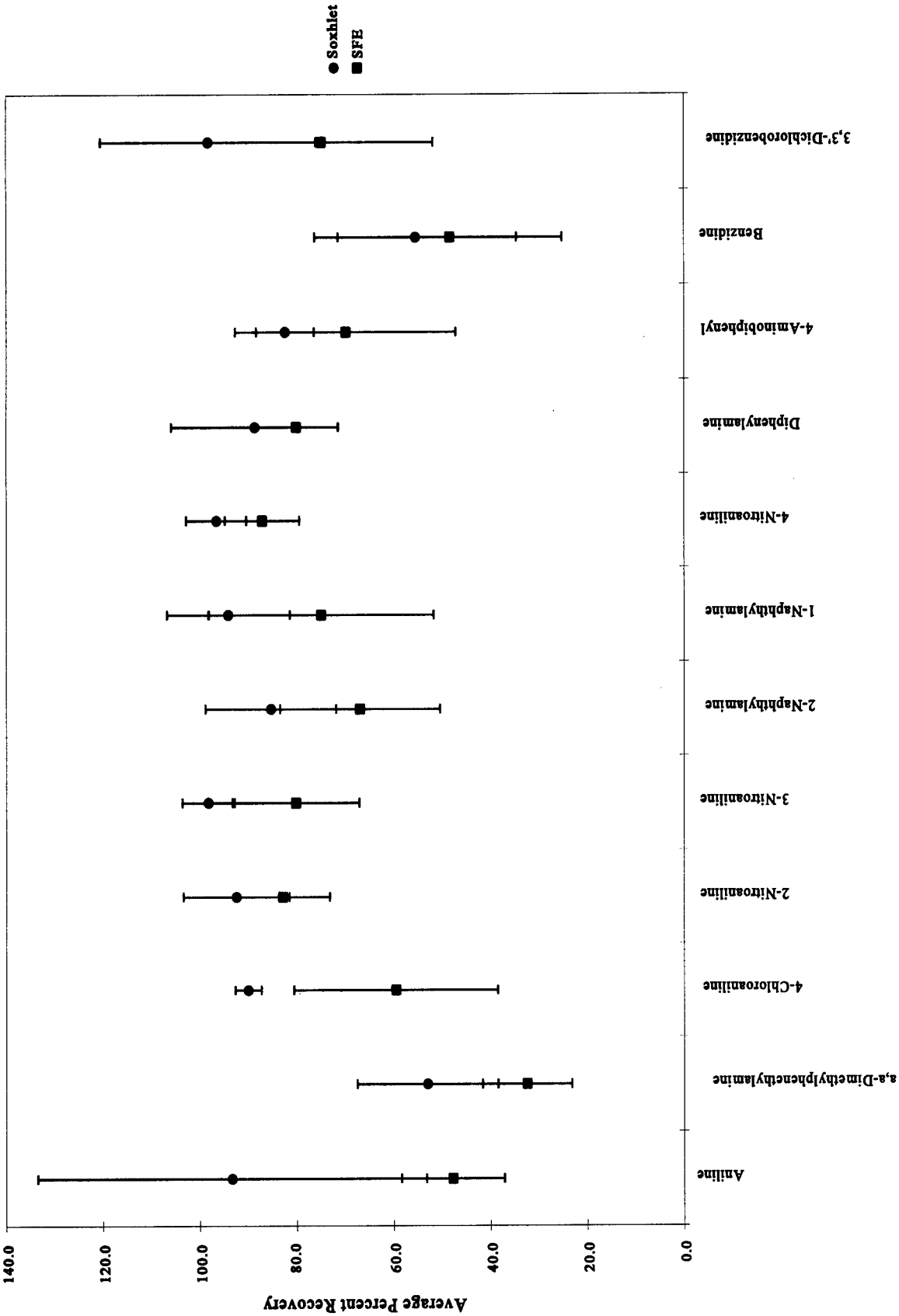

Fig. 16. Comparison between Soxhlet and SFE for the Extraction of Aromatic Amines from HEPA Filters

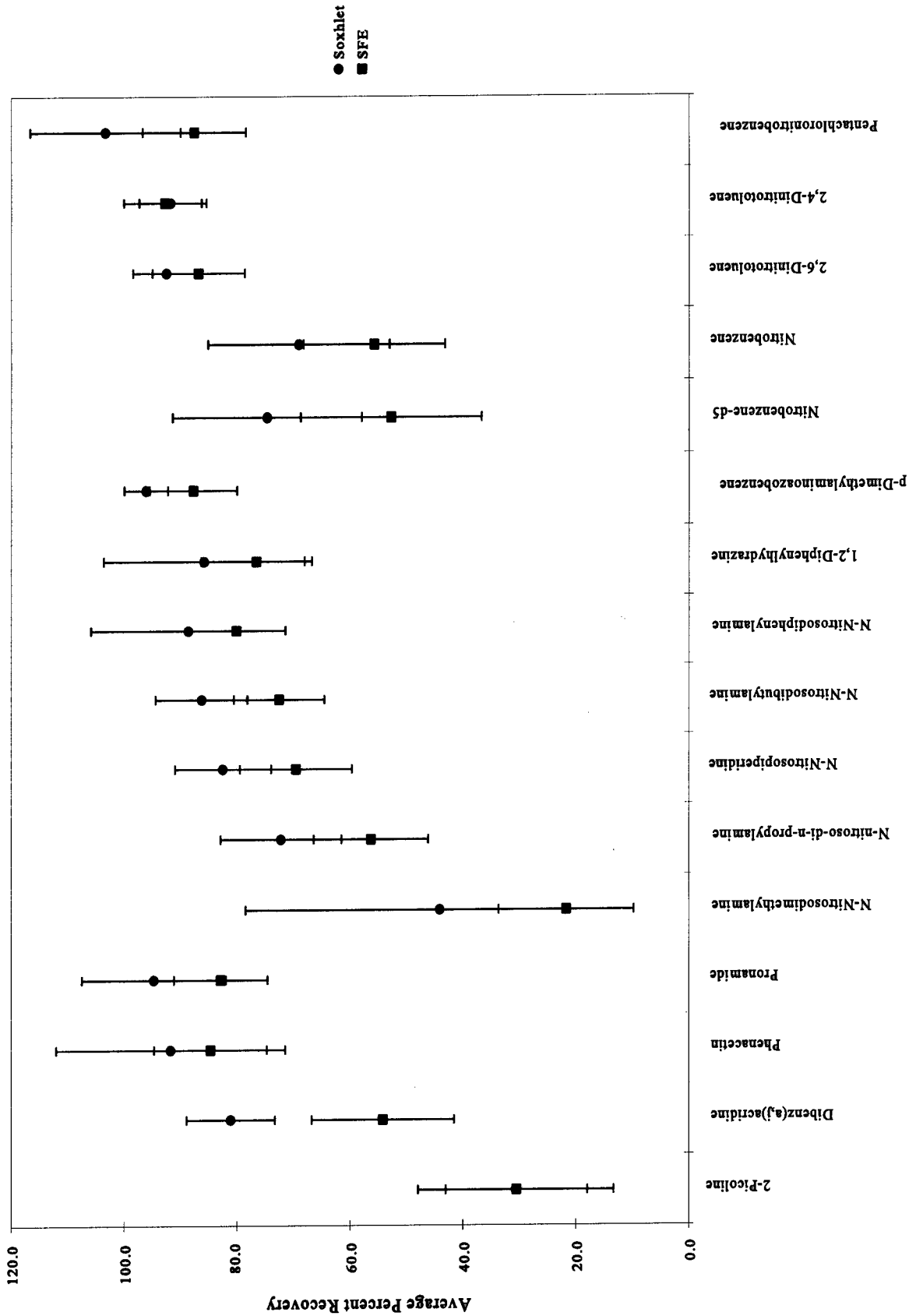

Fig. 17. Comparison between Soxhlet and SFE for the Extraction of Miscellaneous Nitrogen Compounds from HEPA Filters

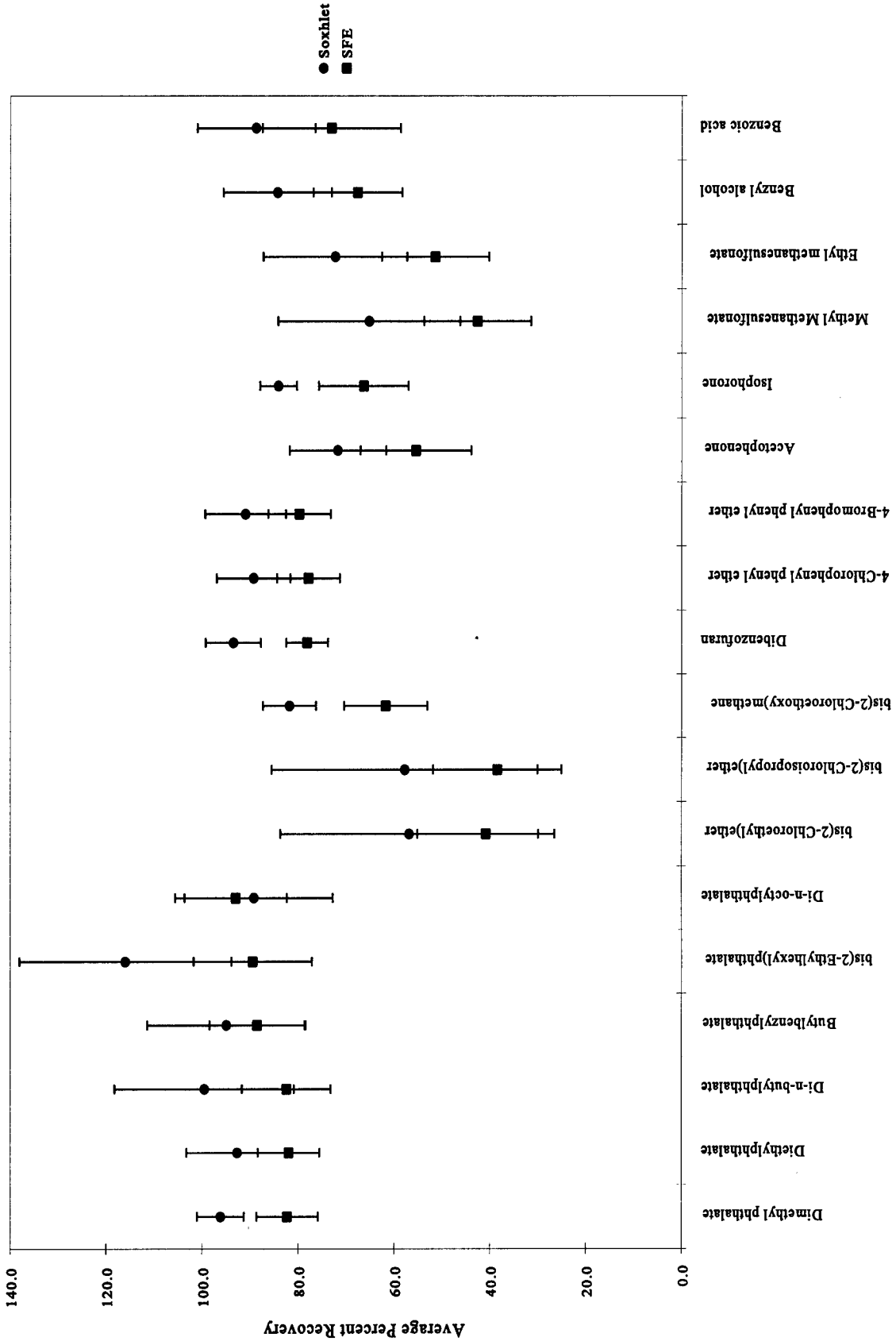

Fig. 18. Comparison between Soxhlet and SFE for the Extraction of Miscellaneous Compounds from HEPA Filters

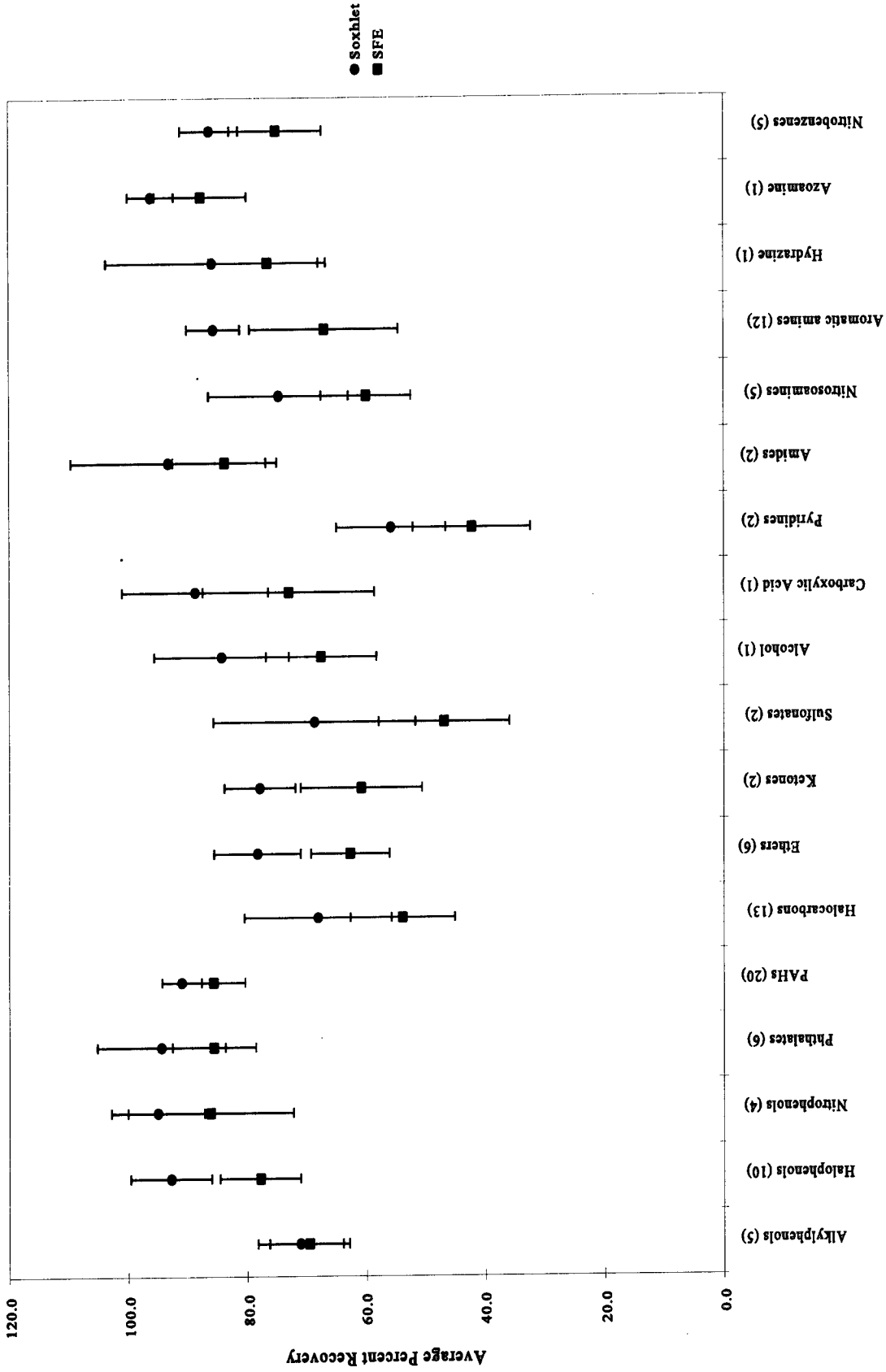

Fig. 19. Comparison between Soxhlet and SFE for the Extraction of Semivolatile Organic Compounds from HEPA Filters (Average Recoveries for Compound Classes)

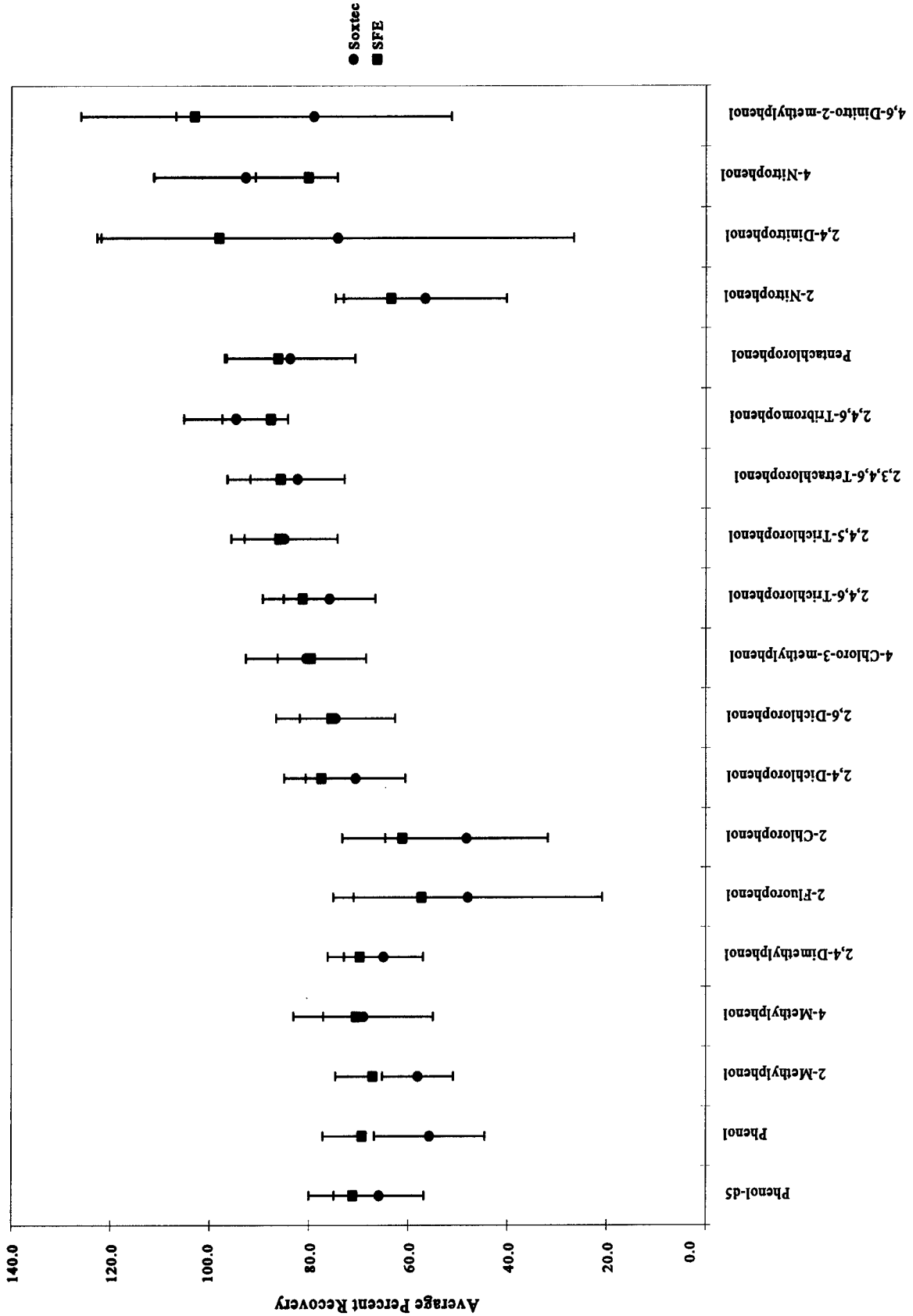

Fig. 20. Comparison between Soxtec and SFE for the Extraction of Phenols from HEPA Filters

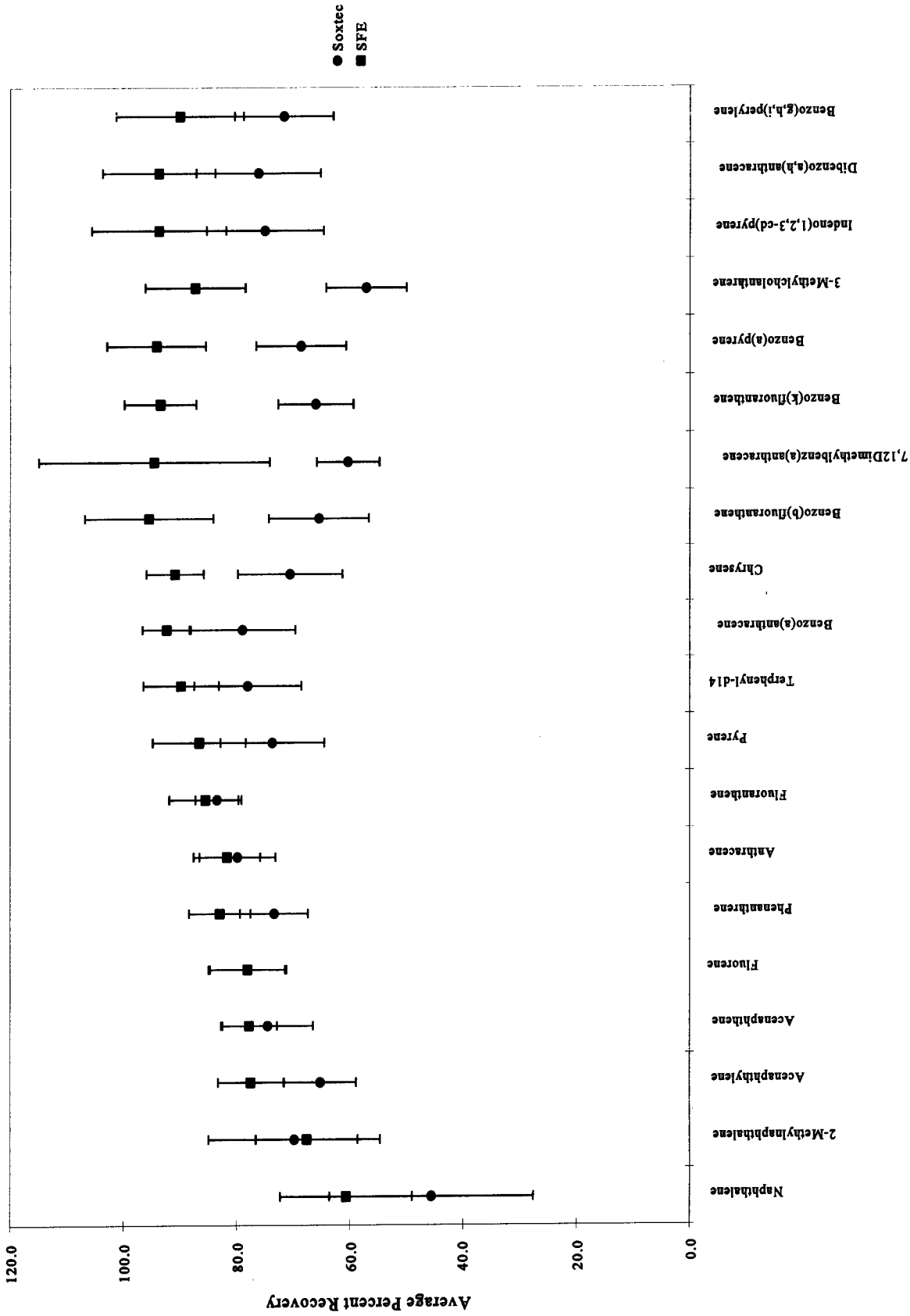

Fig. 21. Comparison between Soxtec and SFE for the Extraction of PAHs from HEPA Filters

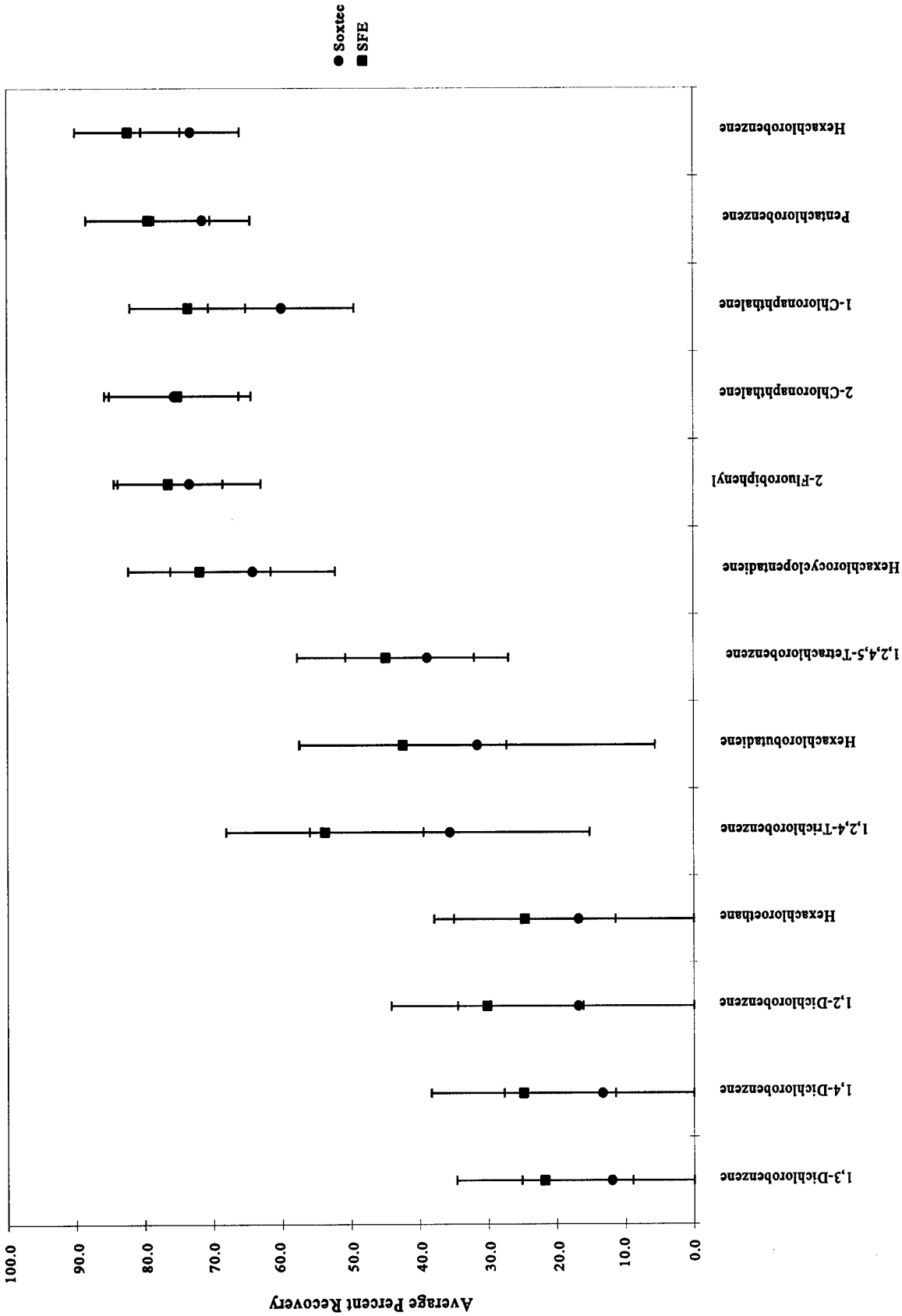

Fig. 22. Comparison between Soxtec and SFE for the Extraction of Halocarbons from HEPA Filters

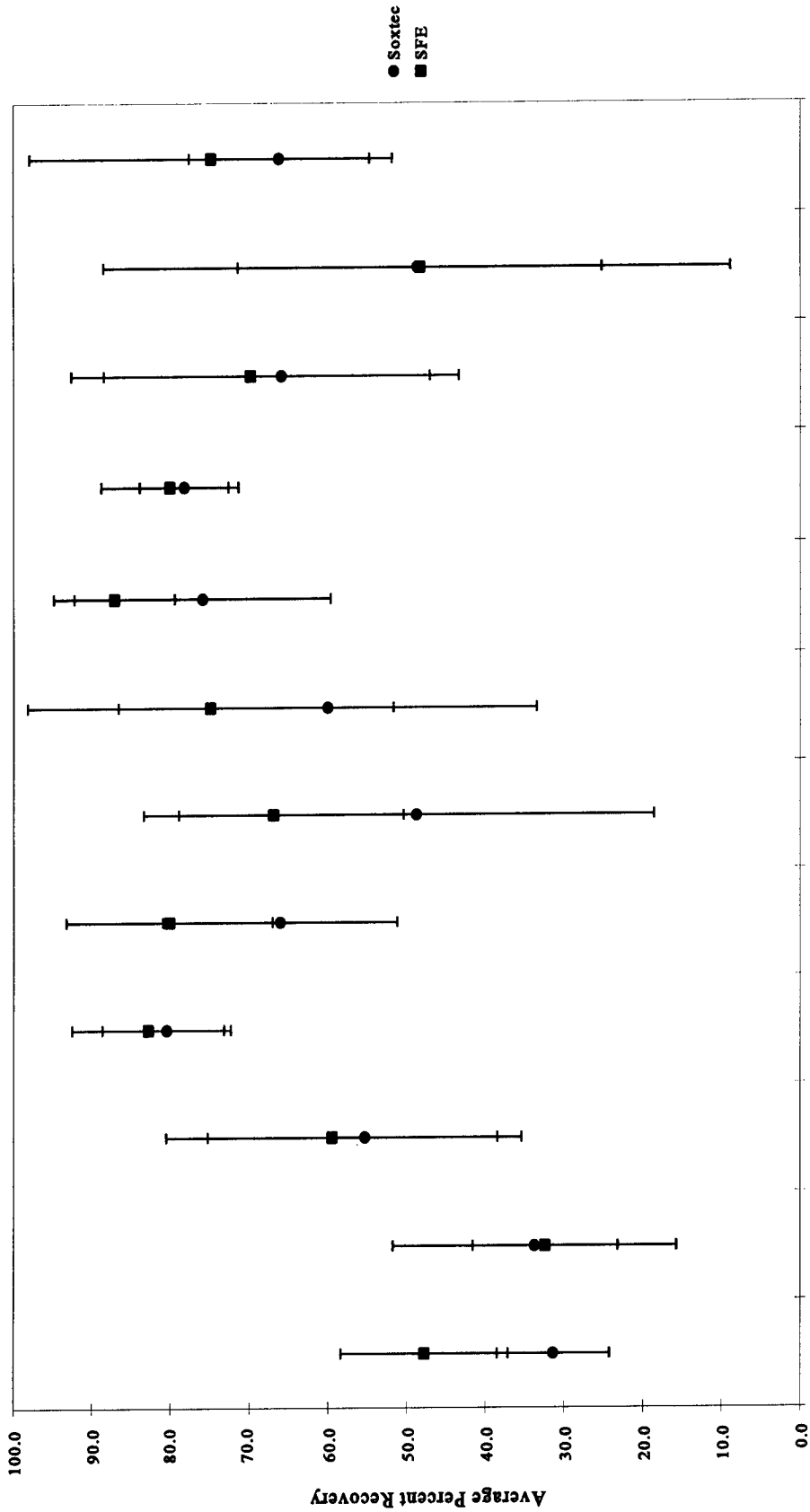

Fig. 23. Comparison between Soxtec and SFE for the Extraction of Aromatic Amines from HEPA Filters

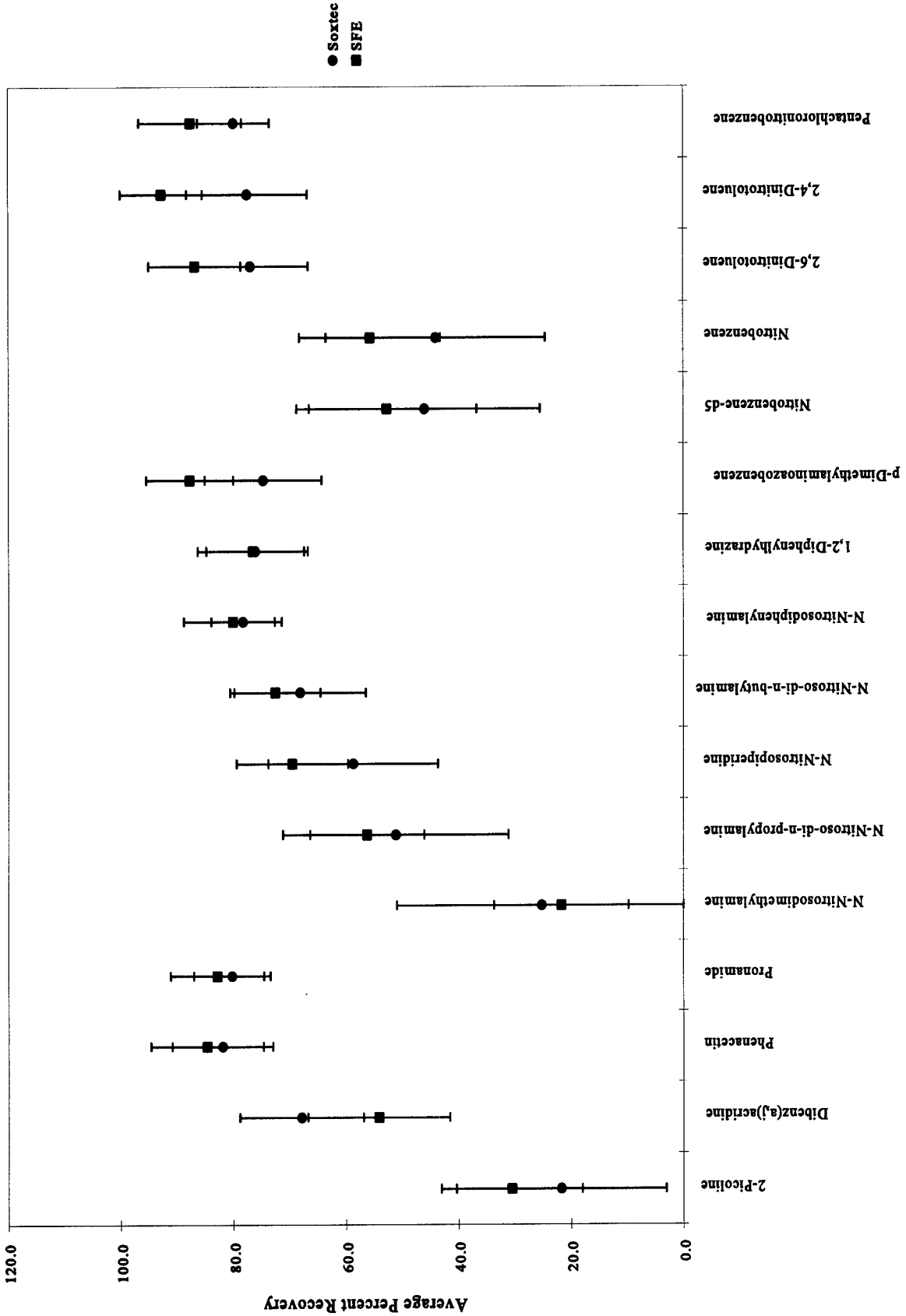

Fig. 24. Comparison between Soxtec and SFE for the Extraction of Miscellaneous Nitrogen Compounds from HEPA Filters

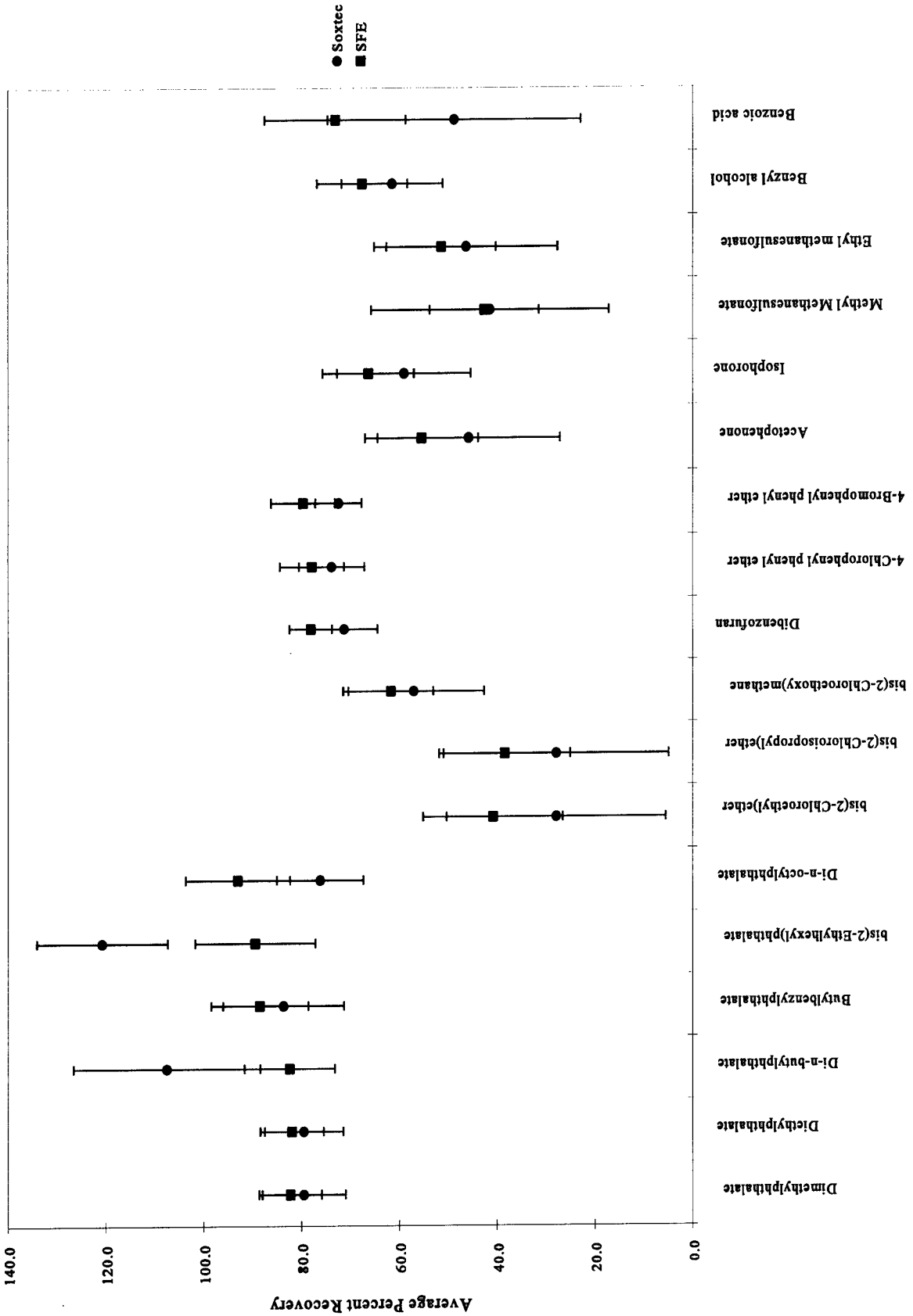

Fig. 25. Comparison between Soxtec and SFE for the Extraction of Miscellaneous Compounds from HEPA Filters

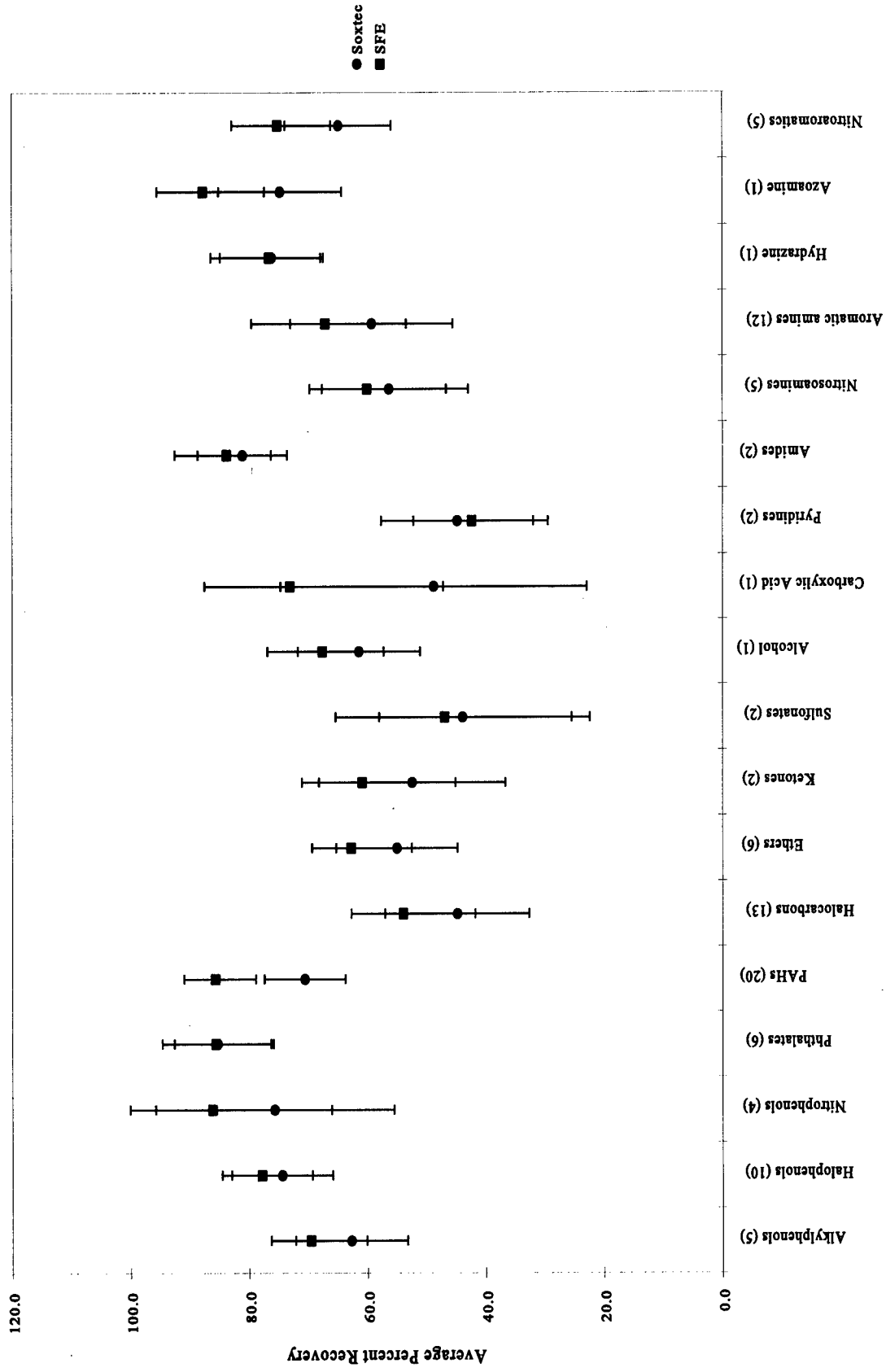

Fig. 26. Comparison between Soxtec and SFE for the Extraction of Semivolatile Organic Compounds from HEPA Filters (Average Recoveries for Compound Classes)

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Report Number (14) ANL/ACL--97/6

Publ. Date (11) 199709

Sponsor Code (18) DOE/HR, XF

UC Category (19) UC-902, DOE/ER

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