

Abstracts of the Chromatography Literature

The literature covering advancements in chromatography is found in many different publications, including numerous journals specializing in chromatography as well as journals that specialize in other fields that use chromatography. Maintaining an awareness of developments in chromatography is a very difficult and time-consuming task. The editors of the *Journal of Chromatographic Science* have implemented a new section, **Abstracts of the Chromatography Literature**, which is intended to simplify this task for the practicing chromatographer by including abstracts of chromatography literature from publications throughout the scientific literature. The articles abstracted in this regular section of our journal are articles published worldwide that the editors of the *Journal of Chromatographic Science* have chosen as significant advances in the field of chromatography. These abstracts are reprinted from *Gas & Liquid Chromatography Literature Abstracts and Index*. As always, your comments and suggestions on this and all aspects of the *Journal* are encouraged.

—Bert Gordon
Co-Editor

Pesticide/Environmental

Single-Step Separation of Organochlorine Pesticide Residues from Fatty Materials by Combined Use of Solid-Matrix Partition and C₁₈ Cartridges

A.D. Muccio, T. Generali, D.A. Barbini, P. Pelosi, A. Ausili, F. Vergori, and S. Girolimetti

ISS-Inst. Superiore di Sanita (Natl. Inst. of Health), Lab. Tossicologia Applicata, Viale Regina Elena 299, 00161 Rome, Italy

J. Chromatogr. **765**(1): 61–68 (1997)

Instr.: HP 5890 II GC
Det.: ECD
Col.: 60 m × 0.25-mm i.d.
Stat. ph.: DB-1701, 0.25 μm; DB-1, 0.25 μm
Carrier gas: Helium, 1.12 mL/min (23.3 cm/s)
Temp.: 90 (2 min) to 170°C at 20°C/min, then to 230°C at 1°C/min, held 15 min, then to 280°C at 2°C/min, and held 10 min
Compds. sepd.: Organochlorine pesticide residues

JH 98-1113G

Simultaneous Determination of Neutral and Acidic Pesticides in Natural Waters at the Low Nanogram per Liter Level

T.D. Bucheli, F.C. Gruebler, S.R. Muller, and R.P. Schwarzenbach

Swiss Federal Inst. for Environ. Sci. and Technol. (EAWAG) and Swiss Federal Inst. of Technol.(ETH), CH-8600 Dübendorf, Switzerland

Anal. Chem. **69**(8): 1569–76 (1997)

Method included enrichment by SPE on graphitized C-black, sequential elution of neutral and acidic pesticides, and derivations of the latter with diazomethane.

Instr.: Fisons HRGC 8000
Det.: FI MD 800 MS

Col.: 32 m × 0.25-mm i.d.
Stat. ph.: OV 240-OH
Carrier gas: Helium
Temp.: Programmed 120 to 260°C
Reten. data: In tables
Compds. sepd.: Triazines, acetamides, and phenoxy acids

PL 98-1174G

Sensitive, Simultaneous Determination of Organomercury, -Lead, and -Tin Compounds with Headspace Solid Phase Microextraction Capillary Gas Chromatography Combined with Inductively Coupled Plasma Mass Spectrometry

L. Moens, T. De Smaele, R. Dams, P. Van Den Broeck, and P. Sandra
Lab. of Anal. Chem., Ghent Univ., Proeftuinstraat 86, B-9000 Gent, Belgium

Anal. Chem. **69**(8): 1604–11 (1997)

Instr.: PE Autosystem
Det.: PE Sciex Elan 5000 ICP
Col.: 30 m × 0.25-mm i.d.
Stat. ph.: FSOT, poly(dimethylsiloxane)
Carrier gas: Xenon/hydrogen (1:99)
Temp.: Programmed 60 to 200°C
Reten. data: In figures
Compds. sepd.: Organo-metallic compounds

PL 98-1180G

Water Pollution Screening by Large-Volume Injection of Aqueous Samples and Application to GC-MS Analysis of a River Elbe Sample

S. Muller, J. Efer, and W. Engewald

Inst. für Anal. Chem., Univ. Leipzig, Linnestr. 3, D-04103 Leipzig, Germany

Fresenius J. Anal. Chem. **357**(5): 558–60 (1997)

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Sample (1 mL) was injected into programmed-temperature vaporizer injector containing Tenax TA.

Instr.: HP 5890
 Det.: MS-EI, 70 eV
 Col.: 25 m × 0.32 mm
 Stat. ph.: CP Sil 19 CB, 0.20 μm
 Carrier gas: Helium, 8 psi
 Temp.: 40 (5 min) to 270°C at 10°C/min and held 5 min
 Compds. sepd.: Hexachlorobenzene, atrazine, and simazine **MP 98-1248C**

Environmental Applications of Gas Chromatography/Atomic Emission Detection

D.F. Gurka, S. Pyle, and R. Titus
 Natl. Exposure Res. Lab., Characterization Res. Div., U.S. Environ. Protection Agcy., P.O. Box 93478, Las Vegas, NV 89193-3478

Anal. Chem. **69(13)**: 2411–17 (1997)

Detection limits, elemental response factors, and regression analysis data were reported for 58 semivolatile environmental contaminants.

Instr.: HP 5890
 Det.: HP 5921A AED
 Col.: 30 m × 0.32-mm i.d.
 Stat. ph.: HP-5
 Carrier gas: Helium
 Temp.: Programmed 45 to 280°C
 Reten. data: In table
 Compds. sepd.: Semivolatile organic compounds **PL 98-1328C**

Separation and Determination of Polycyclic Aromatic Hydrocarbons by Solid Phase Microextraction/Cyclodextrin-Modified Capillary Electrophoresis

A. Nguyen and J.H.T. Luong
 Biotechnol. Res. Inst., Natl. Res. Council Canada, Montreal, PQ, Canada H4P 2R2

Anal. Chem. **69(9)**: 1726–31 (1997)

A glass fiber was prepared and used for absorbing 16 EPA priority PAHs from diluted samples until equilibrium was reached. The fiber was connected to CE capillary, and PAHs were released into CE buffer stream.

Instr.: P/ACE 5000
 Det.: UV, 254 nm
 Col.: 50 cm × 50-μm i.d.
 Mobile ph.: 50mM borate, pH 9.2, containing 35mM sulfobutylxy-β-CD, 10mM Me-β-CD, and 4mM α-CD
 Reten. data: In figures
 Compds. sepd.: PAHs **PL 98-1939L**

Analysis of Carbamate Pesticides by Micellar Electrokinetic Chromatography

M. Rossi and D. Rotilio
 Ist. di Ricerche Farmacologiche Mario Negri, "Gennaro Paone" Environ. Health Ctr., Consorzio Mario Negri Sud, Via Nazionale, 66030 Santa Maria Imbaro, Italy

J. High Res. Chromatogr. **20(4)**: 265–69 (1997)

Capillary electrophoresis was used for the qualitative and quantitative analysis of 11 compounds of the carbamate, thiocarbamate, and dithiocarbamate classes of pesticides. Micellar electrokinetic chromatography was employed for the separation of these substances. The analysis was performed using an uncoated fused-silica column, in borate buffer containing SDS and 10% methanol at basic pH, and UV detection at 230 nm. The addition of methanol to the buffer increased the separation and affected intrinsic migration of analytes. The working standard solutions were prepared in acetonitrile–water (50:50). Although these compounds are structurally different, with a large spectrum of chemical properties such as polarity and solubility, good separation and sensitivity were obtained. Moreover, good recovery of carbamates was obtained from tap water using a styrene–divinylbenzene polymer column with the solid-phase extraction technique. **MT 98-2006L**

Single-Column Method of Chelation Ion Chromatography for the Analysis of Trace Metals in Complex Samples

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J. Chromatogr. **770(1–2)**: 129–35 (1997)

A single-column chelation ion chromatographic system for preconcentration and separation of trace transition metals was described. The system included standard chromatographic equipment with a post-column reagent system based on the reaction with 4-(2-pyridylazo)resorcinol followed by photometric detection at 495 nm. Iminodiacetic acid bonded to 5 μm silica (Diasorb IDA) was used as a chelating stationary phase. **JH 98-2081L**

Calculation of Detection Limits for a Single-Laboratory Ion-Chromatographic Method to Determine Parts-Per-Trillion Ions in Ultrapure Water

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¹Texas Instruments, Box 650311, M/S 301, Dallas TX 75265 and ²Alcoa Technical Ctr., AMCT-D-10, 100 Technical Dr., Alcoa Center, PA 15069

J. Chromatogr. **770(1–2)**: 105–14 (1997)

This paper addressed the calculation of detection limits (DLs) for ion-chromatographic data at low parts-per-trillion (w/w) levels. The main objectives were as follows: to explain two statistical techniques (the EPA or '3 σ' approach and the Hubaux-Vos method); to calculate DLs using each procedure; to discuss the strengths and weaknesses of each statistical approach; and to decide if the analytical method was appropriate for quantitating anions at the 50-ppt level in deionized water. **JH 98-2098**

Chiral/Biomedical

Enantioselective Gas Chromatographic Assay of 2-Alkylamines Using N-(Trifluoroacetyl) Propyl Derivatives and a Chiral Capillary Column

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J. Chromatogr. Biomed. Appl. **689(1)**: 165–73 (1997)

Instr.: HP 5710 A GC
 Det.: FID
 Col.: 25 m × 0.25-mm i.d.
 Stat. ph.: Chirasil-Val, 0.16 μm
 Carrier gas: Helium, 100 kPa
 Temp.: 80 (4 min) then to 220°C at 8 C/min and held 8 min
 Compds. sepd.: 2-Alkylamines as derivatives **JH 98-1128G**

Hair Analysis, a Novel Tool in Forensic and Biomedical Sciences: New Chromatographic and Electrophoretic/Electrokinetic Analytical Strategies

F. Tagliaro¹, F.P. Smith², Z. De Battisti¹, G. Manetto¹, and M. Marigo¹
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J. Chromatogr. Biomed. Appl. **689(1)**: 261–72 (1997)

The present paper presented an updated review (with 102 references) of the modern techniques for hair analysis, including screening methods (e.g., immunoassays) and more sophisticated methodologies adopted for result confirmation and/or for research purposes, with special emphasis on gas chromatography–mass spectroscopy, liquid chromatography, and capillary electrophoresis. **SM 98-1142G**

Miniaturized Supported Liquid Membrane Device for Selective On-Line Enrichment of Basic Drugs in Plasma Combined with Capillary Zone Electrophoresis

S. Palmarsdottir, E. Thordarson, L. Edholm, J.A. Jonsson, and L. Mathiasson
 Anal. Chem., Univ. of Lund, P.O. Box 124, S-221 00 Lund, Sweden

Anal. Chem. **69(9)**: 1732–37 (1997)

A hollow fiber miniaturized supported liquid membrane was connected on-line with capillary CZE and used for determination of bambuterol in human plasma. The analyte was extracted from the outside of the hollow fiber through the liquid membrane into the acceptor solution in the fiber lumen. The process was driven by the difference in pH between the donor and acceptor solution. The whole volume of the acceptor solution was then injected into the CZE capillary by using the double-stacking technique for large volume injection. Good performance was demonstrated. **PL 98-1947L**

Evaluation of an On-Line Solid-Phase Extraction Method for Determination of Almokalant, an Antiarrhythmic Drug, by Liquid Chromatography

H. Svennberg and P.O. Lagerstrom
 Astra Hassle AB, Bioanal. Chem., S-431 83 Molndal, Sweden

J. Chromatogr. Biomed. Appl. **689(2)**: 371–77 (1997)

Instr.: LKB 2150 pumps, two; Valved System
 Det.: Shimadzu RF-535 fluorescence: excitation, 248 nm; emission, 305 nm
 Col.: 100 × 4.6-mm i.d.; 10 × 4.3-mm i.d., guard
 Stat. ph.: Microspher C₁₈, 3 μm; Machery Nagel C₁₈, 5 μm
 Mobile ph.: 2mM DMAO in phosphate buffer, pH 3.1; ACN–2-CH₃H₇OH–2mM DMOA buffer (5:1.7:93.3, v/v/v); flow rate: 1.0 mL/min
 Compds. sepd.: Almokalant **JH 98-1981L**

Analysis of Corticosteroids in Biofluids by Capillary Electrochromatography with Gradient Elution

M.R. Taylor, P. Teale, S.A. Westwood, and D. Perrett
 Horseracing Forensic Lab., P.O. Box 150, Newmarket Rd., Fordham, Ely, Cambridgeshire CB7 5WP, U.K.

Anal. Chem. **69(13)**: 2554–58 (1997)

Urine samples were purified using SPE, and plasma by automated dialysis.

Instr.: Modular
 Det.: UV, 240 nm
 Col.: 24 cm × 50-μm i.d.
 Stat. ph.: Apex ODS
 Mobile ph.: Gradient acetonitrile–5mM aqueous ammonium acetate
 Temp.: 20°C
 Reten. data: In figures
 Compds. sepd.: Corticosteroids **PL98-2176L**

Instrumentation

Electrolyzer-Powered Flame Ionization Detector

A. Amirav and N. Tzanani

School of Chem., Sackler Fac. of Exact Scis., Tel Aviv Univ., Tel Aviv 6997, Israel

Anal. Chem. **69(6)**: 1248–55 (1997)

Based on combustion of a premixed H₂ and O₂ provided by a water electrolyzer with low power and water consumption and without separation, compression, or pressure stabilization of the gases. The flame tip of the detector had a narrow hole (250 μm) to prevent flame flash backs, and the entire structure was maintained above 100°C to prevent water condensation. Sensitivity was similar to FID and response was linear over 6 orders of magnitude. Use of splitless injector with a megabore column quenched the flame, but this could be countered by use of a miniature pump during solvent elution time. **PL 98-1129G**

Determination of Polar Analytes in Aqueous Matrices by Purge and Trap

A. Kaufmann

Schuler Weine, Wein Analytik, CH-6423 Seewen, Switzerland

J. High Res. Chromatogr. **20(1)**: 10–16 (1997)

A fully automated purge-and-trap system was developed for the determination of volatile wine components. The proposed system consists of a sampler, a purge-and-trap instrument, and a GC. All steps, from the uptake of the infiltrated wine from the sampler rack to the printout of the integrated chromatograms, proceeded unattended. Typical problems occurring when analyzing polar analytes by the classical purge-and-trap approach were specifically addressed. The system was designed to handle such difficult polar analytes. Efforts were made to improve the reproducibility and the carry-over of these analytes. The measures adopted include thorough rinsing and subsequent conditioning of the purge-and-trap sparger; elimination of matrix effects by dilution and high salt loads; tight specification of purge-relevant parameters; and split-interface to improve GC retention time reproducibility. **MT 98-1143G**

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Optimization of the Dwell-Time for the Quantitative Analysis by Gas Chromatography–Mass Spectrometry

L.J. Eveleigh and C.J. Ducauze

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J. Chromatogr. **765**(2): 241–45 (1997)

Instr.: HP 5890 II GC
 Det.: HP 5971 MSD
 Col.: 25 m × 0.1-mm i.d.
 Stat. ph.: Methyl silicone gum, 0.125 μm
 Carrier gas: Hydrogen, 50 cm/s
 Compsd. sepd.: Alkanes

JH 98-1161G

Modified Capillary GC–MS System Enabling Dynamic Headspace Sampling with On-Line Cryofocusing and Cold On-Column Injection of Liquid Samples

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Istituto Lattiero Caesario e di Biotecnologie Agroalimentari, via S. Gaetano 74, 36016 Thiene (VI), Italy

J. High Res. Chromatogr. **20**(1): 24–28 (1997)

The paper described a capillary GC–MS hardware arrangement allowing either on-line injection of sample of gases and vapors coming from dynamic headspace or purge-and-trap samplers and their subsequent cryofocusing/thermal desorption prior to the analytical capillary column (first column) or direct injection of liquid samples through a cold on-column injector into a second analytical capillary column (mounted in series with the first). Chromatographic profiles of acacia honey aromas provided confirmation of the suitability of this system.

MT 98-1165G

On-Line Derivatization in Combined High-Performance Liquid Chromatography–Gas Chromatography–Mass Spectrometry

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Analyst **122**(9): 955–61 (1997)

Procedures for HPLC–GC and HPLC–GC–MS of involatile analytes using on-line derivatization between the LC and GC separation stages were reported. Derivatization involved either pre-mixing of reagent and analyte in the HPLC eluent ahead of the GC precolumn or independent delivery of reagent after deposition of the desolvated analytes in the GC precolumn. Derivatization of the stilbene hormones, quinoxaline-2-carboxylic acid, sulfamethazine, and 2-naphthoic acid with silylation, methylation, and acetylation reagents was investigated. Derivation efficiencies of 84–100% were achieved.

PL 98-1970L

Evaluation of Temperature Effects on Selectivity in RPLC Separations Using Polybutadiene-Coated Zirconia

J. Li and P.W. Carr

Dept. of Chem., Kollhoff and Smith Halls, Univ. of Minnesota, 207 Pleasant St., SE, Minneapolis, MN 55455

Anal. Chem. **69**(11): 2202–2206 (1997)

The influence of temperature was found to depend strongly on the solute type. For solutes of similar structure such as PAHs, temperature had almost no effect on selectivity. However, for solutes with very different functional groups such as chlorophenols,

temperature changes did significantly affect selectivity. In complex mixtures with polar and ionizable solutes, optimization by varying

Petrochemical

temperatures should be beneficial.

PL 98-2091L

A Gas Chromatographic Separation for the H and C Stable Isotope Ratio Determination of Coal Compounds

D. Antenucci, J. Bouquegneau, A. Brasseur, P. Dauby, R. Letolle, C. Jacquemin, and J. Pirard

Lab. de Genie Chimique, Universite de Liege, B6a, B-4000 Liege, Belgium

Anal. Chem. **69**(11): 2030–34 (1997)

A completely automated GC technique was described to separate gaseous compounds produced during underground coal gasification, and to determine their stable isotope ratios. H₂, CO, CH₄, and other hydrocarbons are oxidated to CO₂ and/or H₂O and H₂O and H₂S were reduced to H₂.

Instr.: GC 8000 Special
 Det.: TCD with MS on separated oxidation and reduction products.
 Col.: Two 2 m and one 1 m, in series
 Stat. ph.: Porapak QA and molecular sieve 5A
 Carrier gas: Helium
 Temp.: 60°C
 Reten. data: In figures
 Compsd. sepd.: H, CO, CO₂, H₂O, H₂S, CH₄, and heavier hydrocarbons

PL 98-1258G

The Characterization and Quantitation of Sulfur-Containing Compounds in (Heavy) Middle Distillates by LC–GC–FID–SCD

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Univ. of Amsterdam, Amsterdam Inst. of Molecular Studies, Lab. for Anal. Chem., Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands

J. High Res. Chromatogr. **20**(3): 131–37 (1997)

A modified sulfur chemiluminescence detector (SCD) was interfaced to an HPLC–HRGC hyphenated system. This combination enabled the full characterization and quantitation of the sulfur containing compounds in (heavy) middle distillate oil fractions (boiling range 150–450°C). The system was suited to identify and determine the various groups of organo-sulfur structures such as thiols + sulfides + thiophenes, benzothiophenes, and benzo-naphthothiophenes. Within these groups, a separation according to boiling point was accomplished. Therefore, it allowed the separation, identification, and quantitation of a number of individual species, especially those that are refractory to hydrodesulfurization (HDS), such as 3-methyl-benzothiophene, 4-methyl-dibenzothiophene, and 4,6-dimethyl-dibenzothiophene. The analysis of these groups and specific compounds was the key in understanding the kinetics of the chemistry involved in HDS. The complete instrumental set-up was fully automated by computer control. To suppress possible interferences and quenching of the sulfur response of the SCD from (large amounts of) hydrocarbons, it was aligned above the adapted flame ionization detector (FID) of the GC. This rendered a sensitivity of the SCD for sulfur of 2 pg per second, which corresponded to a minimum detectable level for individual sulfur species in oil fractions for the complete system of 1 ppm (mg per

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Foods/Flavors

kg) sulfur. Its linear dynamic range exceeds 10^5 , which means that untreated, high sulfur containing feedstocks can also be analyzed directly. The selectivity of sulfur to carbon of the modified SCD exceeds 10^6 . A number of HDS feedstocks and desulfurized products of different desulfurization levels were analyzed with the system. From the analysis results, the behavior of the refractory compounds in HDS can now be followed closely. **MT 98-1380G**

Use of Capillary Gas Chromatography for Determining the Hydrogenation Level of Edible Oils

M. Naglic and A. Smidovnik

Natl. Inst. of Chem., Hajdrihova 19, 1001 Ljubljana, Slovenia

J. Chromatogr. **767**(1-2): 335-39 (1997)

Instr.: Varian 3400 GC
 Det.: FID
 Col.: 30 m \times 0.32-mm i.d.; 100 m \times 0.25-mm i.d.
 Stat. ph.: SP-2380, 0.2 μ m; SP-2560, 0.2 μ m
 Carrier gas: Helium, 1.2 mL/min
 Temp.: 150 (3 min) to 300°C at 3°C/min
 Compds. sepd.: Methyl esters of fatty acids **JH 98-1271G**

On-Line LC-GC-AED and LC-GC-MS Multidimensional Methods for the Analysis of PAC in Fuels, Combustion Emissions and Atmospheric Samples

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School of Chem., Univ. of Leeds, Leeds LS2 9JT, U.K.

J. High Res. Chromatogr. **20**(2): 109-14 (1997)

The comparison and concentration of polycyclic aromatic compounds (PAC) in fuels, in their combustion products, and in the atmosphere remains a topic of considerable interest. Despite the wealth of literature on the identification of PAC, speciation at low concentrations remains difficult because of the limitations of the instruments and the complexity of fuel and environmental samples. Consequentially, on-line sample preparation procedures (SPE, SFE, LC, etc.) were becoming an increasingly important step in the analysis procedure particularly where sample clean-up and fractionation were essential for improving analytical resolution. In this study, a normal-phase high-pressure liquid chromatography-gas chromatography (LC-GC) system was developed to provide quantitative analysis of samples, as diverse as coal liquids, petroleum fuels, diesel exhaust particulates, and urban air particulates. Separation and identification of parent and alkylated PAH, heterocyclic, nitro-, and oxy-PAC were achieved by direct coupling to an atomic emission detector and a bench-top mass spectrometer. For both systems, the primary LC separation combined with the large sample volume transferred to GC vastly improved detection limits. Furthermore, the complementary nature of the two detectors used enabled the positive identification of many unknowns. **MT 98-1330G**

Assay for the (R)- and (S)-Enantiomers of Salsolinols in Biological Samples and Foods with Ion-Pair High-Performance Liquid Chromatography Using β -Cyclodextrin as a Chiral Mobile Phase Additive

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J. Chromatogr. Biomed. Appl. **689**(2): 313-20 (1997)

Instr.: Shimadzu LC-9A pump
 Det.: ESA Coulochem II
 Col.: 250 \times 4.6-mm i.d.
 Stat. ph.: Inertasil ODS-3
 Mobile ph.: 25mM sodium phosphate buffer, pH 3.0, containing 12mM β -cyclodextrin-1mM SHS-3% ACN; flow rate: 0.5 mL/min
 Compds. sepd.: (R)- and (S)-Enantiomers of salsolinol

JH 98-1938L

Simultaneous Determination of Nicotinamide, Nicotinic Acid, Riboflavin, Thiamin, and Pyridoxine in Enriched Brazilian Foods by HPLC

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J. High Res. Chromatogr. **20**(4): 245-48 (1997)

The control of the enrichment levels in food is difficult, mainly because of the lack of appropriate analytical methodologies. The amounts of the five B-group vitamins (nicotinamide, nicotinic acid, riboflavin, thiamin, and pyridoxine) were determined in enriched Brazilian foods by a high-performance liquid chromatography method. Fifty products, such as biscuits, liquid and dry milks, flavored milk drinks, flour, macaroni, and cereals were analyzed. Some products showed the amounts declared on the package. Although some slight quantitative variations were shown in the biscuits, one showed levels of riboflavin 35% lower than the value declared. Of five different corn cereal brands, only one showed the declared vitamin content, and the others showed levels 30% lower than declared. No B-group vitamins were detected in one brand of enriched macaroni, except for the nicotinic acid naturally present in the flour. On the other hand, one flavored milk drink exhibited vitamins levels 200% higher than the amounts declared and one milk drink mix presented thiamin, riboflavin, and nicotinamide levels three to five times greater than stated. These results suggested an absence of control of the amount of vitamins in enriched foods. **MT 98-1979L**

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