

2017

1. Svinyarov, I., Bogdanov, M.G., **Ionic liquid-assisted micellar extraction for the quantitative determination of sesquiterpenic acids in *Valeriana officinalis* L. (Caprifoliaceae).** *Separation Science and Technology* (2017) DOI: 10.1080/01496395.2017.1295995

Impact Factor(2015): **1.083**

**Abstract:** A series of hydrophilic ionic liquids (ILs) were investigated as extractants for the quantitative determination of valerenic and acetoxyvalerenic acids in *Valeriana radix*. The influence of different factors, e.g. type of cation and anion, alkyl chain length, concentration, temperature, extraction time and solid-liquid ratio was investigated. The results obtained showed that the extraction yield is strongly dependent on the cation type. The best outcome was obtained with IL capable of forming micelles in aqueous solution, e.g. 1-decyl-3-methylimidazolium chloride. As a result, an alternative protocol for quantification of sesquiterpenic acids in valerian was developed and validated.

2016

2. Svinyarov, I., Keremedchieva, R., Bogdanov, M.G., **Ionic liquid-supported solid-liquid extraction of bioactive alkaloids. IV. New HPLC method for quantitative determination of galantamine in *Leucojum aestivum* L. (Amaryllidaceae).** *Separation Science and Technology* 51 (2016) 2691–2699.

Impact Factor (2015): **1.083**

**Abstract:** A series of aqueous solutions of hydrophilic ionic liquids (ILs) were examined as extractive systems in the recovery of galantamine from plant material of *Leucojum aestivum* L. The extractions were carried out both at room temperature and conventional heating conditions. The influence of various factors on the extraction efficiency was comprehensively studied. The conducted comparative analysis with conventional methods showed higher extraction ability of the ILs-based extractive systems for significantly reduced extraction times. The results obtained were implemented in a new HPLC method for quantification of galantamine, avoiding the use of harmful organic solvents during the sample preparation step.

2015

3. Miliovsky, M., Svinyarov, I., Prokopova, E., Batovska, D., Stoyanov, S., Bogdanov, M.G., **Synthesis and antioxidant activity of polyhydroxylated *trans*-restricted 2-arylcinnamic acids.** *Molecules* 20 (2015) 2555–2575.

Impact Factor (2014): **2.416**

**Abstract:** A series of sixteen polyhydroxylated *trans*-restricted 2-arylcinnamic acid analogues **3a–p** were synthesized through a one-pot reaction between homophthalic anhydrides and various aromatic aldehydes, followed by treatment with BBr<sub>3</sub>. The structure of the newly synthesized compounds was confirmed by spectroscopic

methods and the configuration around the double bond was unequivocally estimated by means of gated decoupling  $^{13}\text{C}$ -NMR spectra. It was shown that the *trans*-cinnamic acid fragment incorporated into the target compounds' structure ensures the *cis*-configuration of the stilbene backbone and prevents further isomerization along the carbon–carbon double bond. The antioxidant activity of compounds **3a–p** was measured against 1,1-diphenyl-2-picrylhydrazyl (DPPH $^{\bullet}$ ), hydroxyl (OH $^{\bullet}$ ) and superoxide (O $_2^{\bullet-}$ ) radicals. The results obtained showed that the tested compounds possess higher activities than natural antioxidants such as protocatechuic acid, caffeic acid and gallic acid. Moreover, it was shown that a combination of two different and independently acting fragments of well-known pharmacological profiles into one covalently bonded hybrid molecule evoke a synergistic effect resulting in higher than expected activity. To rationalize the apparent antioxidant activity and to establish the mechanism of action, a SAR analysis and DFT quantum chemical computations were also performed.

4. Bogdanov, M.G., Keremedchieva, R., Svinyarov, I., **Ionic liquid-supported solid-liquid extraction of bioactive alkaloids. III. Ionic liquid regeneration and glaucine recovery from ionic liquid-aqueous crude extract of *Glaucium flavum* Cr. (Papaveraceae). *Separation and Purification Technology* 115 (2015) 13–19.**

Impact Factor (2014): **3.091**

**Abstract:** In continuation of a research project aiming at introducing ionic liquids (ILs) as an alternative to the widely applied for the recovery of natural products of industrial interest conventional molecular solvents, we developed a concise procedure for isolation of the biologically active alkaloid S-(+)-glaucine from IL-based aqueous crude extract. To this end, a comparative study of the behavior of 1 M [C $_4$ C $_1$ im][Ace]-aqueous solution and methanol in a series of consecutive extractions with the same extractant was conducted. The results obtained proved the better performance of the IL-based system in the solid–liquid extraction step, since the latter showed constantly higher extraction efficiency (ca. 35% enhanced) compared to methanol. The above procedure allows glaucine accumulation from at least ten successive extractions, while simultaneously reduces the total solid–liquid ratio from 1:40 to 1:7.2, without loss of efficiency. Furthermore, the loss of IL into the matrix pores after extraction was also considered, suggesting the need for IL recycling by posttreatment of the residual biomass. To recover glaucine from the crude IL-based aqueous extract, a series of non-miscible with water molecular solvents were tested. As a result, optimal conditions for quantitative extraction into chloroform were found, from which, after solvent removal and subsequent crystallization from ethanol, the target compound was isolated as a hydrobromide salt, the latter being the marketed form of glaucine.

5. Keremedchieva, R., Svinyarov I., Bogdanov, MG., **Ionic liquid-based aqueous biphasic systems — a facile approach for ionic liquid regeneration from crude plant extracts.** *Processes* 3 (2015) 769-778.

Impact Factor: **NA**

**Abstract:** The possibility for simultaneous controlled partition of the biologically active alkaloid S-(+)-glaucine, presented in a crude ionic liquid-aqueous plant extract, and separation of the ionic liquid (IL) employed by means of ionic liquid-based aqueous biphasic systems (IL-ABS) was investigated in this study. The ABS were formed by the addition of inorganic salts with distinct water-structuring properties such as Na<sub>2</sub>CO<sub>3</sub>, MgSO<sub>4</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and NaH<sub>2</sub>PO<sub>4</sub> to an enriched of glaucine extract of *Glaucium flavum* Cr. (Papaveraceae). The influence of the salt type and concentration on the phase-forming ability, as well as the pH value on the partition of glaucine between the two phases formed, was comprehensively studied. It was found that the target alkaloid is predominantly transferred into the IL-rich phase, regardless the influential factors. The results obtained were further used as a platform for the development of an improved extractive procedure, ensuring simultaneous glaucine recovery, IL recycling and water removal in a single technological step. Thus, based on the formation of a three-phase system consisting of butyl acetate, IL-rich phase, and salt-rich phase, nearly quantitative recovery of glaucine (>99%), IL recycling (ca. 90%), and water removal (ca. 85%) were achieved by salt-induced separation of the crude plant extract.

6. Живкова, С., Тоновa, С., Свиняров, И., Богданов, М.Г., **Ре-екстракция на кумарини от разтвори на йонни течности чрез разпределение във водни двуфазни системи.** *Scientific works* LXII (2015) 591–596.

Impact Factor: **NA**

**Abstract:** Coumarins are natural compounds widely spread in plant materials, notably in high concentration in the tonka beans, vanilla grass, sweet woodruff, mullein, lavender, sweet grass, strawberries, apricots, cherries, cassia cinnamon, sweet-clover, which are recognized as valuable additives in perfumes and cosmetics industries. Coumarins are used as aroma enhancers in pipe tobaccos and certain alcoholic drinks. Their extraction from the vegetable matrix could be achieved by using environmentally benign “green” solvents, ionic liquids (ILs), instead of polluting volatile organic ones, thus attaining both ecology safety and better extraction efficiency. In this study, a series of four natural coumarins, namely 4-hydroxycoumarin, 7-hydroxy-4-methylcoumarin, 7-methoxy-4-methylcoumarin, 6,7-dihydroxy-4-methylcoumarin, were dissolved in aqueous solution of a common hydrophilic IL, 1-butyl-3-methylimidazolium bromide ([C<sub>4</sub>C<sub>1</sub>im]Br). Based on such a model source, we studied the possibility for their selective back extraction by means of a liquid-liquid demixing technique. Aqueous biphasic systems (ABS) were formed by addition of various kosmotropic (inorganic) salts into the model source and the partitioning of different coumarin representatives were measured in both salt and IL-rich phases. The

dependence of individual coumarin recovery on the system pH was studied and the optimal conditions were evaluated. The composed "IL/salt" ABS were also studied for partitioning of coumarins dissolved in ethanol solutions.

7. Svinyarov, I., Bogdanov, M.G., **3-(3,4-Dihydroxyphenyl)-8-hydroxy-2H-chromen-2-one**. *Molbank* (2015) M870.

SJR: **0.129**

**Abstract:** We report a novel protocol for the synthesis of 3-(3,4-dihydroxyphenyl)-8-hydroxy-2H-chromen-2-one via demethylation/delactonization/elimination/lactonization/decarboxylation domino reaction sequence of diastereomeric mixture of *cis*- and *trans*-3-(2,3-dimethoxyphenyl)-6,7-dimethoxy-1-oxoisochroman-4-carboxylic acids in boiling HBr/AcOH. The structure of the target compound was established for the first time by means of spectral methods such as  $^1\text{H}$ -,  $^{13}\text{C}$ -, DEPT-135-NMR, IR and HRMS.

8. Свиняров, И.В., **Синтез и биологична активност на полизаместени бензопиранони**. *Автореферат на дисертация*, София, 2015.

**Резюме:** Дисертацията има за цел синтез и изследване на биологичната активност на серии от нови полизаместени бензопиранони (3,4-дихидроизокумарини и кумарини) с използването на хомофталови анхидриди и алдехиди като изходни съединения. Изследвани са факторите влияещи върху вида и добивите на крайните продукти от бензопиранонов тип, и са изведени конкретни зависимости по отношение на структурата на изходните реагенти и използваните условия. В хода на работата е наблюдавана неочаквана трансформация до образуването на съединения с кумаринова структура, въз основа на която е разработена нова опрочот процедура за синтез на полихидроксизаместени 3-арилкумарини. Посредством  $^1\text{H}$  ЯМР и X-Ray са изследвани диастереоселективността на реакциите и предпочетените конформация в разтвор и в твърда фаза за конформационно подвижните 3,4-дихидроизокумаринови производни. За всички синтезирани съединения е изследвана антимикуробната активност спрямо широка гама от бактериални и гъбни щамове, като е установена висока активност на 3,4-дихидроизокумарините съдържащи в структурата си дълга алифатна верига ( $\text{C}_{9-10}$ ) в комбинация с групи повишаващи хидрофилността им. За полихидроксизаместените 3-арилкумарини са изследвани радикалоулавящите и антиоксидантните им свойства. Получените резултати показаха, че синтезираните съединения демонстрират по-висока антиоксидантна активност от използваните като референти природни антиоксиданти.

2014

9. Tonova, K., Svinyarov, I., Bogdanov, M.G., **Hydrophobic 3-alkyl-1-methylimidazolium saccharinates as extractants for L-lactic acid recovery.** *Separation and Purification Technology* 125 (2014) 239–246.

Impact Factor (2013): **3.065**

**Abstract:** *N*-octyl- and *N*-decylsubstituted *N*-methylimidazolium saccharinates ( $[C_{8/10}C_1im][Sac]$ ) were synthesized and found to form biphasic systems after saturation with water. Therefore, they could be considered as hydrophobic ionic liquids (ILs), which suggests their potential application as extraction media. The performance of these ILs as extractants of L-lactic acid (LA) from aqueous solutions was evaluated. The pH dependence and the influence of additives such as kosmotropic salts or alcohols on the LA extraction yield (*EY*%) were comprehensively studied. As a result, *EY* = 90% was achieved with  $[C_8C_1im][Sac]$  in two extraction steps. Furthermore, based on the observed effects of pH and kosmotropic salt, successful LA re-extraction (95%) from the IL-rich phase was attained by means of  $K_2HPO_4$ -stripping solution. The latter represents the highest re-extraction efficiency for LA/IL system described so far in the literature. Based on the results obtained, a plausible mechanism of LA extraction, which considers the secondary structure of the water saturated long side chain imidazolium saccharinates, is proposed. As a result, the IL extraction capacity was attributed to the formation of polar channels, separated by non-polar domains, which channels are capable of swelling and liberating water and other hydrophilic solutes.

10. Svinyarov, I., Bogdanov, M.G., **One-pot synthesis and radical scavenging activity of novel polyhydroxylated 3-aryl coumarins.** *European Journal of Medicinal Chemistry*, 78 (2014) 198–206.

Impact Factor (2013): **3.432**

**Abstract:** An unexpected domino rearrangement brought about the development of a novel one-pot procedure for synthesis of coumarins. This protocol allowed the gram-scale synthesis of a variety of polyhydroxylated derivatives **3a–p**, from readily available starting materials at a low cost. Based on two proven intermediates, a probable mechanism consisting of boron tribromide induced demethylation/lactone ring opening/elimination/isomerization/lactone ring closure reaction sequence of in situ formed 3-aryl-3,4-dihydroisocoumarin-4-carboxylic acids was deduced. Compared to the common methods, used for the synthesis of coumarins, the proposed herein possesses great advantages, such as mild conditions, good yields for short reaction time, simple work-up procedure and easy isolation of the final products. The structure of the newly synthesized compounds **3a–p** was established by spectroscopic methods ( $^1H$  NMR,  $^{13}C$  NMR, IR, MS and HRMS) and their radical scavenging activity was evaluated *in vitro* against 1,1-diphenyl-2-picrylhydrazyl free radical (DPPH $^{\bullet}$ ). The results obtained show that compounds **3a–p** possess higher radical scavenging activity

( $3.16 \leq SC_{50} [\mu M] \leq 6.82$ ) than well-known antioxidants such as trolox, protocatechuic acid, caffeic acid and gallic acid ( $SC_{50} [\mu M] = 9.34, 8.83, 9.48, 5.33$ , respectively), which is a precondition for promising antioxidant activity of these compounds to be expected.

### 2013

11. Miliovsky, M., Svinyarov, I., Mitrev, Y., Evstatieva, Y., Nikolova, D., Chochkova, M., Bogdanov, M.G. **A novel one-pot synthesis and preliminary biological activity evaluation of *cis*-restricted polyhydroxy stilbenes incorporating protocatechuic acid and cinnamic acid Fragments.** *European Journal of Medicinal Chemistry* 66 (2013) 185–192.

Impact Factor (2012): **3.449**

**Abstract:** A series of new stilbenes **4a–e**, **5** were synthesized through a novel one-pot Perkin-like reaction between 6,7-dimethoxyhomophthalic anhydride and aromatic aldehydes, followed by treatment with  $BBr_3$ . This synthesis is straightforward and allows polyhydroxylated *cis*-stilbenes gathering two well-known pharmacophoric fragments to be obtained in good yields and for short reaction times. The structure of the newly synthesized compounds was established by spectroscopic methods ( $^1H$  NMR,  $^{13}C$  NMR, IR and HRMS) and the double bond configuration was unequivocally elucidated by means of gated decoupling  $^{13}C$  NMR spectra and 2D NOESY experiments. Preliminary differentiating screening of their radical scavenging, antibacterial, anti-fungal and tyrosinase inhibitory activity was further performed. The results obtained suggest that the tested compounds possess a triple biological action as potent radical scavengers, antifungal agents and tyrosinase inhibitors in micromolar concentration. The most promising bioactive compound amongst the others was **4a**, acting as excellent radical scavenger against DPPH radical ( $IC_{50} \leq 10 \mu M$ ), antifungal agent suppressing the growth of *Fusarium graminearum* (89% inhibition at  $0.17 \mu mol/mL$ ), and tyrosinase inhibitor showing higher activity than hydroquinone at  $23 \mu M$ .

12. Bogdanov, M.G., Svinyarov, I., **Ionic liquid-supported solid–liquid extraction of bioactive alkaloids. II. Kinetics, modeling and mechanism of glaucine extraction from *Glaucium flavum* Cr. (Papaveraceae).** *Separation and Purification Technology* 103 (2013) 279–288.

Impact Factor (2012): **2.894**

**Abstract:** The temperature dependence on the global kinetics of both methanol and 1-butyl-3-methylimidazolium acesulfamate ( $[C_4mim][Ace]$ )-supported extraction of the bioactive alkaloid *S*-(+)-glaucine from plant material of *Glaucium flavum* Crantz (Papaveraceae) was measured and a comparative analysis in respect to the extractant type was performed. The experimental data was fitted with high coefficients of determination by means of an empirical model showing that the extraction process in both cases follows second-order kinetics. The concentration at saturation,  $C_s$ , extraction rate constants,  $k_2$ , initial rates of extraction,  $h$ , and activation energies of

extraction,  $E_d$ , were then calculated and discussed. The obtained results give a considerable advantage to the ionic liquid-supported (IL-supported) extraction procedure due to the faster and quantitative glaucine transfer, regardless the temperature. A plausible extraction mechanism scenario, describing the whole system in terms of solute–solvent, solute–matrix and matrix–solvent interactions at every stage of the extraction process was proposed. As a result, the apparent kinetics and yield of the IL-supported extraction were attributed to the cell wall modification by Hbonding interactions between both ions of  $[C_4mim][Ace]$  and cellulose, resulting in the plant tissues disruption. The latter was further unequivocally confirmed by means of scanning electron microscopy. The kinetics parameters obtained were then implemented into a model for the prediction of glaucine amount extracted in 1 M  $[C_4mim][Ace]$  water solution at any temperature and time with good predictive ability.

13. Свиняров, И., Тодорова, Н., Богданов, М., **Синтез и биологична активност на 3-алкил и 3-алкенилзаместени изокумаринови производни.** *Българско списание за химия 2* (2013) 93–103.

Impact Factor: **NA**

**Резюме:** Настоящата статия представлява кратък обзор на методи за синтез на 3-алкил и 3-алкинилзаместени изокумарини и 3,4-дихидроизокумарини. Последователно са разгледани различни подходи за формиране на изокумариновата пръстенна систем посредством метал катализирани реакции, орто-лителиране, лителиране в бензилово положение, кондензация между хомофталова киселина и хлориди, окисление на индан-1-они и др. Отделен акцент е поставен върху проявяваната от някои природни и синтетични представители биологична активност.

## 2012

14. Bogdanov, M.G., Svinyarov, I., Keremedchieva, R., Sidjimov, A., **Ionic liquid-supported solid–liquid extraction of bioactive alkaloids. I. New HPLC method for quantitative determination of glaucine in *Glaucium flavum* Cr. (Papaveraceae).** *Separation and Purification Technology* 97 (2012) 221–227.

Impact Factor (2011): **2.924**

**Abstract:** A series of hydrophilic 1-alkyl-3-methylimidazolium-based ionic liquids were used as extractants for the effective extraction of S-(+)-glaucine from plant material of *Glaucium flavum* Crantz (Papaveraceae). The extractions were carried out both under ultrasonic and conventional heating conditions and the extraction efficiency was monitored by HPLC. The influence of the anion (chloride, bromide, acesulfamate and saccharinate), alkyl chain length in the imidazolium ion, concentration, extraction time, and plant material/ extractant ratio was investigated. A comparative study between conventional Soxhlet extraction with methanol as a solvent (total extraction) and water solutions of ILs shows the same extraction ability of ILs, but with significantly reduced extraction time. Moreover, the extraction efficiency was shown to be strongly

anion dependent. The results obtained provide a fast and safe method for determination of glaucine in *G. flavum* Cr.

15. Митрев, Я., Свиняров, И., К.-Йотова, М., Богданов, М., **Реакцията между хомофталов анхидрид и имини – 35 години по-късно.** *Българско списание за химия*, 1 (2012) 29–41.

Impact Factor: **NA**

**Резюме:** Цел на настоящият обзор е да се направи кратък преглед на синтетичните възможности, които представя реакцията между хомофталов анхидрид и имини. Предложена за първи път през 1977 година, днес тя представлява утвърден метод за получаване на полизаместени хетероциклени съединения, съдържащи изохинолинов фрагмент в структурата си. В първата част от статията е описан класическият, некатализиран вариант на реакцията, като последователно са разгледани реакционните условия и нейният обег. В следващата част са разгледани различни модификации, като е акцентирано върху селективното получаване на стереоизомери с желана конфигурация. В последната част са илюстрирани конкретни примери за следващи трансформации на получените изохинолини и употребата им като прекурсори в синтеза на природни продукти или съединения с потенциално приложение във фармацевтичната промишленост.

#### 2011

16. Bogdanov, M.G., Svinyarov, I.V., Atanasov, V.N., **Synthesis and mass spectral study of *trans*- and *cis*- ( $\pm$ )-3-phenyl-3,4-dihydroisocoumarin-4-carboxamides.** *Annuaire de l'Universite de Sofia* 102-103 (2011) 145–152.

Impact Factor: **NA**

**Abstract:** A series of *trans*- and *cis*- ( $\pm$ )-3-phenyl-3,4-dihydroisocoumarin-4-carboxamides (**4a-c**) were synthesized and the electron impact (EI) ionization mass spectral fragmentation was described. For all studied compounds, both diastereomers follow the same fragmentation pattern, but differ on the abundance of the base peaks, which shows the opportunity to distinguish 3,4-dihydroisocoumarin diastereomers using mass spectrometry.

#### 2010

17. Bogdanov, M.G., Svinyarov, I., Kunkel, H., Steinle, C., Kantlehner, W., Maas, G., **Empirical polarity parameters for hexaalkylguanidinium-based room-temperature ionic liquids.** *Zeitschrift fur Naturforschung* 65B (2010) 791–797.

Impact Factor (2009): **0.953**

**Abstract:** The polarity of a series of 36 hexaalkylguanidinium-based room-temperature ionic liquids (RTILs), featuring different unbranched alkyl substituents in the cation and eight different anions, has been determined by means of Reichardt's solvatochromic betaine dye;  $E_T(30)$  and the corresponding normalized  $E_T^N$  values are presented. The positively solvatochromic probe 5-dimethylamino-5'-nitro-2,2'-bithiophene was used



to characterize unspecific solvent/solute interactions (effects of dipolarity/polarizability) of ten hexaalkylguanidinium and, for comparison, two 1-alkyl-3-methylimidazolium ionic liquids.

18. Bogdanov, M.G., Petkova, D., Hristeva, S., Svinyarov, I., Kantlehner, W., **New guanidinium-based room-temperature ionic liquids. Substituent and anion effect on density and solubility in water.** *Zeitschrift fur Naturforschung* 65B (2010) 37–48.

Impact Factor (2009): **0.953**

**Abstract:** In order to examine the influence of the alkyl chain length on some physical properties of guanidinium salts, the synthesis of a homologous series of new *N''-n*-alkylsubstituted *N,N*-diethyl-*N',N'*-di-*n*-propyl-*N''-n*-hexyl guanidinium ionic liquids (gILs), containing chloride (Cl), tetrafluoroborate (BF<sub>4</sub>), acesulfamate (Ace), saccharinate (Sac), and tosylate (Tos) as anions, is reported. **C<sub>n</sub>-gILAce**, **C<sub>n</sub>-gILSac**, and **C<sub>n</sub>-gILBF<sub>4</sub>** were obtained by ion exchange reaction of the corresponding hexasubstituted guanidinium chlorides (**C<sub>n</sub>-gCl**, *n* = 3, 4, 6, 8, 10), which were synthesized by a quaternization reaction of the pentaalkyl-substituted guanidine **3** and the corresponding alkylchloride in DMF. The tosylates gILs C<sub>n</sub>-gTos (*n* = 1, 2, 4, 6, 8, 10) were synthesized by alkylation of **3** with the corresponding alkyltosylates. Some physical properties, such as solubility in water and organic solvents, refractive index and density, are considered as a function of the length of the *n*-alkyl substituent R and the nature of the anion.

19. Bogdanov, M.G., Svinyarov, I. V., Ivanova, B., Spitteller M., **Synthesis, spectroscopic and structural study of *trans*- and *cis*- (±)-3-phenyl-4-(pyrrolidine-1-carbonyl)isochroman-1-ones.** *Spectrochimica Acta* 77A (2010) 902–907.

Impact Factor (2009): **1.566**

**Abstract:** Synthesis, spectroscopic and structural study of *trans*- and *cis*-(±)-3-phenyl-4-(pyrrolidine-1-carbonyl)- isochroman-1-ones is performed in order to obtain the correlation between the crystal structure and spectroscopic properties of both isomers in solid-state. The methods such as single crystal X-ray diffraction, conventional and linear-polarized IR-spectroscopy, UV-spectroscopy, mass spectrometry, <sup>1</sup>H and <sup>13</sup>C NMR are employed. Quantum chemical ab initio and DFT calculations are performed, to support the experimental data for the electronic structure and optical properties.

## 2009

20. Akkurt, M., Baktr, Z., Bogdanov, M.G., Svinyarov, I.V., Büyükgüngör, O., ***trans*-6,7-Dimethoxy-1-oxo-3-(2-thienyl)isochroman-4-carboxylic acid.** *Acta Crystallographica* 65E (2009) o1377.

Impact Factor (2008): **0.367**

**Abstract:** The title compound, C<sub>16</sub>H<sub>14</sub>O<sub>6</sub>S, was synthesized by the reaction of 6,7-dimethoxy-homophthalic anhydride with thiophene-2-carbaldehyde in the presence of 4-(dimethylamino)pyridine (DMAP) as a basic catalyst. The thiophene ring of the title molecule is disordered over two sites with occupancies of 0.877 (3) and 0.123 (3). The

disorder corresponds to an approximate  $180^\circ$  rotation of the thiophene ring with respect to the C–C bond linking it to the rest of the molecule. The six-membered ring of the 3,4-dihydro-isochromanone ring system is not planar [puckering parameters  $Q_T = 0.571$  (2) Å,  $\vartheta = 115.2$  (2) $^\circ$  and  $\varphi = 99.1$  (2) $^\circ$ ]. The benzene ring of the 3,4-dihydro-isochromanone ring system makes dihedral angles of  $75.0$  (2) and  $77.2$  (5) $^\circ$  with the disordered thiophene rings. Intermolecular O–H $\cdots$ O and C–H $\cdots$ O hydrogen bonds, as well as C–H $\cdots$  $\pi$  interactions, lead to the observed supramolecular structure.

**2007**

21. Bogdanov, M.G., Mitrev, Y.N., Svinyarov, I.V., Palamarev, Ch.E., Palamareva, M.D., **Automatic selection of mobile phases. VII. Thin-layer chromatography on silica and alumina of 11,12-disubstituted *trans/cis*-11,12-dihydro-6H-dibenzo[*c,h*]chromen-6-ones.** *Journal of Liquid Chromatography and Related Technologies* 30 (2007) 2155–2169.

Impact Factor (2006): **0.825**

**Abstract:** Fifteen title compounds with four fused rings and two varying substituents were used to further specify the scope and limitation of a theoretical approach (the Snyder theory and LSChrom software) for selection of mobile phases avoiding any trial and error experiments. Based on the structure of the compounds, the theoretical treatment predicted values of strength 1 of suitable mobile phases for TLC, namely 0.311 for silica and 0.446 for alumina. Sixteen specific mobile phases with such or close values of  $\epsilon$  were arbitrarily selected from lists prepared by complex calculations. They were used to perform TLC. The data obtained showed that the retentions in all 256 measurements done were above the origin and below the solvent front. Thus, a good agreement between the theoretical and experimental data was established, thus proving a successful application of the approach. The retentions of all compounds and separation of one *cis/trans* pair depended on the stationary phase,  $\epsilon$ , and the tuning parameters  $m$  and  $P'$  of the mobile phases.