56º CONGRESSO BRASILEIRO DE QUÍMICA
07 a 11/novembro/2016
Belém / Para
Química: Tecnologia, Desafios e Perspectivas na Amazônia

LABBES
*LABORATÓRIO DE ANALÍTICA*
*•BIOANALÍTICA*
*•BIOSSENSORES*
*•ELETROANALÍTICA &*
*•SENSORES*

# PROCEDIMENTOS ANALÍTICOS AMBIENTALMENTE SUSTENTÁVEIS

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# Agradecimentos

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**Profa. Dra. Kellen Heloizy Freitas**

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FAPESP

CNPq

CAPES

# A química “verde”

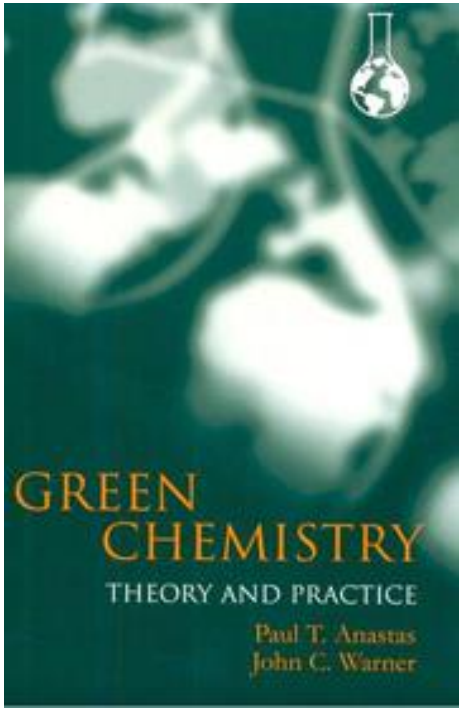
**Os 12 Princípios da Química Verde, foram originalmente publicados por Paul Anastas e John Warner no livro:**

**Green Chemistry: Theory and Practice (Oxford University Press: New York, 1998)**

**Estes princípios fornecem uma orientação para os químicos implementarem a química verde.**

**Paul T. Anastas**

**John C. Warner**



# Os 12 Princípios da Química Verde

1)**Prevenção:** É melhor prevenir a formação de subprodutos do que tratá-los posteriormente;

2)**Economia de átomos:** Os métodos sintéticos devem ser desenvolvidos para maximizar a incorporação dos átomos dos reagentes nos produtos finais desejados;

3)**Sínteses com compostos de menor toxicidade:** Sempre que possível deve-se substituir compostos de alta toxicidade por compostos de menor toxicidade nas reações químicas;

4)**Desenvolvimento de compostos seguros:** Os produtos químicos deverão ser desenvolvidos para cumprirem a função desejada, apresentando a menor toxicidade possível;

5)**Diminuição de solventes e auxiliares:** A utilização de substâncias auxiliares (solventes, agentes de separação, etc) deverá ser evitada quando possível, ou utilizar auxiliares inócuos no processo;

6)**Eficiência energética:** Os métodos sintéticos deverão ser conduzidos sempre que possível à pressão e temperatura ambientes, para diminuir a energia gasta durante um processo químico que representa um impacto econômico e ambiental.

# Os 12 Princípios da Química Verde

**7)Uso de substâncias recicladas: Os produtos e subprodutos de processos químicos deverão ser reutilizados sempre que possível;**

**8)Redução de derivativos: A derivatização desnecessária (uso de reagentes bloqueadores, de proteção ou desproteção, modificadores temporários) deverá ser minimizada ou evitada quando possível, pois estes passos reacionais requerem reagentes adicionais e, consequentemente, podem produzir subprodutos sem proveito;**

**9)Catálise: A aplicação de catalisadores para aumentar a velocidade e o rendimento dos processos químicos;**

**10)Desenvolvimento de compostos para degradação: produtos químicos deverão ser desenvolvidos de tal maneira que, ao fim de sua função se degrade em produtos inócuos, e não persista no ambiente;**

**11)Análise em tempo real para a prevenção da poluição: métodos analíticos precisam ser desenvolvidos para permitir o monitoramento do processo em tempo real, para controlar a formação de compostos tóxicos e**

**12)Química segura para a prevenção de acidentes: as substâncias usadas nos processos químicos deverão ser escolhidas para minimizar potenciais acidentes, tais como explosões e incêndios.**

# Química ambientalmente sustentável, amigável, verde, limpa...

**Desenvolvimento e implementação de processos e produtos químicos para reduzir ou eliminar o uso ou geração de substâncias nocivas à saúde humana e ao ambiente sustentável**


**P. T. Anastas, *Acc. Chem. Res*. 35, 686 (2002)**

# Desenvolvimento sustentável*

**Supre as necessidades do presente sem comprometer a habilidade das gerações futuras de suprir suas próprias necessidades.**

***Do latin: "sustinere" e significa "manter vivo", "defender".**

**Comissão Mundial sobre Meio Ambiente e Desenvolvimento –ONU (1987)**

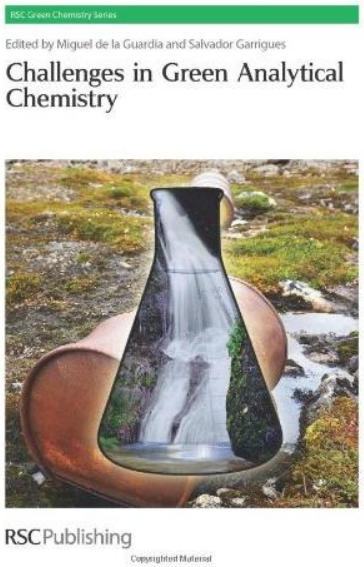
It is an interesting irony that in the process of measuring environmental problems the analytical chemistry methodologies used themselves contribute to further environmental problems

P. T. Anastas, *Crit. Rev. Anal. Chem.*, 29(3), 167 (1999)

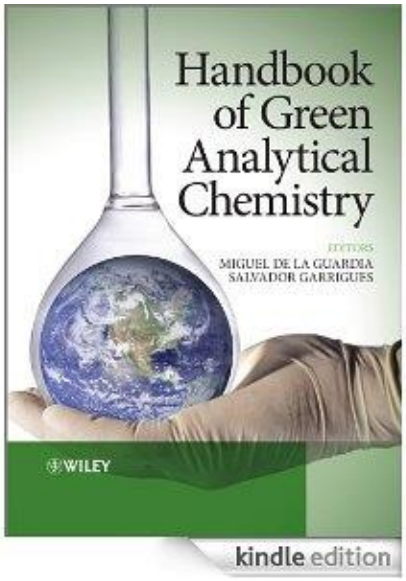
## Métodos verdes, limpos ou sustentáveis de análise

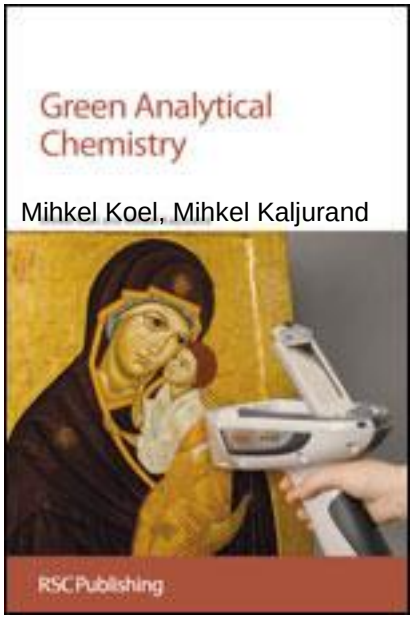
# Green (analytical) chemistry

# Publicações especializadas

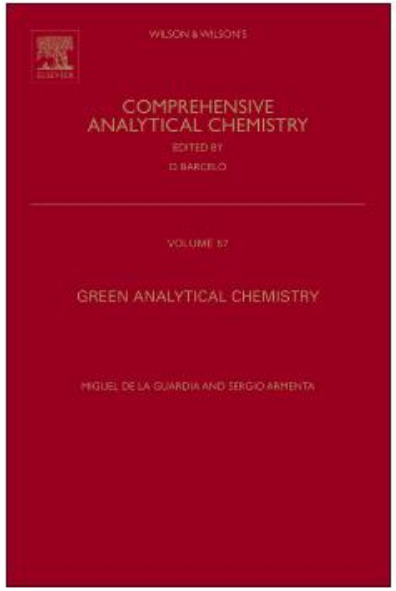




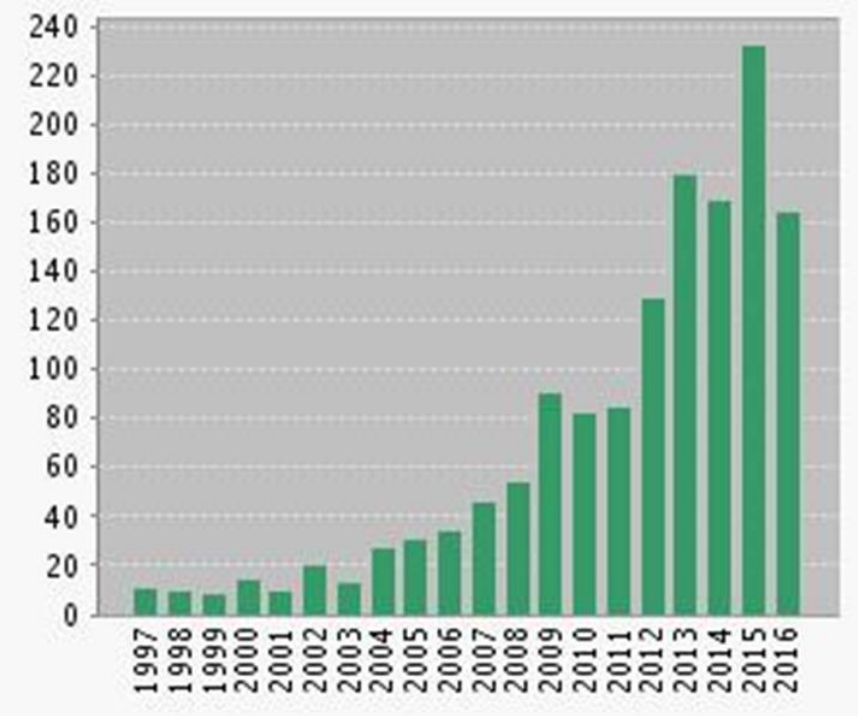


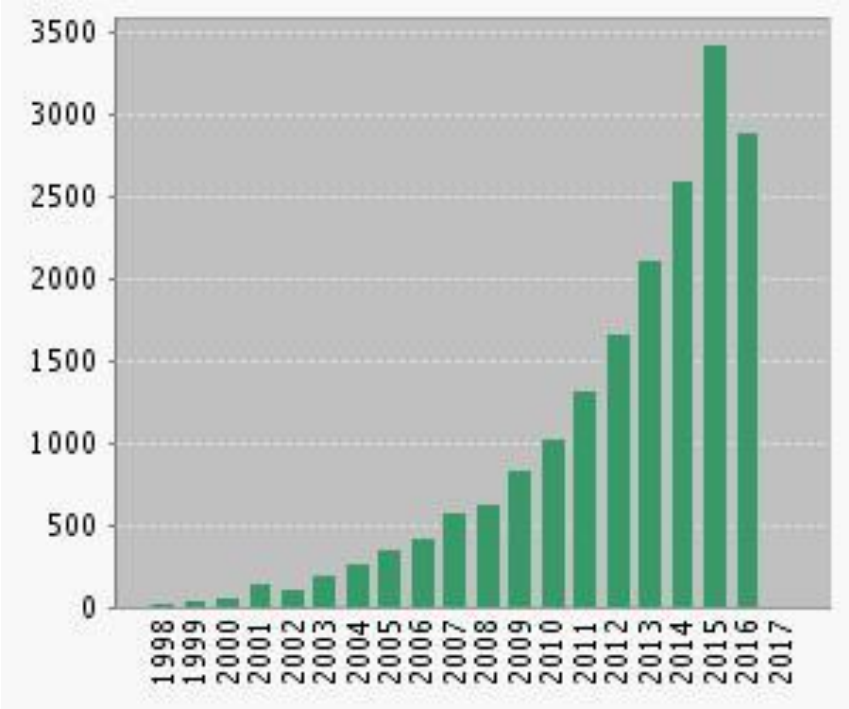







# Green analytical chemistry




**Web of science (12/09/2016): green analytical chemistry or sustainable analytical methods or benign analytical chemistry**

# Características analíticas

- ✓ Seletividade
- ✓ Sensibilidade
- ✓ Precisão
- ✓ Exatidão
- ✓ Geração de resíduos

# Avaliação dos métodos analíticos no contexto da Química Verde

## 4.1.
## *National Environmental Methods Index (NEMI)*

O *National Environmental Methods Index* (*NEMI*) foi criado nos EUA, em 2002, fruto de uma iniciativa conjunta do National Water Quality Monitoring Council e do Advisory Committee on Water designada como '*Methods and Data Comparability Board*' (MDCB). Desde então, o *NEMI* vem contribuindo para que a comunidade científica possa avaliar e comparar o desempenho de métodos de monitoramento ambiental, por meio do acesso a uma base de dados *on-line,* que contém informações sobre mais de 1.000 métodos e é consultada por 6.500 pessoas por mês, em média.

A iniciativa '*Methods and Data Comparability Board*' (MDCB) congrega especialistas de todos os níveis de governo, representantes da indústria e de organizações privadas nos EUA. Graças a um esforço de cooperação do Green

**www.nemi.gov**

TRI= Inventário de liberação tóxica

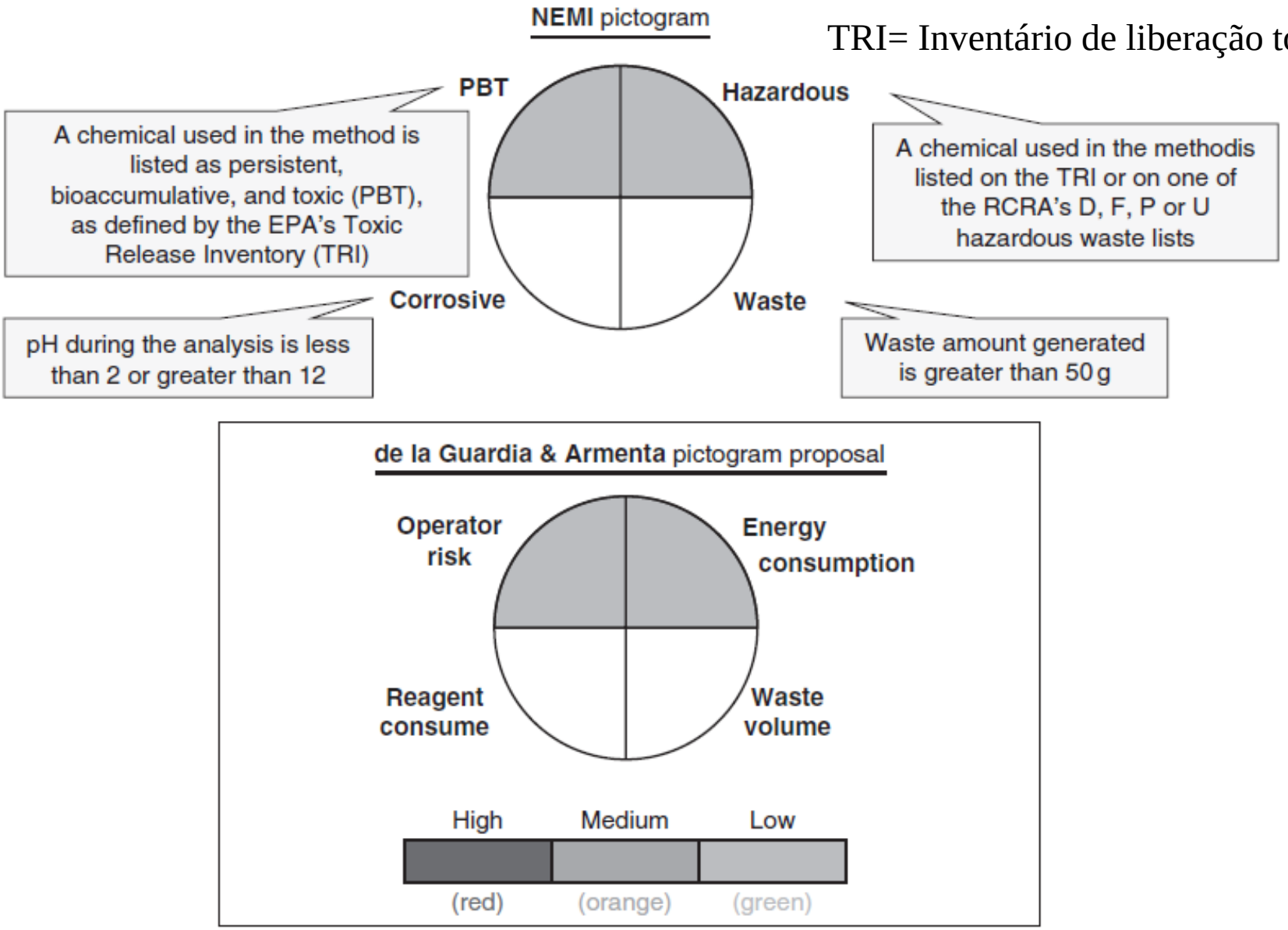



**Figura- a) Pictograma representativo do perfil verde segundo a National Environmental Methods Index (NEMI); b) . Pictograma de 'perfil verde' complementar de de la Guardia & Armenta (2001) ao pictograma do *NEMI***

# Flow analysis strategies to greener analytical chemistry. An overview

**Fábio R. P. Rocha,* Joaquim A. Nóbrega and Orlando Fatibello Filho**

*Departamento de Química, Centro de Ciências Exatas e de Tecnologia, Universidade Federal de São Carlos, Rodovia Washington Luíz, km 235, P.O. Box 676, 13560-970, São Carlos, SP, Brazil. E-mail: frprocha@cena.usp.br*

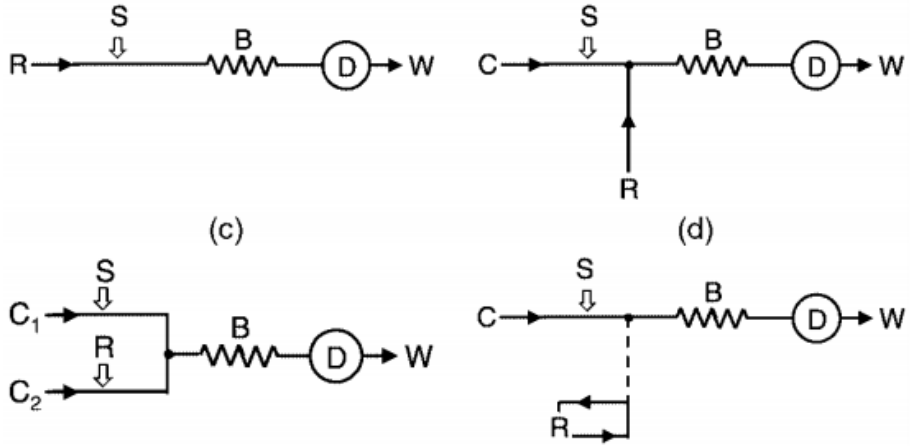



**Fig. 1** Flow-injection manifolds: (a) single line; (b) confluent streams; (c) merging zones and (d) intermittent stream. R = reagent; C, $C_1$, $C_2$ = carrier streams; S = sample; B = reactor; D = detector; W = waste vessel. Arrows indicate the points of sample or reagent introduction and the flow direction.

# Métodos limpos de análise

① Não há a produção de resíduos

② Emprego de reagentes não tóxicos

③ Minimização da quantidade de resíduos

④ Reciclagem/reutilização dos resíduos

⑤ Tratamento dos resíduos

F. R. P. Rocha, J. A. Nóbrega, O. Fatibello-Filho, *Green Chem.*, 3, 216 (2001)

Trends in Analytical Chemistry, Vol. 45, 2013



# Greening sample preparation in inorganic analysis

Diogo L. Rocha, Alex D. Batista, Fábio R.P. Rocha, George L. Donati, Joaquim A. Nóbrega

**Sample preparation aims to convert analytes to a more suitably detectable form, to separate them from the sample matrix or to concentrate species for trace analysis. Classical procedures often yield large amounts of toxic waste, but environment-friendly alternatives can be implemented without impairing the analytical performance. Green methods are also safer, and reduce costs and risks of sample contamination. This overview focuses on application of these strategies to inorganic analysis.**

**Table 1.** Greener approaches for sample decomposition

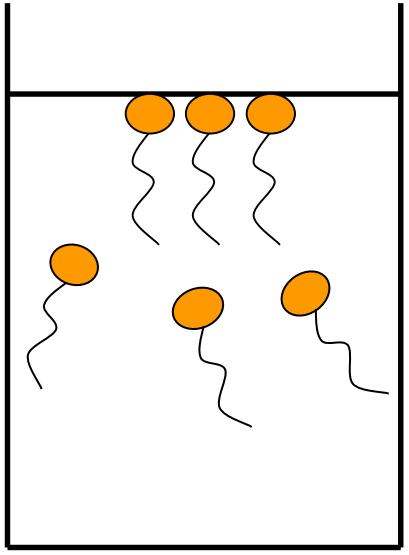
| Approach | Principle | Advantages | Drawbacks | Illustrative application | Green feature |
|---|---|---|---|---|---|
| Microwave with diluted acids | Regeneration of nitric acid promoted by oxygen and as a result of the temperature gradient in the microwave-assisted heated closed vessel | Greater safety, low sample contamination, lower blank values and less acidic digests | Relatively high RCC hinders application of some analytical techniques and determination of some analytes | Plant materials and biological fluids | Lower acid consumption |
| Vapor-phase decomposition | Interaction of the sample with acid vapors in closed systems | Low sample contamination, purification of the acids by distillation, lower blank values | Limited to volatile acids; different decomposition efficiencies in different vessels | Sediments and biological materials | |
| Microdigestions | Decomposition in micro-flasks with small amounts of sample and acid | Safety, low sample contamination | Sample representativity affected by small amount of sample; time-consuming procedures due to mild experimental conditions; small volume of the digest limits application for some detection techniques | Biological materials and airborne particulate | |
| Combustion | Elimination of organic matter at high temperatures and analyte transfer to an absorbing solution | Low RCC; low blank values; low acidic digests | Unsuitable for non-flammable samples | Petroleum coke and coal | |
| Photo-catalytic UV decomposition | Generation of highly-oxidizing free radicals under UV irradiation | Safer procedures; avoid using corrosive reagents; low energy consumption | More time-consuming; limited application for samples with high organic matter content | Waters, plant extracts and biological fluids | Use of less toxic reagents |

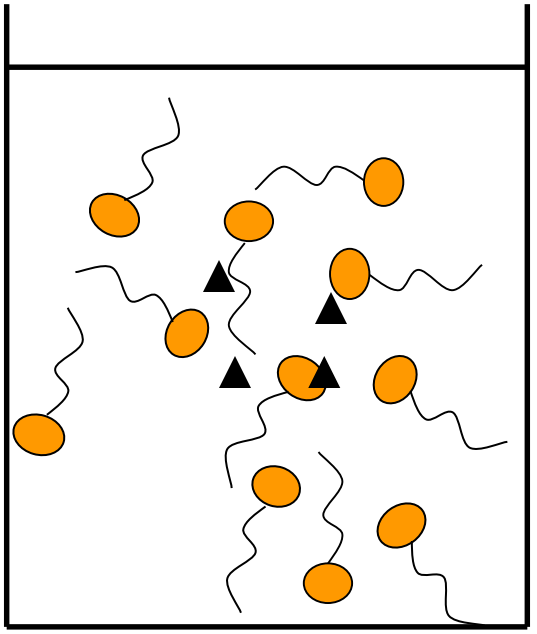
# Extração líquido-líquido

$$A_{(aq)} \rightleftharpoons A_{(org)}$$

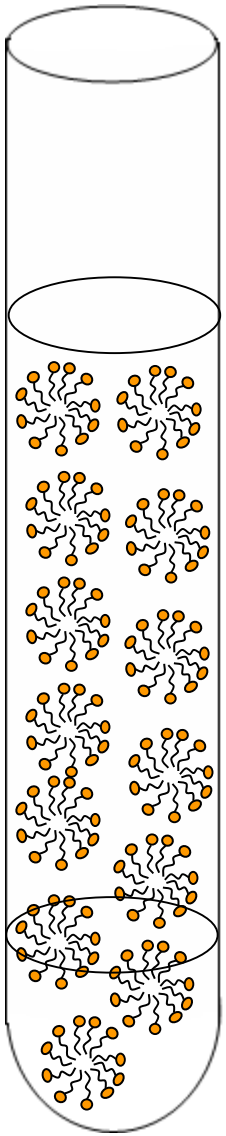
- Exaustivo
- Exposição do analista
- Solventes orgânicos
- Geração de efluentes

# Extração em ponto nuvem





# Extração em ponto nuvem



Q. Fang, M. Du, C.W. Huie, Anal. Chem. 73 (2001) 3502

**Fonte: Prof. Fábio R.P. Rocha- CENA-USP**

*Quim. Nova,* Vol. 35, No. 8, 1600-1605, 2012

# EXTRAÇÃO/PRÉ-CONCENTRAÇÃO DE ÍONS COBRE NO PONTO NUVEM EXPLORANDO A FORMAÇÃO DE COMPLEXOS COM DMIT [4,5-DIMERCAPTO-1,3-DITIOL-2-TIONATO]

**Bruna Fabrin Somera, Fernanda Midori de Oliveira, Wagner José Barreto e Sônia Regina Giancoli Barreto**
Departamento de Química, Universidade Estadual de Londrina, Rod. Celso Garcia Cid, PR 445 km 380, Campus Universitário, 86051-990 Londrina - PR, Brasil
**César Ricardo Teixeira Tarley***
Departamento de Química, Universidade Estadual de Londrina, Rod. Celso Garcia Cid, PR 445 km 380, Campus Universitário, 86051-990 Londrina – PR / Instituto Nacional de Ciência e Tecnologia de Bioanalítica, Universidade Estadual de Campinas, CP 6154, 13083-970 Campinas – SP, Brasil.

Recebido em 24/1/12; aceito em 17/4/12; publicado na web em 20/7/2012

# Extração com fluído supercrítico

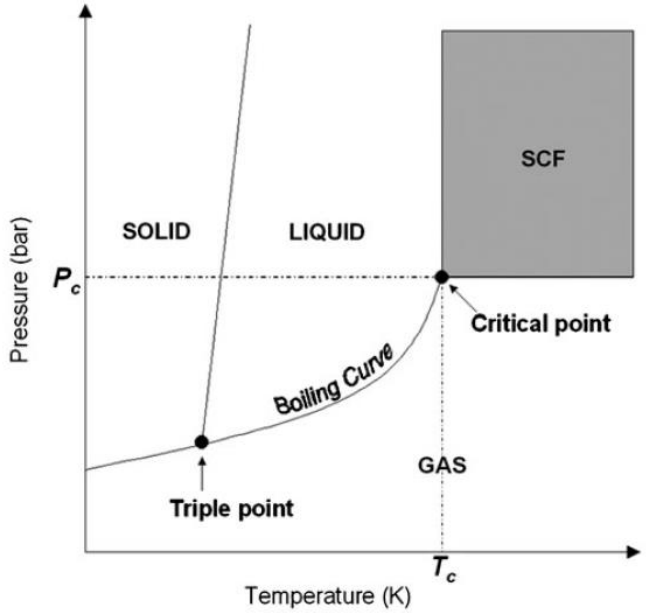
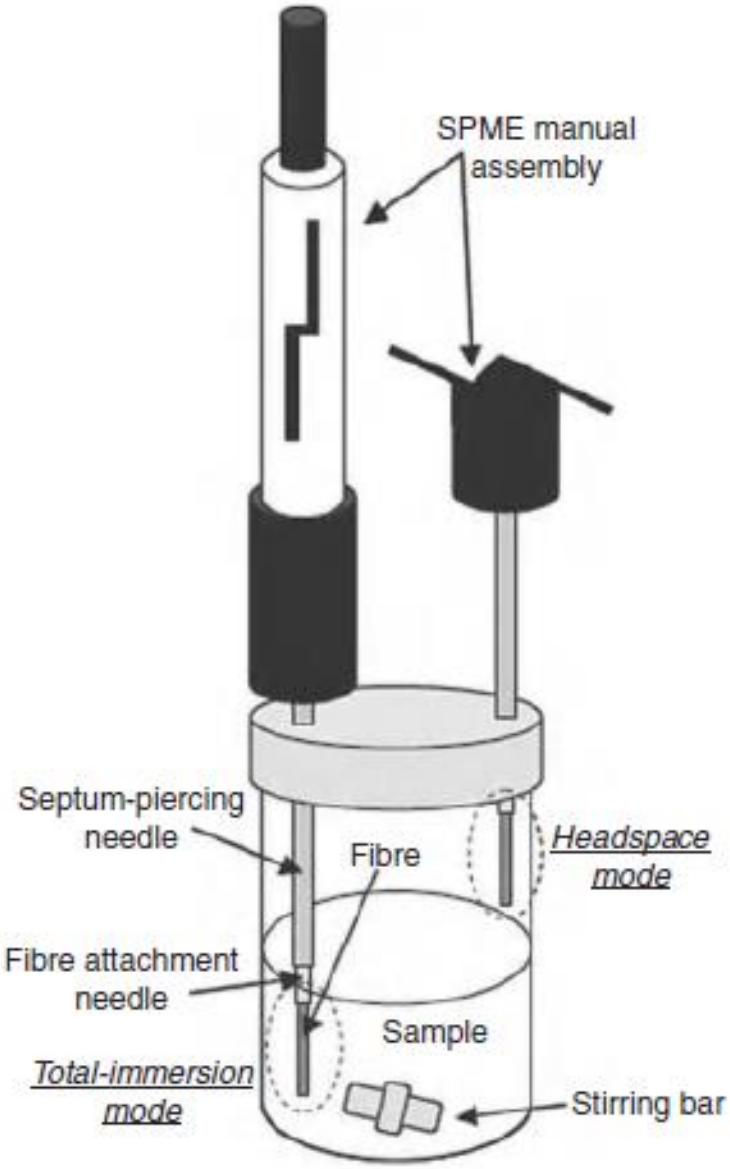



**Figure 4.1** Single-component phase diagram highlighting the supercritical fluid (SCF) region and the critical point.

**Table 4.1** Critical point ($T_c$ and $P_c$) and critical density for selected compounds.

| *Substance* | $T_c/K$ | $P_c/atm$ | $\rho/g\,ml^{-1}$ |
|---|---|---|---|
| $CHF_3$ | 299.3 | 46.9 | 0.528 |
| $CH_4$ | 190.5 | 41.4 | 0.162 |
| $C_2H_4$ | 282.3 | 50.5 | 0.215 |
| $C_2H_6$ | 305.2 | 48.2 | 0.203 |
| $CO_2$ | 304.1 | 72.8 | 0.469 |
| $H_2O$ | 647.1 | 218.3 | 0.348 |
| $CH_3CH_2OH$ | 513.9 | 60.6 | 0.276 |
| Xe | 289.7 | 58 | 1.110 |

# Microextração em fase sólida



# 1. Redução ou eliminação de reagentes


# Determinação de cloreto com redução dos resíduos

$$Hg(SCN)_2 + 2\ Cl^- \longrightarrow HgCl_2 + 2\ SCN^-$$

$$SCN^- + Fe^{3+} \longrightarrow [FeSCN]^{2+} \quad (\lambda = 480\ nm)$$

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Talanta 65 (2005) 965–970

Talanta

www.elsevier.com/locate/talanta

## Flow injection spectrophotometric method for chloride determination in natural waters using $Hg(SCN)_2$ immobilized in epoxy resin

Claudineia R. Silva[a], Heberth J. Vieira[a], Larissa S. Canaes[a], Joaquim A. Nóbrega[b], Orlando Fatibello-Filho[a,*]

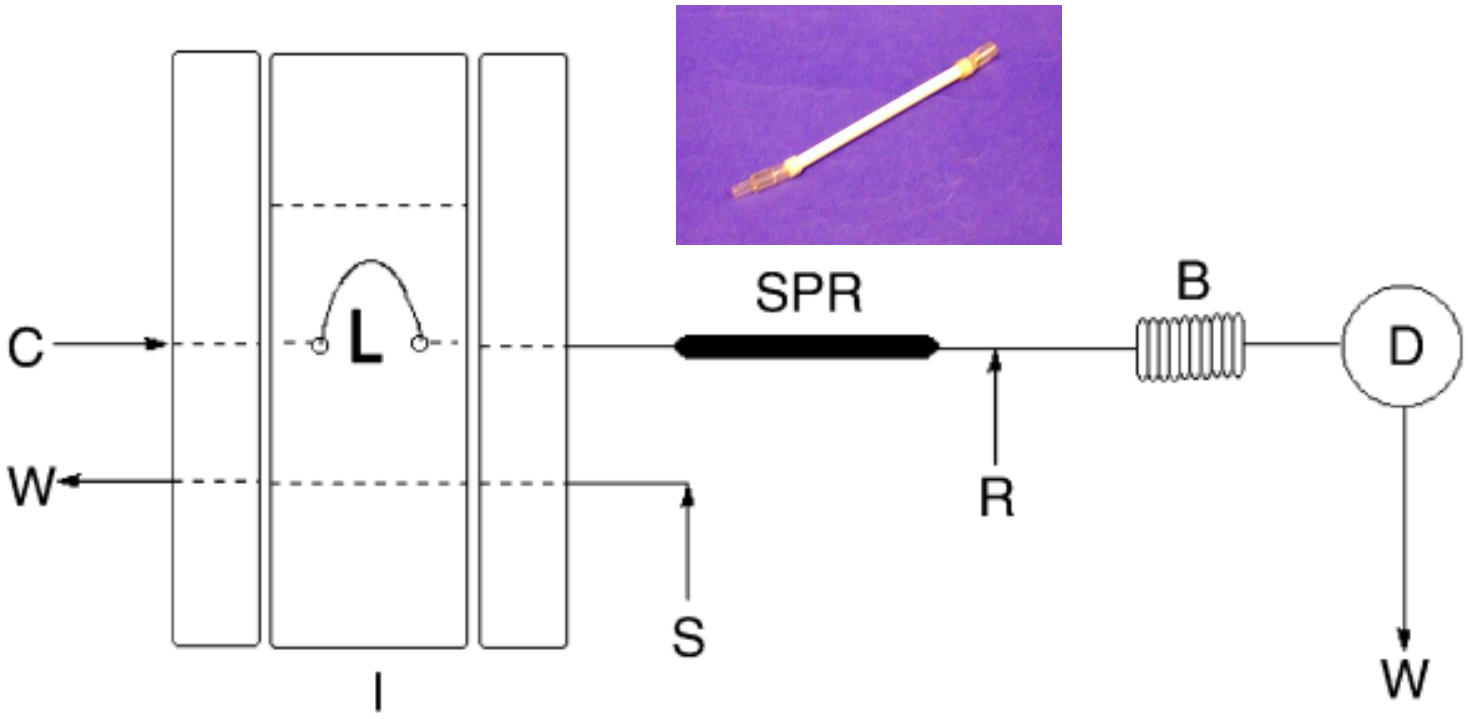



**Fig. Diagrama esquemático do sistema em fluxo. (a) C= água; S= amostra ou padrões de cloreto; SPR = reator em fase sólida com $Hg(SCN)_2$ imobilizado; B= bobina reacional, D= detector (λ= 480 nm), W= resíduos**

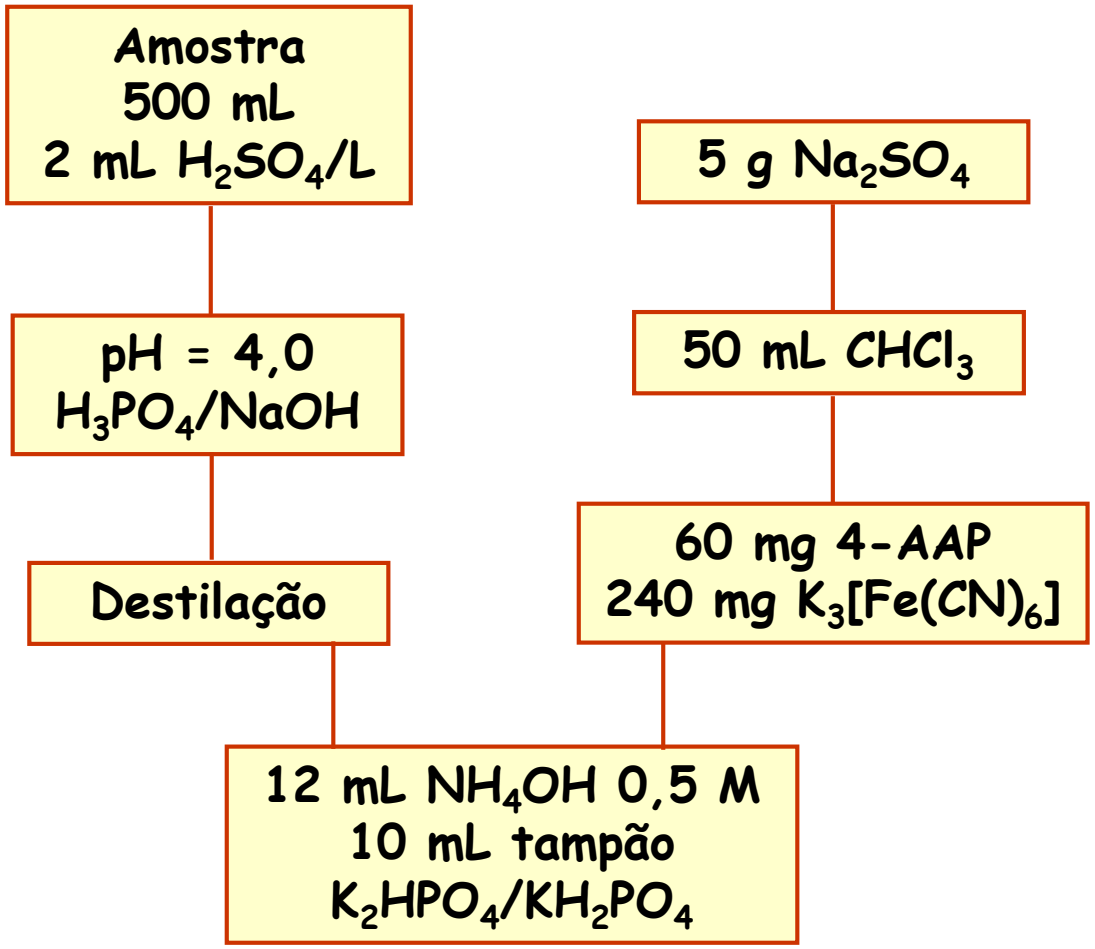
**Consumo de apenas 120 µg Hg e W ([Hg]) < 2 µg/L) CONAMA 357/2005) ou < 10 µg/L CONAMA 430/2011 (efluentes)**

**W (Mesmo assim os resíduos foram tratados com tioacetoamida)**

**Tabela-Alguns trabalhos publicados pelo LABBES empregando RFS com diferentes reagentes imobilizados para a determinação de produtos farmacêuticos**

| Analito | Reagente | Produto Detectado | λ / nm |
|---|---|---|---|
| ácido ascórbico | $Cu_3(PO_4)_2$ | Cu(I)-batocuproína | 480 |
| aspartame | $Zn_3(PO_4)_2$ | $Zn(VA\text{-}BO_3)_3$ | 540 |
| N-acetilcisteína | $Zn_3(PO_4)_2$ | $Zn(VA\text{-}BO_3)_3$ | 540 |
| ácido ascórbico | $Fe(OH)_3$ | ferroína | 510 |
| adrenalina | $I_3^-$ | adrenocromo | 488 |
| adrenalina | $MnO_2$ | adrenocromo | 488 |
| L-dopa | $MnO_2$ | dopacromo | 520 |
| isoproterenol | $MnO_2$ | isoproterocromo | 492 |

# Determinação de fenóis totais




**Quantidades para 100 determinações**

- 50 mL $NH_4OH$
- 100 g $K_2HPO_4$
- 70 g $KH_2PO_4$
- 6 g 4-aminoantipirina
- 24 g $K_3[Fe(CN)_6]$
- 2,5 – 5 L $CHCl_3$
- 500 g $NaSO_4$

~ 60 L resíduo

** A. D. Eaton, L.S. Clesceri, A.E. Greenberg, Standard Methods for the Examination of Water and Wastewater, 20ª ed., American Public Health Association, Washington, 1998.

Available online at www.sciencedirect.com
SCIENCE DIRECT®
Talanta 62 (2004) 463–467
Talanta
www.elsevier.com/locate/talanta

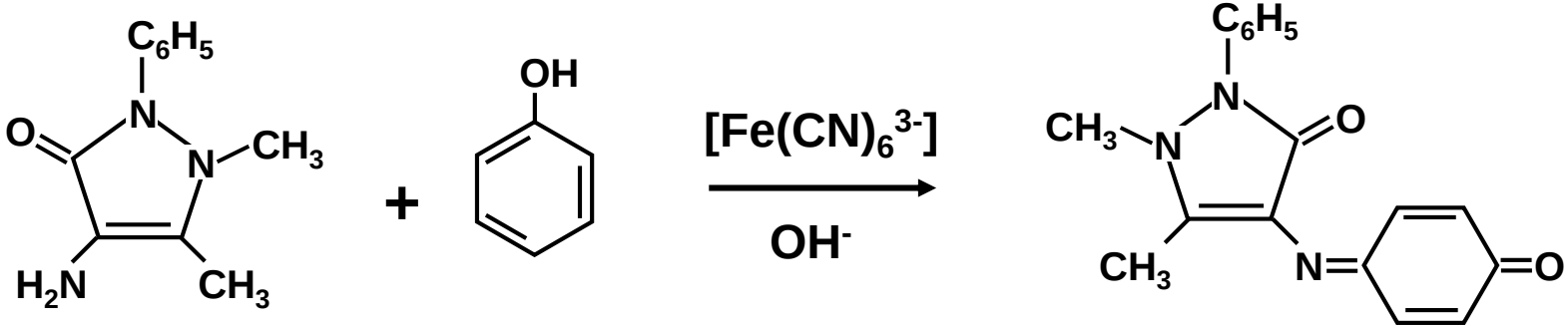
# An improved flow system for phenols determination exploiting multicommutation and long pathlength spectrophotometry

Karina Omuro Lupetti [a], Fábio R.P. Rocha [b,*], Orlando Fatibello-Filho [a]

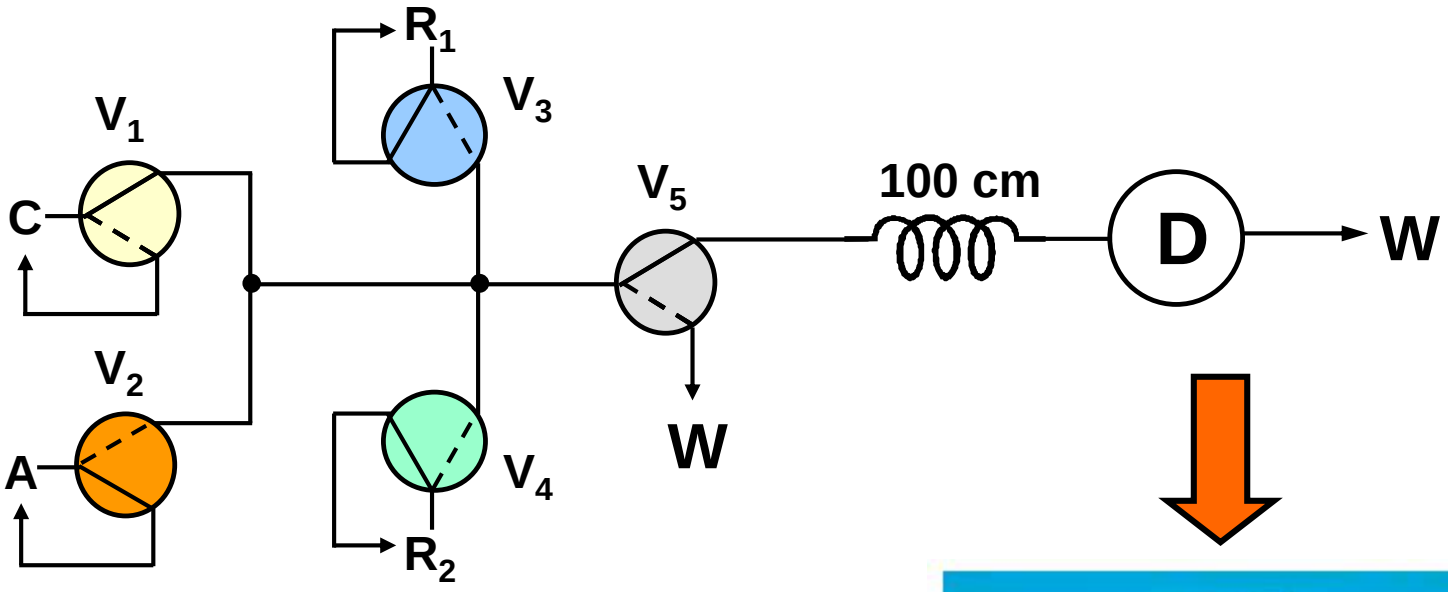
[a] *Departamento de Química, Universidade Federal de São Carlos, PO Box 676, 13560-970 São Carlos-SP, Brazil*
[b] *Instituto de Química, Universidade de São Paulo, PO Box 26077, 05513-970 São Paulo-SP, Brazil*

Received 23 September 2002; received in revised form 6 January 2003; accepted 18 August 2003

- **4-aminoantipirina**



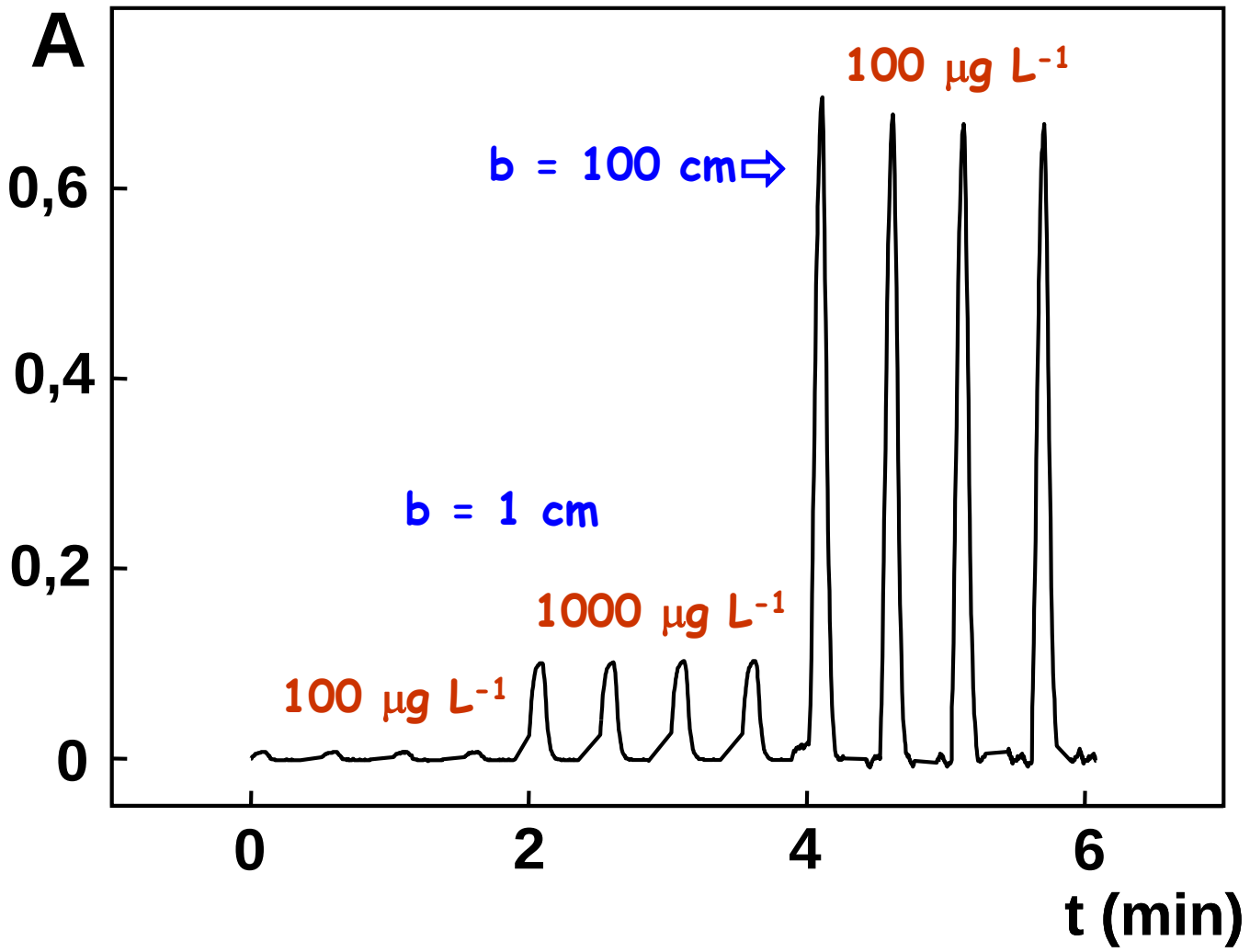
# Determinação de fenóis totais





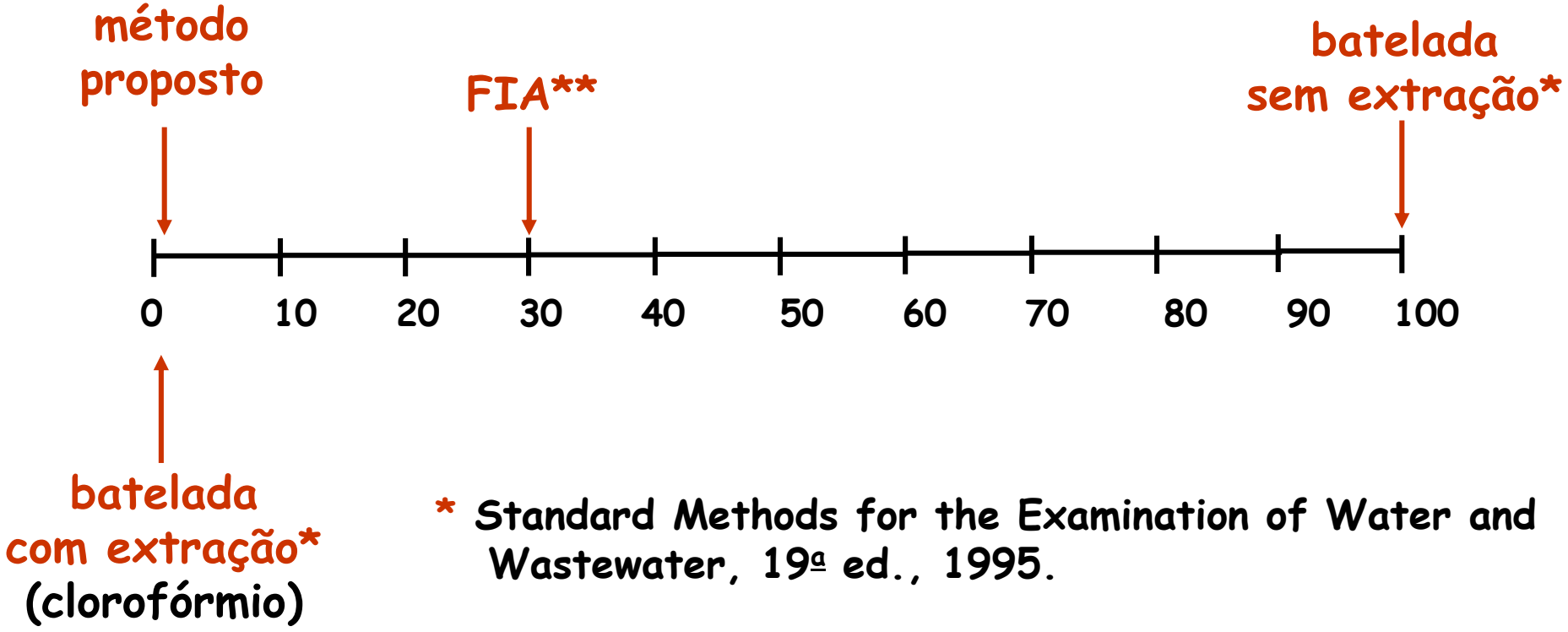
Fonte: Prof. Fábio R.P. Rocha- CENA-USP

# Determinação de fenóis totais



# Características analíticas

Limite de detecção ($\mu g\ L^{-1}$)




* Standard Methods for the Examination of Water and Wastewater, 19ª ed., 1995.

** Anal. Chim. Acta, 261 (1992) 253.

Table 3
Consumption of reagents and sample per determination in different procedures for determination of phenols

| | Proposed method | Literature | |
|---|---|---|---|
| | | Flow injection [14][a] | Batch method [10] |
| 4-AAP (mg) | 0.10 | 0.80 | 20 |
| $K_3[Fe(CN)_6]$ (mg) | 0.12 | 2.4 | 80 |
| $CHCl_3$ (ml) | – | – | 25–50 |
| Sample (ml) | 0.66 | 0.40 | 500 |

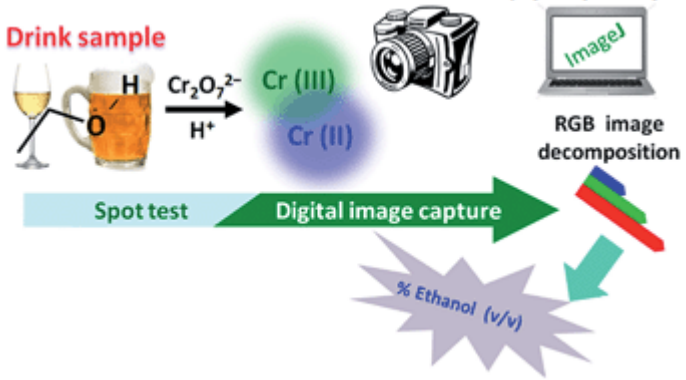
[a] Flow-injection system with continuous reagent addition.

CrossMark click for updates

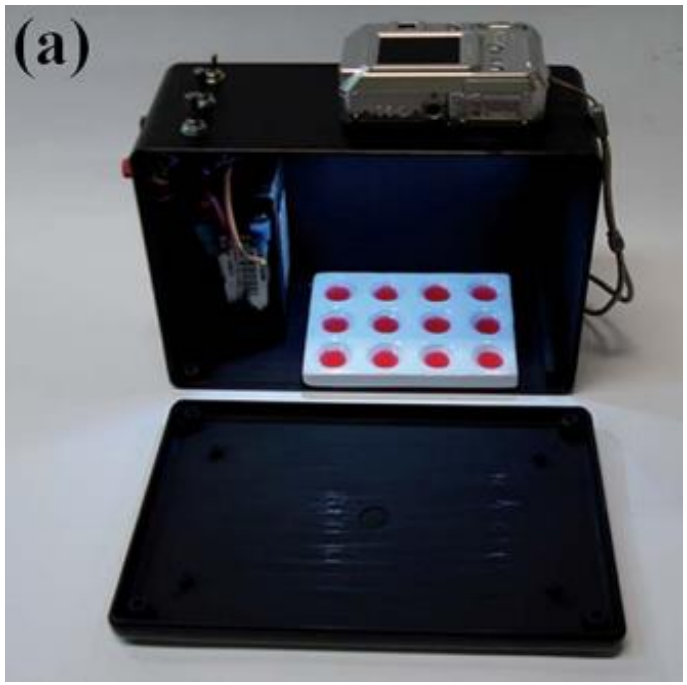
Cite this: *Anal. Methods*, 2015, 7, 4138

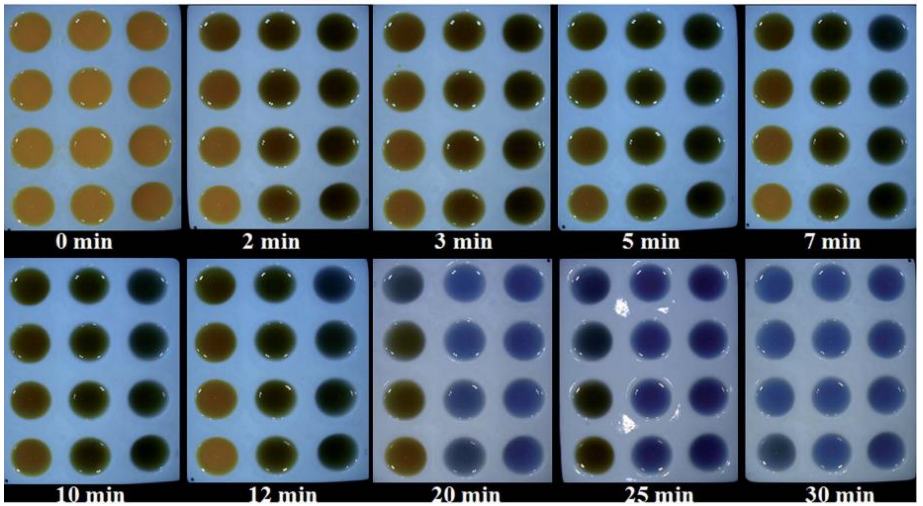
# A digital image-based method employing a spot-test for quantification of ethanol in drinks†

Luzia Pires dos Santos Benedetti,[a] Vagner Bezerra dos Santos,*[a] Tiago Almeida Silva,[a] Edemar Benedetti Filho,[b] Valdomiro Lacerda Martins[c] and Orlando Fatibello-Filho[a]




$$Cr_2O_7^{2-}{}_{(aq)} + 8H_{(aq)}^{+} + 3CH_3CH_2OH_{(l)} \rightarrow 2Cr_{(aq)}^{3+} + 3CH_3CHO_{(aq)} + 7H_2O_{(l)}$$




## Digital image treatment

The captured images were analyzed using the freely available software ImageJ. In this software, a defined number of pixels from the digital image was selected (42 × 36), and these pixels were decomposed using the RGB approach, *i.e.*, values of mean and mode referent to the color channels red (R), green (G), and blue (B) partially absorbed by the solution and further reflected to the detector, and then imported to Excel® software.

# 2. Substituição de reagentes e/ou material

# Método mais limpo para cloreto  com substituição do reagente

Available online at www.sciencedirect.com
ScienceDirect
Talanta 72 (2007) 663–667
Talanta
www.elsevier.com/locate/talanta

## An improved flow system for chloride determination in natural waters exploiting solid-phase reactor and long pathlength spectrophotometry

Viviane G. Bonifácio, Luiz C. Figueiredo-Filho, Luiz H. Marcolino Jr., Orlando Fatibello-Filho *

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Received 30 August 2006; received in revised form 22 November 2006; accepted 22 November 2006
Available online 22 December 2006

**Concentração de cloreto em águas naturais variou de 14,9 a 43,2 mg/L**

**Ácido cloroanílico + 2 $Ag^+$ ⇌ $Ag_2Ch$ + 2 $H^+$**

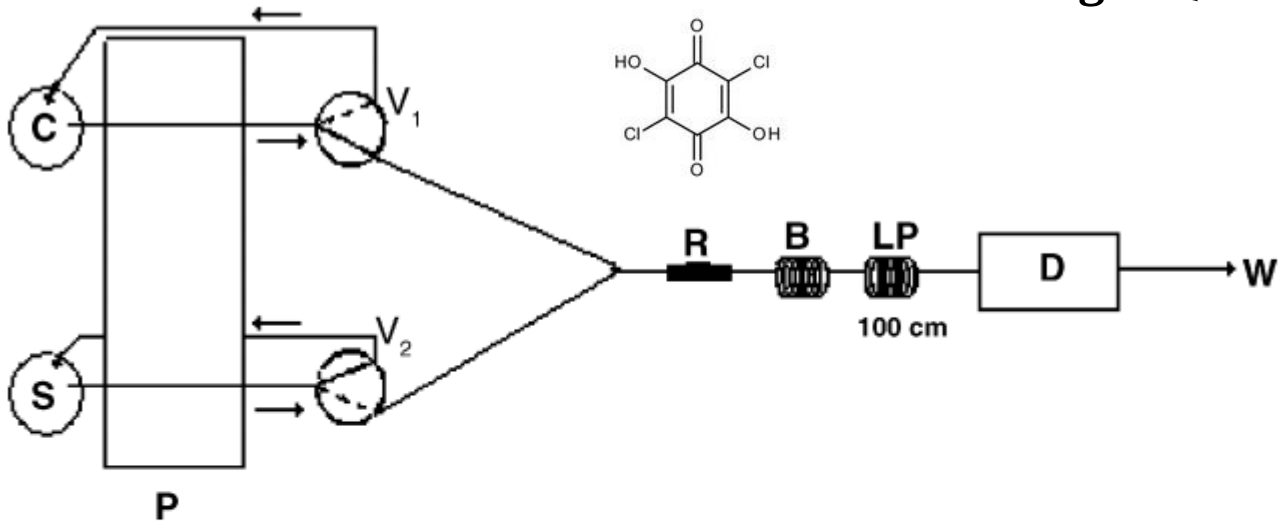


Fig. 1. Flow diagram of the system for determination of chloride ion in natural water: P, peristaltic pump; C, water carrier (2.0 ml $min^{-1}$); S, sample (2.0 ml $min^{-1}$); $V_1$ and $V_2$, three-way solenoid valves; R, solid phase reactor with immobilized silver chloranilate; B, reaction coil (50 cm); LP, long path-length flow cell (100 cm optical path); D, spectrophotometer USB2000 (Ocean Optics); W, waste. Dashed lines represent the flow paths when the valves are switched on.

$$2Cl^-(aq) + Ag_2Ch(s) \rightleftharpoons 2AgCl(s) + Ch^{2-}(aq) \quad \lambda = 530\,nm$$

where $Ch^{2-}$ represents the chloranilate anion.

# Fruits and vegetables employed as the PER, PPO, urease sources

**Avocado**
***(Persea americana)***

**Zucchini**
***(Cucurbita pepo)***

**Artichoke**
***(Cynara scolymus L.)***

**Banana**
***(Musa paradisiaca)***

**Sweet potato**
***(Ipomoea batatas L. Lam.)***

**Potato**
**(*Solanum tuberosum*)**

**Eggplant**
***(Solanum melongena)***

**Cara root**
***(Dioscorea bulbifera)***

**Coconut**
***(Cocus nucifera L.)***

**Yam**
***(Alocasia macrorhiza)***

**Jack fruit**
***(Artocarpus integrifolia L.)***

**Manioc**
***(Manihot utilissima)***

**Turnip**
***(Brassica campestre ssp.)***

**Peach**
***(Prunus persica)***

**Radish**
**(*Raphanus sativus*)**

# The case for the use of unrefined natural reagents in analytical chemistry—A green chemical perspective

**Kate Grudpan,[a] Supaporn Kradtap Hartwell,[a] Somchai Lapanantnoppakhun[a] and Ian McKelvie[b]**

***Received 19th April 2010, Accepted 16th July 2010***
**DOI: 10.1039/c0ay00253d**

An important part of the Green Chemistry philosophy is the need to develop and adopt green analytical techniques and procedures. These include the reduction of reagent and solvent usage, the minimization of solid, liquid and gaseous materials produced by analytical processes, the replacement of reagents and solvents of high occupational or environmental toxicity with much more innocuous materials, and the reduction of energy use in analytical processes. One aspect that has received little attention in this context is the use of unrefined natural reagents derived from plant and animal tissues or microbial cells. Crude plant extracts may contain chemical compounds that enable their use as indicators in acid–base or redox titrations, or as chromogenic or fluorogenic agents. Enzymes extracted from plants may be used directly in soluble form, or incorporated in biosensors or solid phase reactors, for analytical measurements, or as a means of removing interferences or performing speciation studies. The use of natural reagents in conjunction with a flow injection system can confer a number of advantages. The enhanced kinetic control that flow analysis offers may assist in avoiding undesirable side reactions that would otherwise occur using unpurified reagents. The lifetime of natural reagents may be prolonged when used in a flow analysis system because their exposure to light or air can be controlled. Any changes in response that do occur may be readily corrected by regular standard checking and recalibration. This article reviews the use of natural reagents with an emphasis on flow-based analytical systems, and makes the case for further research in this latent area of green analytical chemistry.

**K. Grudpan *et al.*; *Anal. Methods*,2, 1651 (2010).**

*Quim. Nova,* Vol. 25, No. 3, 455-464, 2002.

# USO ANALÍTICO DE TECIDOS E DE EXTRATOS BRUTOS VEGETAIS COMO FONTE ENZIMÁTICA

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Recebido em 22/3/01; aceito em 9/8/01

ARTIGO

# BIOSSENSOR AMPEROMÉTRICO PARA A DETERMINAÇÃO DE FENÓIS USANDO UM EXTRATO BRUTO DE INHAME *(Alocasia macrorhiza)*

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Recebido em 7/6/93; cópia revisada em 9/9/93

**Crude extracts of various vegetables and fruits such as eggplant (*Solanum melongena*), potato (*Solanum tuberosum*), manioc (*Manihot utilissima*), yam (*Alocasia macrorhiza*), apple (*Pirus malus*), dwarf banana (*Musa acuminata*), apple-flavored banana(*Musa parasidiaca*), most common variety of banana-"banana-prata" (*Musa parasidiaca*), pear (*Pirus communis*) and peach (*Prunus persica*) are studied as a biocatalytic material for the aerobic oxidation of phenolic substrates. Of those, yam (*Alocasia macrorhiza*) crude extract is found to be the best source of *polyphenol oxidase* [E.C.1.10.3.1], due their stability and/or activity (u/mg). Thus, an amperometric biosensor is constructed by the immobilization of yam crude extract with glutaraldehyde and bovine serum albumin onto an oxygen electrode. The proposed bioamperometric sensor provides a linear response for pyrogallol, catechol, phenol and p-cresol in the concentration ranges of $2.5x10^{-5}$— $8.0x10^{-5}$M; $1.0x10^{-5}$— $8.5x10^{-5}$M; $1.0x10^{-5}$— $9.0x10^{-5}$ M and $1.0x10^{-5}$—$1.0x10^{-4}$M respectively; with response time of 1-4 min. and a useful lifetime of at least two weeks. Application of this biosensor for the determination of phenols in local industries waste waters is presented.**

**Keywords: enzyme electrode; phenolic substrates; catechol; pyrogallol; yam crude extract.**

**C. A. Signori; O. Fatibello-Filho, *Quimica Nova*, 17(1), 38-42 (1994)**

# UTILIZAÇÃO DO EXTRATO BRUTO DE FRUTOS DE *Solanum nigrum L* NO ENSINO DE QUÍMICA

Luiz Antônio RAMOS*
Karina Omuro LUPETTI*
Éder Tadeu Gomes CAVALHEIRO*
Orlando FATIBELLO-FILHO*

**RESUMO:** As antocianinas, pigmentos da classe dos flavonóides, são os principais substâncias cromogênicas encontrados nas flores vermelhas, azuis e púrpuras. Uma das principais características das antocianinas, com aproveitamento didático é a sua mudança de coloração em função do pH do meio. Este trabalho se insere em um projeto mais amplo, que visa propor experimentos simples e de baixo custo para ensino, utilizando-se de vegetais facilmente encontrados no Brasil. Os corantes utilizados se prestam ao ensino desde conceitos básicos de equilíbrio químico para estudantes de ensino médio, de indicadores em titulação para cursos de química geral e até da Lei de Lambert-Beer e de Espectros de Absorção, para cursos de instrumentação. A espécie utilizada foi *Solanum nigrum L* (maria-preta), cujo extrato bruto foi utilizado como agente cromóforo. Usando o extrato bruto como indicador em titulações ácido-base, observaram-se erro relativos da ordem de 0,11-1,0%, quando comparado com os resultados potenciométricos. A mudança de cor observada foi de vermelho para amarelo, entre pH 4 e 10. Espectros de absorção na região do UV e do visível foram obtidos em diferentes pH, para determinação dos comprimentos de onda dos máximos de absorção, bem como para demonstração da mudança da forma destes espectros em função da acidez do meio.
**PALAVRAS-CHAVE:** Corantes naturais, Antocianinas, Ensino de química.

**Eclética Quimica (2000)**

# Experimento Simples e Rápido Ilustrando a Hidrólise de Sais

**Orlando Fatibello-Filho, Lúcia Daniela Wolf, Mônica Helena M.T. Assumpção e Oldair D. Leite**

Neste artigo é proposto um experimento simples realizado com material de fácil aquisição para ilustrar as reações de hidrólise de cátions e ânions. São empregadas algumas soluções salinas, sendo a mudança de pH dessas soluções visualizada com um indicador universal de pH extraído do repolho roxo. O experimento permite assimilar os conceitos e/ou conteúdos envolvidos nas reações de hidrólise de sais ácidos, básicos e/ou neutros, bem como calcular o pH final dessas soluções salinas e relacioná-lo à mudança de cor do(s) indicador(es) de pH.

▶ hidrólise, sais ácidos, sais básicos, sais neutros, cálculo de pH ◀

Recebido em 28/3/05, aceito em 2/6/06

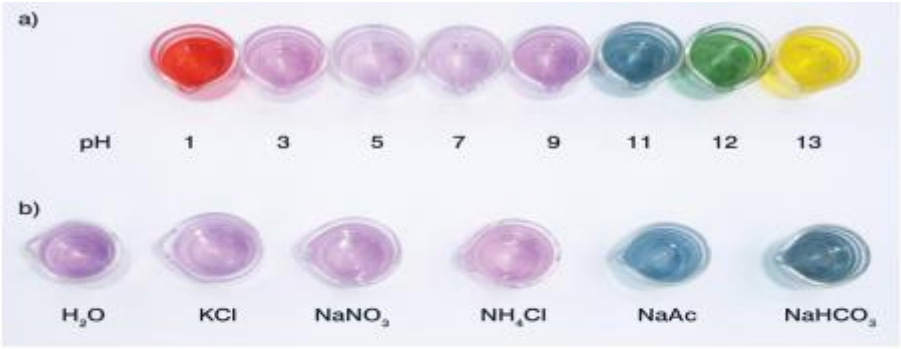


Figura 2: a) Escala padrão de pH: soluções de referência de ácido e base nos valores de pH variando de 1 a 13 contendo extrato de repolho roxo como indicador universal de pH. b) Água e soluções de cloreto de potássio, nitrato de sódio, cloreto de amônio, acetato de sódio e hidrogenocarbonato de sódio de concentração 0,5 mol/L contendo extrato de repolho roxo como indicador de pH.

J Solid State Electrochem (2009) 13:665–669
DOI 10.1007/s10008-008-0580-9

ORIGINAL PAPER

# Determination of the chemical oxygen demand (COD) using a copper electrode: a clean alternative method

**Claudineia R. Silva · Cleone D. C. Conceição · Viviane G. Bonifácio · Orlando Fatibello Filho · Marcos F. S. Teixeira**

$$Cu^{II}O_{(s)} + OH^{-}_{(ads)} \rightarrow Cu^{III}OOH^{\bullet}_{(s)} + e^{-}$$

$$Cu^{III}OOH^{\bullet}_{(s)} + Organic_{(red)} \rightarrow Cu^{II}O_{(s)} + Organic_{(oxid)} + H_2O \quad ($$

**Table 1** Comparison of COD values (mg $l^{-1}$) obtained by the proposed method with those by standard method [1]

| Samples | COD values (mg $l^{-1}$) | | $E_r$ (%) |
|---|---|---|---|
| | Standard method[a] | Proposed method[a] | |
| A | 13,010±25 | 12,893±18 | −8.9 |
| B | 16,032±20 | 15,006±12 | −6.4 |
| C | 19,209±23 | 17,800±20 | −7.3 |
| D | 1,740±10 | 1,410±5 | −19 |

$E_r$ relative error = proposed method vs. conventional method
[a] Mean of three determinations ± SD

# Substituição do eletrodo de mercúrio pelo eletrodo de amálgama

J Solid State Electrochem (2011) 15:2023–2029
DOI 10.1007/s10008-010-1225-3

ORIGINAL PAPER

## The effect of composition of solid silver amalgam electrodes on their electrochemical response

Djenaine De Souza · Lucia Helena Mascaro · Orlando Fatibello-Filho

Revisão

*Quim. Nova,* Vol. 34, No. 3, 487-496, 2011

## UTILIZAÇÃO DE ELETRODOS SÓLIDOS DE AMÁLGAMA PARA A DETERMINAÇÃO ANALÍTICA DE COMPOSTOS ORGÂNICOS E INORGÂNICOS

**Djenaine de Souza*, Luisa Célia Melo, Adriana Nunes Correia e Pedro de Lima-Neto**
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**Orlando Fatibello-Filho e Lucia Helena Mascaro**
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Recebido em 17/4/10; aceito em 28/9/10; publicado na web em 26/1/11

UTILIZATION OF THE SOLID AMALGAM ELECTRODES IN THE ANALYTICAL DETERMINATION OF ORGANIC AND INORGANIC COMPOUNDS. This review reports the use of solid amalgam electrodes in the electroanalytical determination of organic and inorganic compounds. The different types of amalgam electrodes are presented, and attention is paid to solid amalgam electrode, and here is presented details about the pre-treatment for activation and renovation and also possible modifications in its surface. The wide potential range, higher signal-to-noise ratio, mechanical stability enabling their application in flowing systems, and principally their resistance toward passivation, indicate that the solid amalgam electrodes are environmentally friendly alternatives to mercury electrodes, without loss in the sensitivity and reproducibility in voltammetric responses.

Keywords: solid amalgam electrodes; mercury; environmentally friendly sensor.

# Substituição do eletrodo de mercúrio pelo eletrodo de filme de bismuto

## Determination of Atrazine in Natural Water Samples by Differential Pulse Adsorptive Stripping Voltammetry Using a Bismuth Film Electrode

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Received: August 5, 2011
Accepted: November 11, 2011

**Abstract**
A new method using differential pulse adsorptive stripping voltammetry for the determination of atrazine (ATZ) in natural water samples using a bismuth film electrode (BiFE) is proposed. The calibration curve was linear in the atrazine concentration range from $6.7\times10^{-7}$ to $2.0\times10^{-5}$ mol $L^{-1}$, with a limit of detection (*LOD*) of $1.4\times10^{-7}$ mol $L^{-1}$. The proposed electrode was applied for atrazine determination with satisfactory results compared with a high-performance liquid chromatography method (HPLC).

**Keywords:** Bismuth film electrode (BiFE), Environmental samples, Differential pulse adsorptive stripping voltammetry (DPAdSV), Atrazine determination

# Substituição do eletrodo de mercúrio pelo eletrodo de filme de bismuto

Sensors and Actuators B 188 (2013) 334–339

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

## Differential pulse adsorptive stripping voltammetric determination of nanomolar levels of methotrexate utilizing bismuth film modified electrodes

Deborah Asbahr[a], Luiz Carlos S. Figueiredo-Filho[a], Fernando Campanhã Vicentini[a], Geiser Gabriel Oliveira[a], Orlando Fatibello-Filho[a], Craig E. Banks[b,*,1]

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Sensors and Actuators B 240 (2017) 749–756

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

# A disposable and inexpensive bismuth film minisensor for a voltammetric determination of diquat and paraquat pesticides in natural water samples

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[c] Departamento de Ciências da Natureza, Matemática e Educação, Universidade Federal de São Carlos, 13600-970 Araras, SP, Brazil

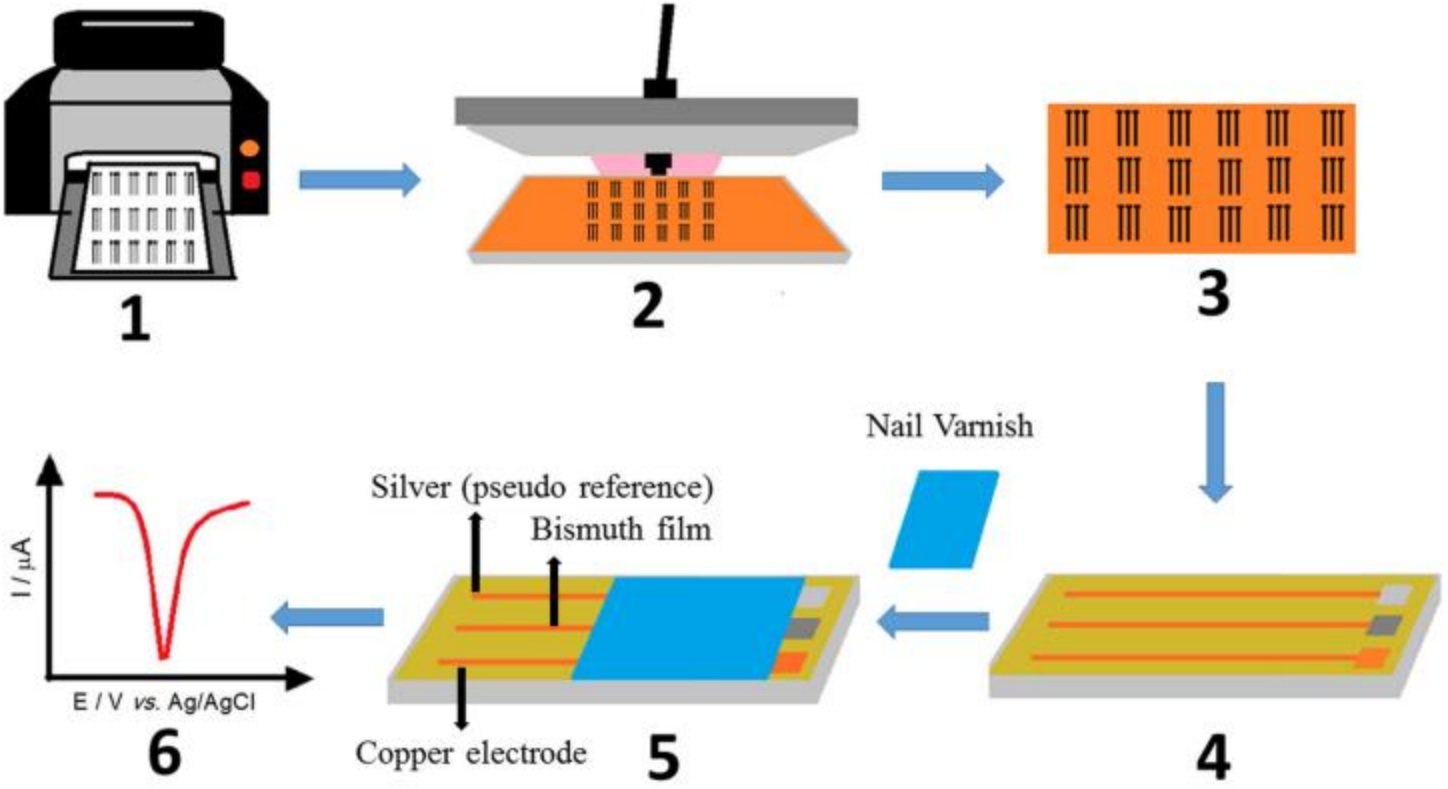


**Fig. 1.** Scheme of the disposable minisensor fabrication. Step 1: Print the layouts of the devices upon a polyester paper. Step 2: The design is transferred to the copper board at 120 °C for 240 s, using a thermal press. Step 3: The copper board with the layouts printed is washed with a concentrated $FeCl_3 \cdot 4H_2O$ solution to remove all the uncovered copper. Step 4: The board is cut out with the appropriate tools to obtain the electrodes and these are cleaned with acetone. Step 5: To define the electrode geometry, nail varnish is used as insulator and the *ex situ* bismuth film is electro-deposited. Step 6: The proposed minisensor is used for analytical applications.

# Substituição do eletrodo de mercúrio pelo eletrodo de filme de bismuto

Analytical Methods

RSC Publishing

PAPER

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## Inexpensive and disposable copper mini-sensor modified with bismuth for lead and cadmium determination using square-wave anodic stripping voltammetry

Cite this: *Anal. Methods*, 2013, 5, 202

Luiz Carlos S. Figueiredo-Filho,[a] Bruno C. Janegitz,[a] Orlando Fatibelilo-Filho,*[a] Luiz Humberto Marcolino-Junior[b] and Craig E. Banks*[c]

The fabrication and evaluation of a disposable copper mini-sensor *ex situ* modified bismuth film for the sensing of lead(II) and cadmium(II) *via* square-wave anodic stripping voltammetry (SWASV) is presented. The sensor was *ex situ* modified with a bismuth film *via* electro-deposition through applying a potential of −0.18 V *vs.* Ag/AgCl (3.0 mol $L^{-1}$ KCl) for 200 seconds in a 0.02 mol $L^{-1}$ $Bi(NO_3)_3$, 0.15 mol $L^{-1}$ sodium citrate in 1.5 mol $L^{-1}$ HCl solution. Under the optimal experimental conditions, the voltammetric response was linearly dependent on the analyte concentrations over the range from $1.3 \times 10^{-6}$ to $1.3 \times 10^{-5}$ mol $L^{-1}$ and from $9.9 \times 10^{-7}$ to $1.2 \times 10^{-5}$ mol $L^{-1}$ with a limit of detection of $8.3 \times 10^{-7}$ mol $L^{-1}$ and $5.3 \times 10^{-7}$ mol $L^{-1}$ for Pb(II) and Cd(II), respectively. The determination of both toxic metallic species was carried out in natural waters using the sensor obtaining results which are in close agreement with those obtained using Flame Atomic Absorption Spectrometry at a 95% confidence level.

Received 21st September 2012
Accepted 24th October 2012

DOI: 10.1039/c2ay26078f

www.rsc.org/methods

# Substituição do eletrodo de mercúrio pelo eletrodo de filme de bismuto

Talanta 126 (2014) 82–90

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta


## A thermostated electrochemical flow cell with a coupled bismuth film electrode for square-wave anodic stripping voltammetric determination of cadmium(II) and lead(II) in natural, wastewater and tap water samples

Vagner B. dos Santos [a], Elson L. Fava [a], Newton S. de Miranda Curi [b], Ronaldo C. Faria [a], Orlando Fatibello-Filho [a,*]

[a] *Departamento de Química, Centro de Ciências Exatas e de Tecnologia, Universidade Federal de São Carlos, Caixa Postal 676, CEP 13560-970 São Carlos, SP, Brazil*

[b] *FAC, Cândido de Arruda Botelho, São Carlos, SP, Brazil*

# Substituição do eletrodo de mercúrio pelo eletrodo de diamante dopado com boro

Analytical Methods

ROYAL SOCIETY OF CHEMISTRY

PAPER

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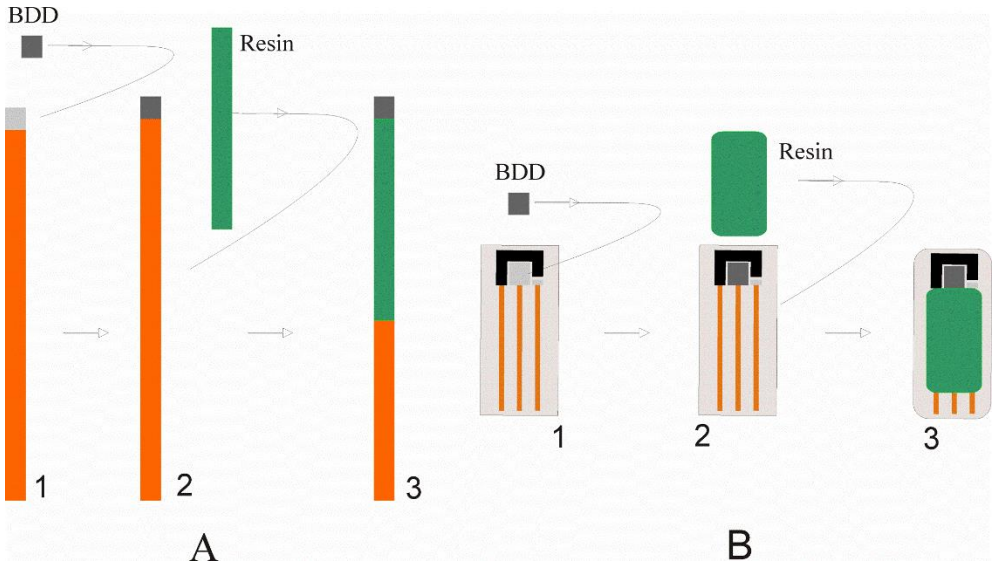
Cite this: *Anal. Methods*, 2014, 6, 8526

## A versatile and robust electrochemical flow cell with a boron-doped diamond electrode for simultaneous determination of $Zn^{2+}$ and $Pb^{2+}$ ions in water samples†

Vagner Bezerra dos Santos,*[a] Elson Luiz Fava,[a] Osmundo Dantas Pessoa-Neto,[a] Silmara Rossana Bianchi,[b] Ronaldo Censi Faria[a] and Orlando Fatibello-Filho[a]




**Steps to produce the conventional electrode of the BDD coupled to a copper rod (A) and to produce the BDD embedded in the SPE (B).**

Methods

PAPER

View Article Online
View Journal

Cite this: DOI: 10.1039/c5ay00012b

# An electrochemical analyzer for *in situ* flow determination of Pb(II) and Cd(II) in lake water with on-line data transmission and a global positioning system†

Vagner Bezerra dos Santos,*[a] Elson Luiz Fava,[a] Newton Sá de Miranda Curi,[b] Ronaldo Censi Faria,[a] Thiago Brito Guerreiro[a] and Orlando Fatibello-Filho[a]


Fig. 3 The PG004 (A), 1: GPS, 2: touchscreen, 3: panel with an optional keyboard, keys, and command bottoms, 4: flow module, and 5: solar boards. The internal view of the PG004 (B), 1: 12 V batteries, 2: actuator of the flow system, 3: galvanostat board, 4: potentiostat board, 5: thermostated control, 6: fan, 7: USB hub, 8: CPU, and 9: micro-controlled board to control the batteries. The Wi-Fi and Bluetooth board is inserted in the CPU. Flow module, with 1: EFC, 2: SVs, 3: solutions compartment, and 4: μPs.

# *In situ determination - PG004*


2

**(2) 21°59.1203'S; 47°52.9038'W;**

3

**(3) 21°59.1763'S; 47°52.8790'W**

**(1) 21°59.1655'S; 47°52.9343'W;**

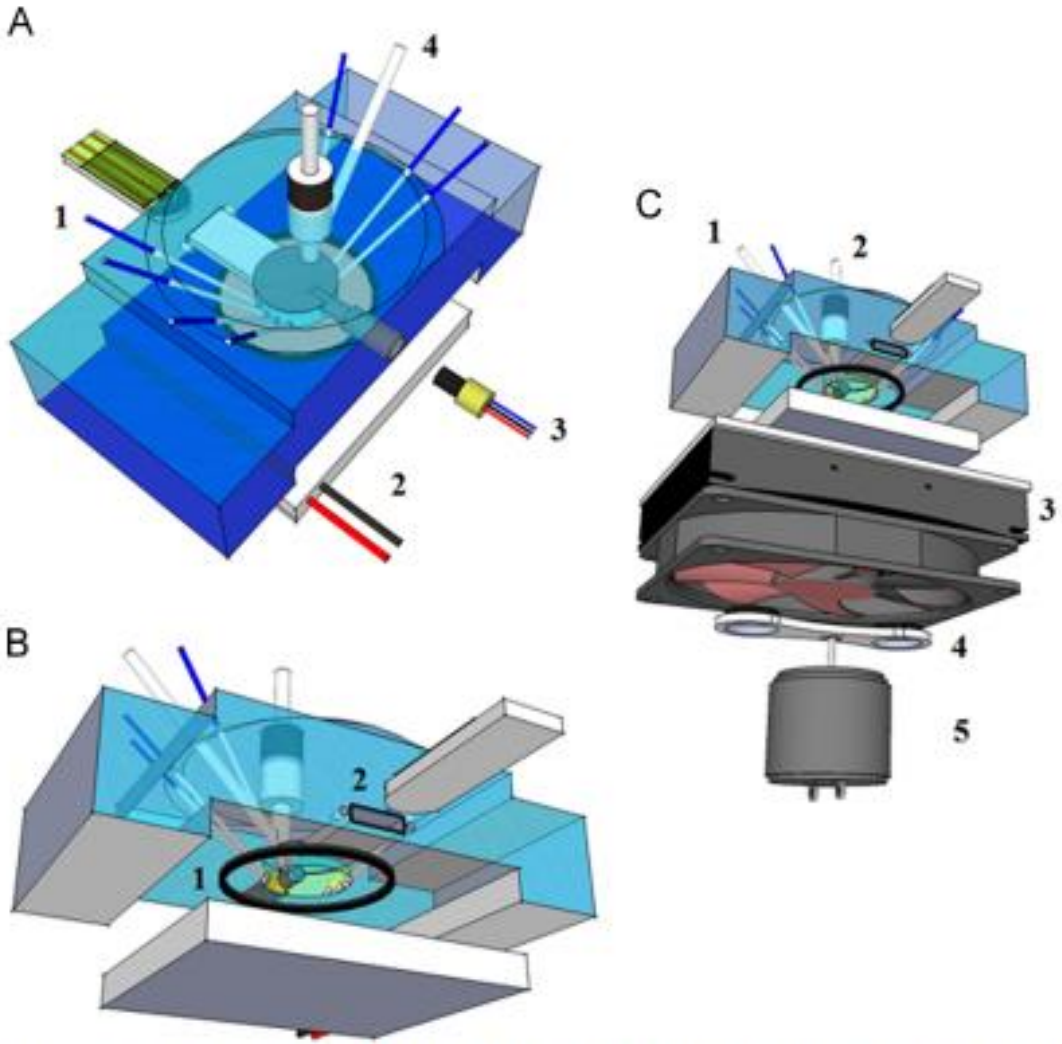




**Fig. 2.** Thermostated EFC design. PTFE tube with 0.8 mm i.d. coupled to the EFC used for inlet flow (1), TEC-Peltier (2), temperature sensor (3) and the blank Tygon® tube or an external CE with 3.0 mm i.d. used as the outlet flow (4) (A). Inside view from the bottom up of the EFC with details of the O-rings used to seal the base of the EFC (1) and the SPE (2) (B). Tygon® tube or an external CE (1), an external RE (Ag/AgCl (3.0 mol $L^{-1}$ KCl)) (2), a TEC-Peltier with a heat dissipation system composed of an aluminum block and a fan (3), a pair of magnets (4) coupled to a motor axis (5) for mechanical agitation (C).

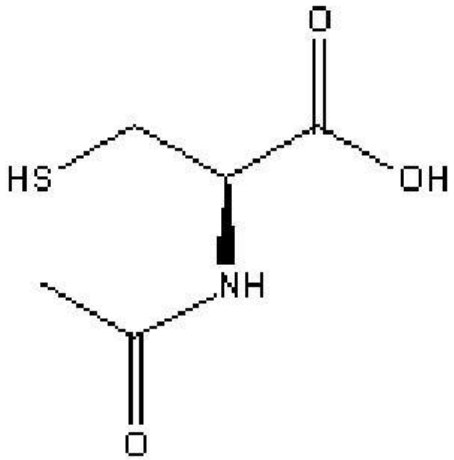
# 3. Reciclo e/ou recuperação e reagentes

Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Pharmaceutical and Biomedical Analysis 37 (2005) 771–775
JOURNAL OF PHARMACEUTICAL AND BIOMEDICAL ANALYSIS
www.elsevier.com/locate/jpba

# Generation and destruction of unstable reagent in flow injection system: determination of acetylcysteine in pharmaceutical formulations using bromine as reagent

Willian Toito Suarez, Heberth Juliano Vieira, Orlando Fatibello-Filho*

*Centro de Ciências Exatas e de Tecnologia, Departamento de Química, Universidade Federal de São Carlos, P.O. Box 676, CEP 13.560–970 São Carlos, SP, Brazil*



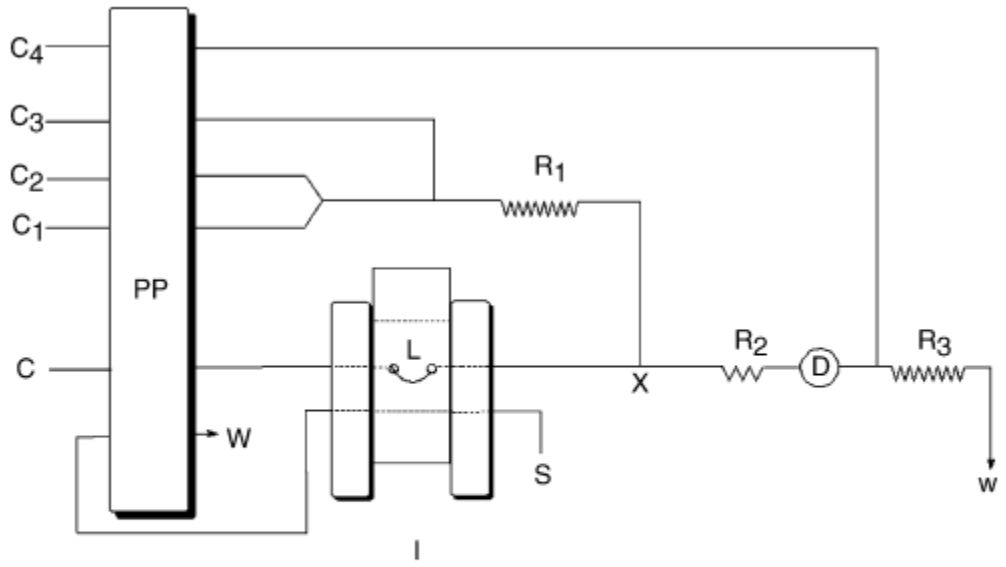
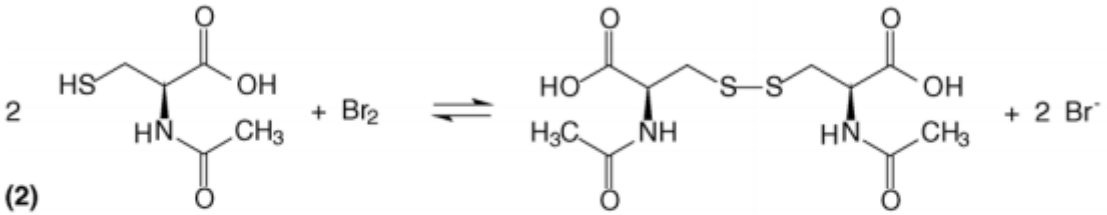


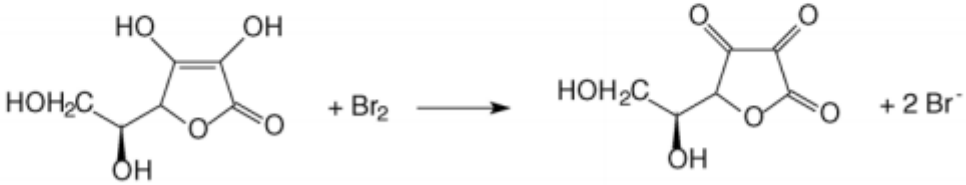

Fig. 1. Schematic diagram of flow injection system for acetylcysteine determination. (PP) peristaltic pump; (C) deionized water; ($C_1$) 0.22 mol/l sodium bromide solution; ($C_2$) 0.24 mol/l hydrochloric acid solution; ($C_3$) $1.25 \times 10^{-2}$ mol/l bromate solution; ($C_4$) 5% (w/v) ascorbic acid solution; (I) injector-commutator; (L) sample volume (250 μl); (S) reference or sample solutions; ($R_1$), ($R_2$) and ($R_3$) 140, 60 and 60 cm helicoidal coils length, respectively; (D) detector and (W) waste.

**(1)** $BrO_3^- + 5\,Br^- \rightleftharpoons 3\,Br_2 + 3\,H_2O$

**(2)**




**(3)**




ascorbic acid

dehydroascorbic acid

# Obrigado

**Is the chemistry I am doing the  most benign I can make it?**

**(P. T. Anastas (1999)**

**Que a geração futura possa desfrutar de um mundo melhor**

**Melissa**

**Lua**


OBRIGADO PESSOAL
OBRIGADO PESSOAL
OBRIGADO PESSOAL
OBRIGADO PESSOAL


FIGURA 6.16. Figura descritiva do PG004 (A), com 1: GPS, 2: painel frontal, 3: módulo de fluxo, 4: placas solares. Detalhes internos do PG004 (B), com 1: baterias de 12 V com 1,3 A $h^{-1}$, 2: interface do módulo em fluxo, 3: placa do galvanostato, 4: placa do potenciostato, 5: placa microcontroladora de controle térmico, 6: *cooler*, 7: placa Wi-Fi® e Bluetooth®, 8: USB HUB, 9: CPU. Módulo em fluxo, com 1: EFC2, 2: $V_S$, 3: compartimento das soluções, 4: µPs.

# Termos empregados nos processos químicos ambientalmente amigáveis ou sustentáveis

**Economia atômica (de átomos)***

**Ecoquímica**

**Química ambientalmente amigável**

**Química ambientalmente benigna**

**Química ambientalmente sustentável**

**Química ecológica**

**Química limpa**

**Química verde**


*B. M. Trost, *Science*, 254, 1471 (1991)

# The case for the use of unrefined natural reagents in analytical chemistry—A green chemical perspective

**Kate Grudpan,[a] Supaporn Kradtap Hartwell,[a] Somchai Lapanantnoppakhun[a] and Ian McKelvie[b]**

***Received 19th April 2010, Accepted 16th July 2010***
**DOI: 10.1039/c0ay00253d**

An important part of the Green Chemistry philosophy is the need to develop and adopt green analytical techniques and procedures. These include the reduction of reagent and solvent usage, the minimization of solid, liquid and gaseous materials produced by analytical processes, the replacement of reagents and solvents of high occupational or environmental toxicity with much more innocuous materials, and the reduction of energy use in analytical processes. One aspect that has received little attention in this context is the use of unrefined natural reagents derived from plant and animal tissues or microbial cells. Crude plant extracts may contain chemical compounds that enable their use as indicators in acid–base or redox titrations, or as chromogenic or fluorogenic agents. Enzymes extracted from plants may be used directly in soluble form, or incorporated in biosensors or solid phase reactors, for analytical measurements, or as a means of removing interferences or performing speciation studies. The use of natural reagents in conjunction with a flow injection system can confer a number of advantages. The enhanced kinetic control that flow analysis offers may assist in avoiding undesirable side reactions that would otherwise occur using unpurified reagents. The lifetime of natural reagents may be prolonged when used in a flow analysis system because their exposure to light or air can be controlled. Any changes in response that do occur may be readily corrected by regular standard checking and recalibration. This article reviews the use of natural reagents with an emphasis on flow-based analytical systems, and makes the case for further research in this latent area of green analytical chemistry.

**K. Grudpan *et al.*; *Anal. Methods*,2, 1651 (2010).**

Trends in Analytical Chemistry, Vol. 45, 2013



# Greening sample preparation in inorganic analysis

Diogo L. Rocha, Alex D. Batista, Fábio R.P. Rocha, George L. Donati, Joaquim A. Nóbrega

**Sample preparation aims to convert analytes to a more suitably detectable form, to separate them from the sample matrix or to concentrate species for trace analysis. Classical procedures often yield large amounts of toxic waste, but environment-friendly alternatives can be implemented without impairing the analytical performance. Green methods are also safer, and reduce costs and risks of sample contamination. This overview focuses on application of these strategies to inorganic analysis.**

# Substituição do eletrodo de mercúrio pelo eletrodo de amálgama

*Quim. Nova*, Vol. 34, No. 3, 487-496, 2011

Revisão

## UTILIZAÇÃO DE ELETRODOS SÓLIDOS DE AMÁLGAMA PARA A DETERMINAÇÃO ANALÍTICA DE COMPOSTOS ORGÂNICOS E INORGÂNICOS

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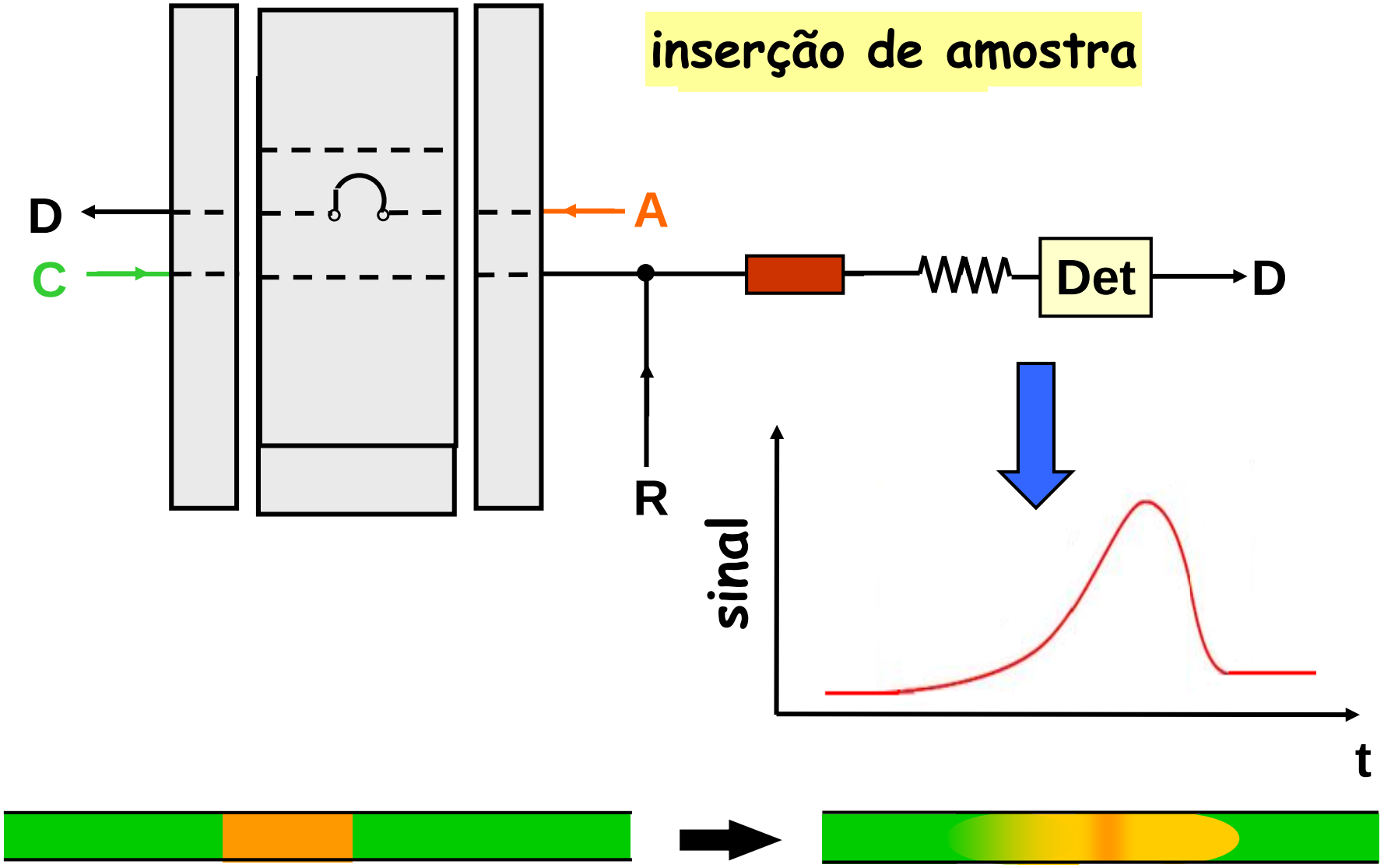
Recebido em 17/4/10; aceito em 28/9/10; publicado na web em 26/1/11

UTILIZATION OF THE SOLID AMALGAM ELECTRODES IN THE ANALYTICAL DETERMINATION OF ORGANIC AND INORGANIC COMPOUNDS. This review reports the use of solid amalgam electrodes in the electroanalytical determination of organic and inorganic compounds. The different types of amalgam electrodes are presented, and attention is paid to solid amalgam electrode, and here is presented details about the pre-treatment for activation and renovation and also possible modifications in its surface. The wide potential range, higher signal-to-noise ratio, mechanical stability enabling their application in flowing systems, and principally their resistance toward passivation, indicate that the solid amalgam electrodes are environmentally friendly alternatives to mercury electrodes, without loss in the sensitivity and reproducibility in voltammetric responses.

# Sistemas de Análise por Injeção em Fluxo



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Talanta 57 (2002) 135–143

Talanta

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# Flow injection spectrophotometric determination of isoproterenol using an avocado (*Persea americana*) crude extract immobilized on controlled-pore silica reactor

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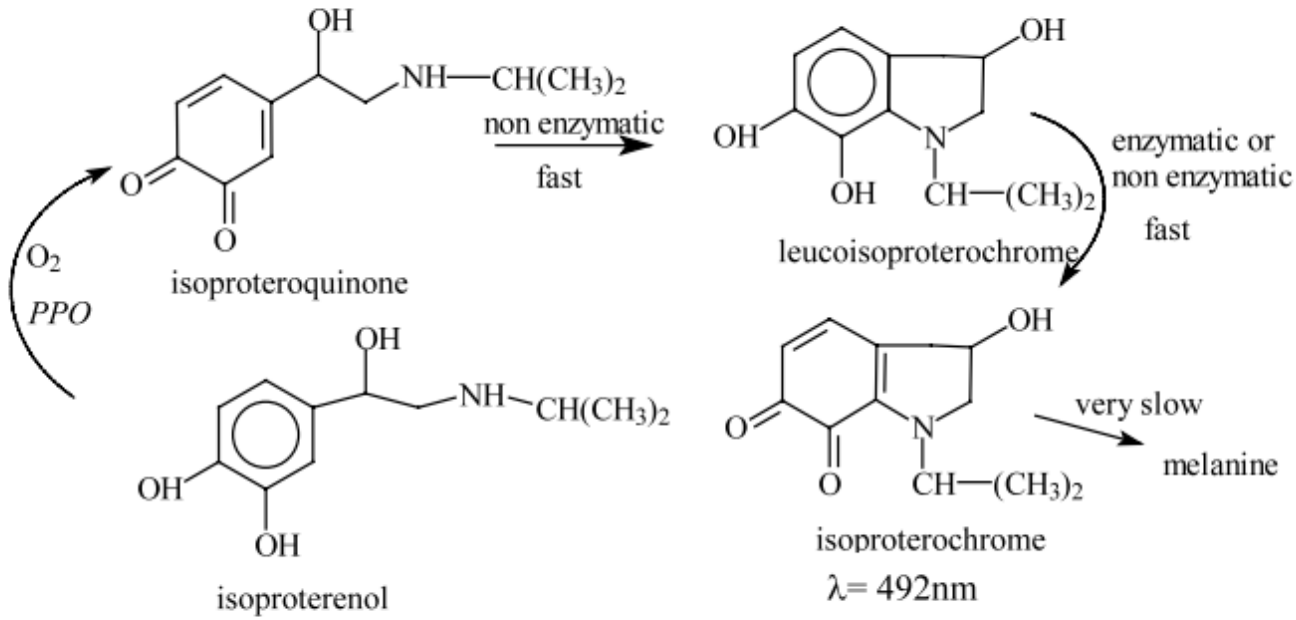


Fig. 3. Reaction steps of the catalytic oxidation of isoproterenol by PPO of avocado crude extract immobilized on the CPS.

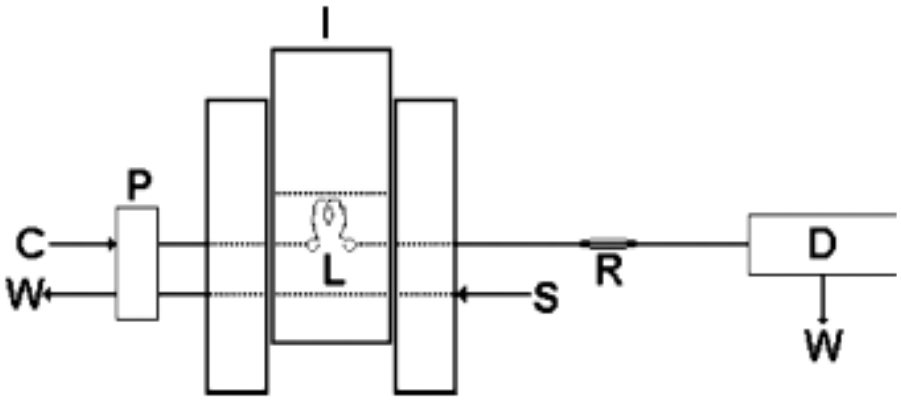


Fig. 1. Schematic diagram of the flow injection system used for the spectrophotometric determination of isoproterenol. The central bar of the manual injector–commutator (I) shows the injection position after commutation. P, peristaltic pump; S, sample or reference solutions; L, sample loop (375 μl); C, carrier solution (0.1 mol $l^{-1}$ phosphate buffer solution at flow rate of 2.1 ml $min^{-1}$); R, enzymatic reactor (3.0 mm i.d. and 30 mm long); D, spectrophotometric detector at 492 nm and W, waste, at 25 °C.

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Journal of Pharmaceutical and Biomedical Analysis 37 (2005) 771–775
JOURNAL OF PHARMACEUTICAL AND BIOMEDICAL ANALYSIS
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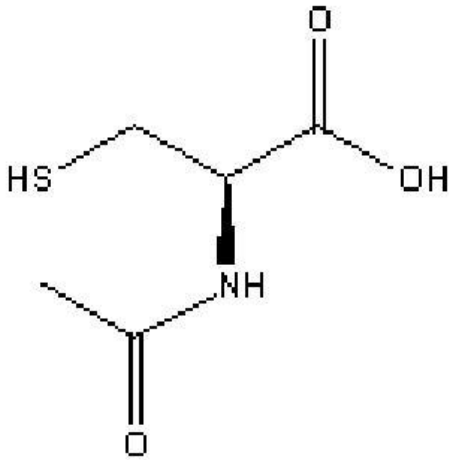
# Generation and destruction of unstable reagent in flow injection system: determination of acetylcysteine in pharmaceutical formulations using bromine as reagent

Willian Toito Suarez, Heberth Juliano Vieira, Orlando Fatibello-Filho*

*Centro de Ciências Exatas e de Tecnologia, Departamento de Química, Universidade Federal de São Carlos, P.O. Box 676, CEP 13.560–970 São Carlos, SP, Brazil*



# Economia de átomos

**Barry M. Trost (1991) ⇒ Quanto dos reagentes empregados termina incorporado nos produtos? Assim, a reação química ideal é aquela em que todos os átomos dos reagentes são incorporados no(s) produto(s), não gerando assim resíduos**

$$\%EA = \frac{massa\ do\ produto}{\sum massas\ reagentes} \times 100$$

B. M. Trost, *Science*, 254, 1471 (1991)

# 3.1 The E Factor

## *3.1.1 History and Development*

As the chemical industry started to witness increased environmental pressures in the early 1990s, Roger Sheldon proposed the environmental (E) factor as a metric for quantifying the amount of waste produced in a chemical process. With waste

**Table 3.1** E factor estimates for different chemical industries based on Sheldon's original findings [1]

| Industry sector | Annual production (tonnes) | E Factor |
|---|---|---|
| Oil refining | $10^6$–$10^8$ | <0.1 |
| Bulk chemicals | $10^4$–$10^6$ | 1–5 |
| Fine chemistry | $10^2$–$10^4$ | 5–50 |
| Pharmaceuticals | $10^1$–$10^3$ | 25–100 |

**Fator E = kg do sub-produtos/kg do produto**

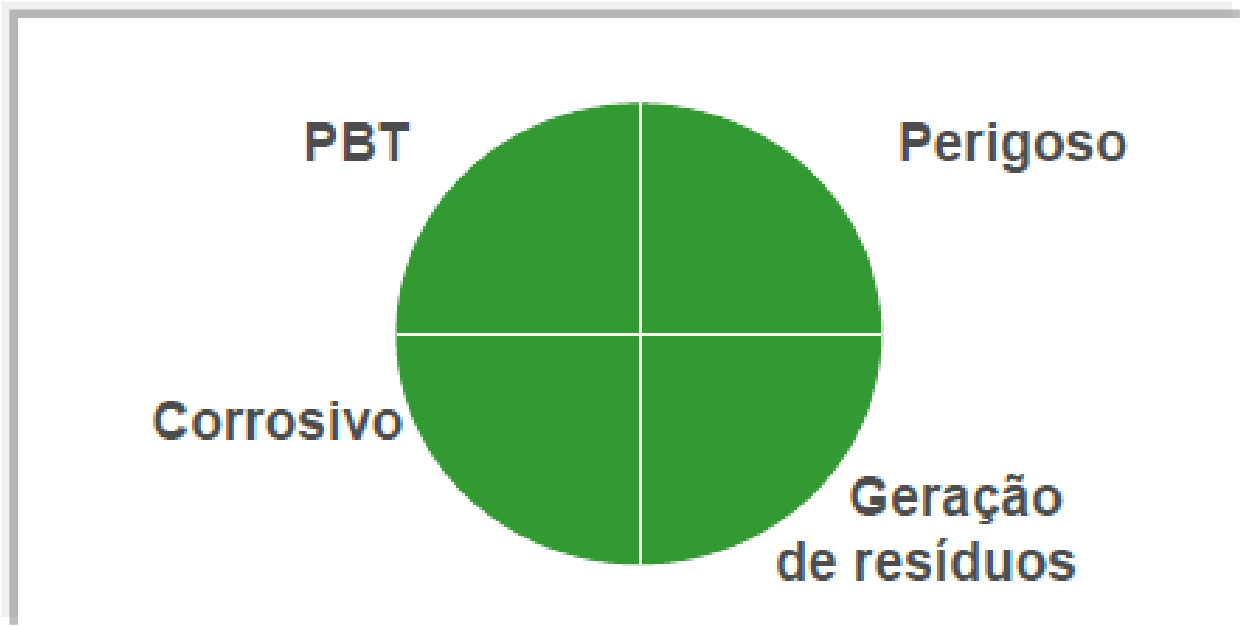



**Figura- Pictograma representativo do perfil verde segundo a National Environmental Methods Index (NEMI)**

**PBT= persistentes, bioacumulativos e tóxicos**

**www.nemi.gov**

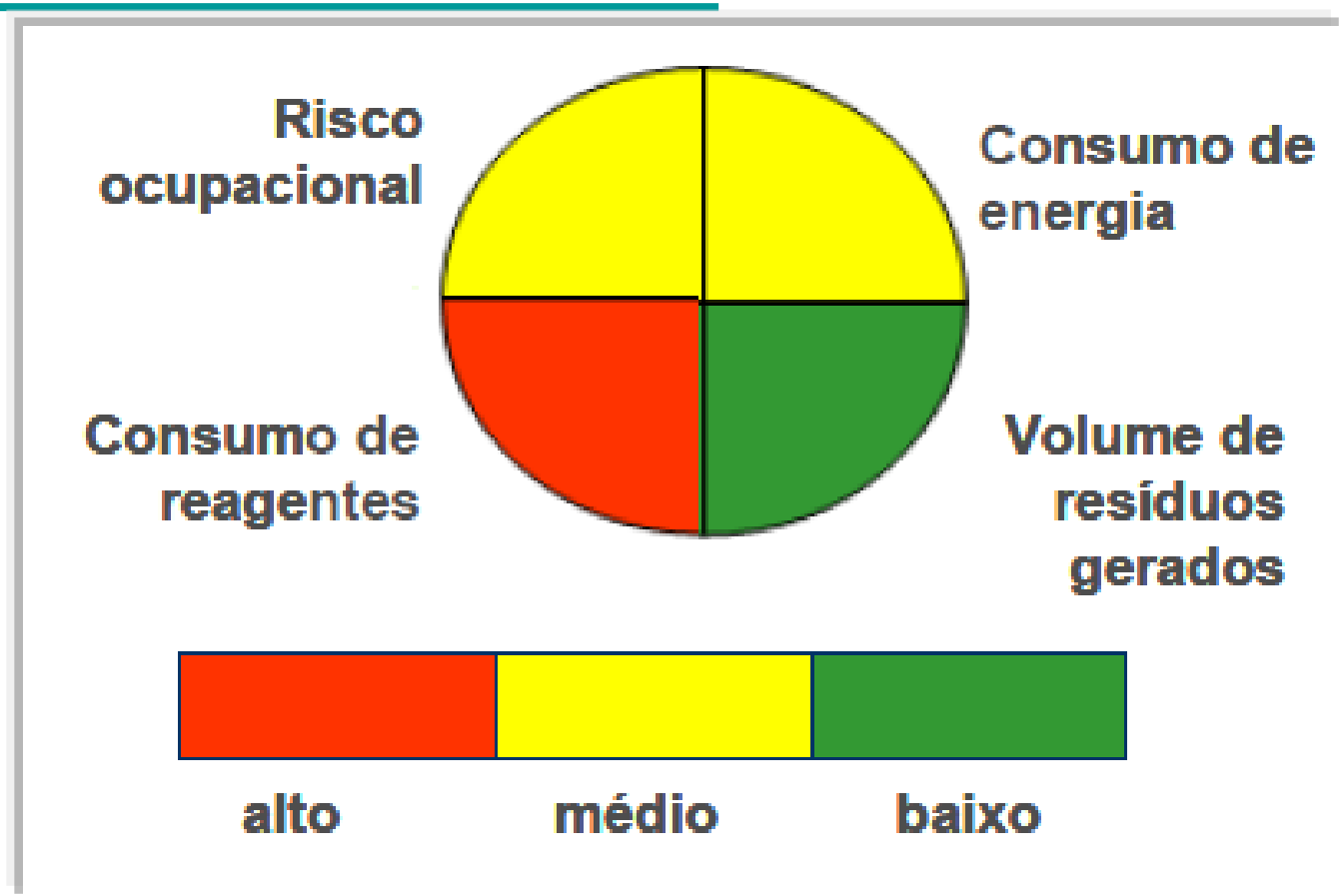



**Fig. Pictograma de 'perfil verde' complementar ao pictograma do *NEMI***

**de la Guardia e Garrigues (2011)**