



# SCIFINDER®

A CAS SOLUTION

## SciFinder®

# The choice for chemistry research

October 25<sup>th</sup> – 28<sup>th</sup>, 2016

Bucharest, Romania

Veli-Pekka Hyttinen  
ACS International Ltd  
vhyttinen@cas.org

Tetiana Khristova  
ACS International Ltd  
tkhristova@acsi.info



SCIFINDER®

A CAS SOLUTION

# Chemical Abstracts Service

The leading global source of chemical information for scientific and patent research



## Renowned scientists trust SciFinder for research in scientific disciplines beyond traditional chemistry



**Dr. Nick Terrett**  
Chief Scientific Officer of  
Ensemble Therapeutics

*“SciFinder, in the past, had the reputation of being primarily chemistry-focused, but this is no longer the case.*

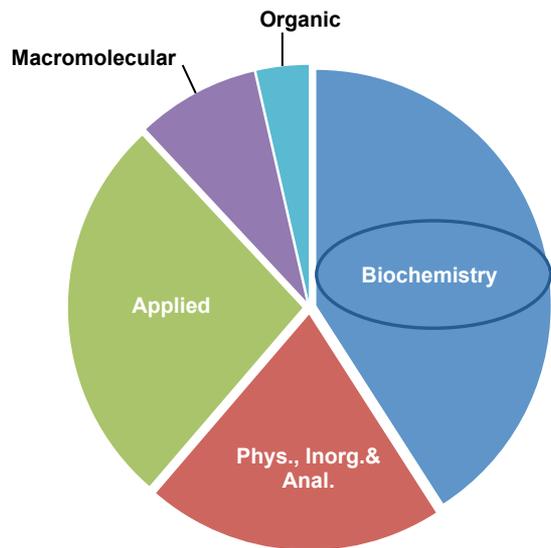
*In fact, SciFinder covers a multitude of different scientific fields. It covers chemistry; it covers biology and pharmacology. And from my perspective in drug discovery, importantly, it covers many other branches of the life sciences.*

***I use it as much for biology as I do for chemistry.”***

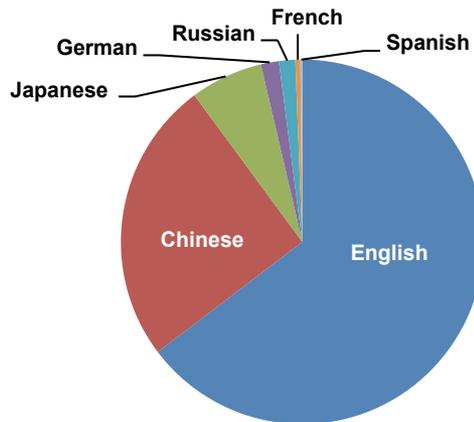
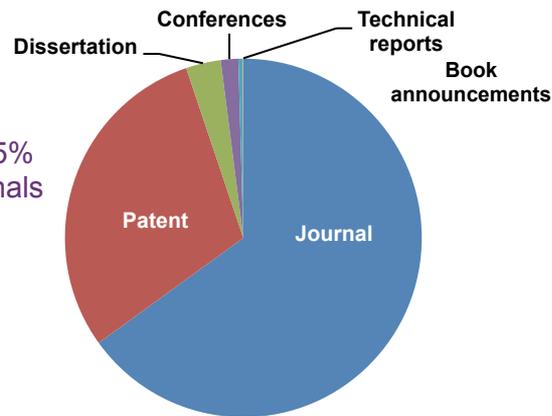


# CAS informs your research by providing comprehensive coverage of science disclosed in a variety of scientific disciplines, publications and languages

Discipline areas – 61%  
not in traditional chemistry

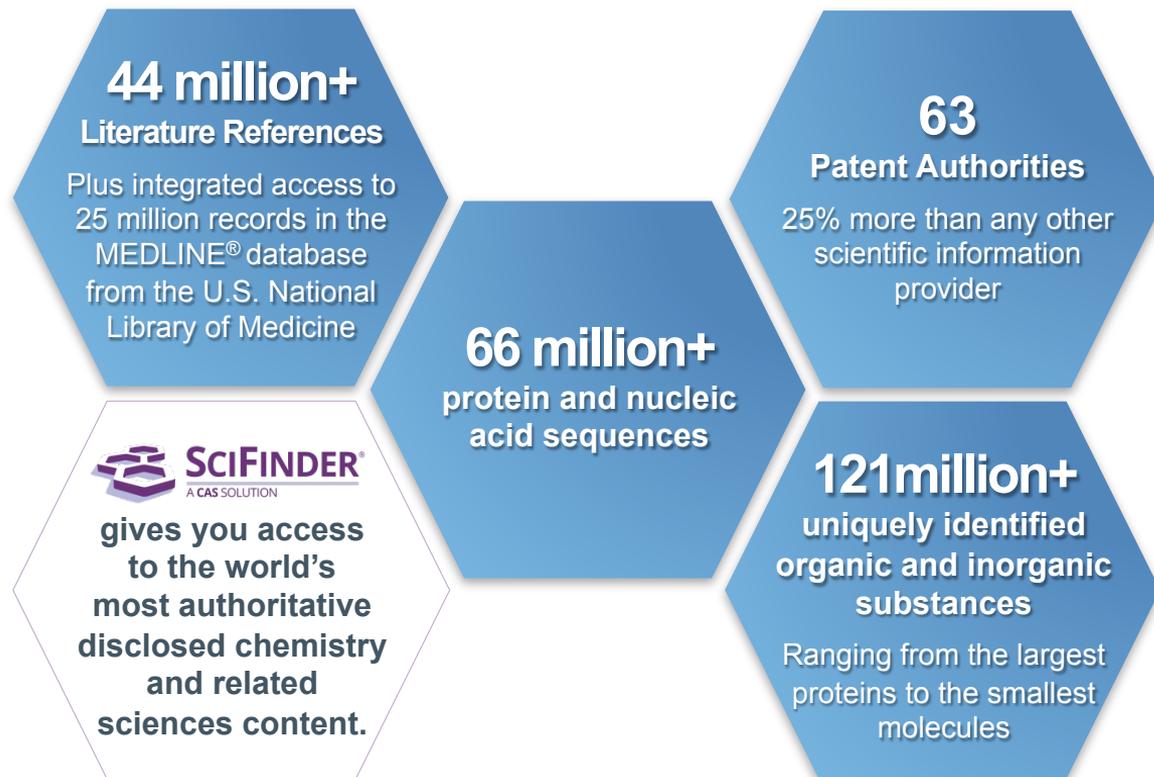


Disclosure types – 35%  
not published in journals



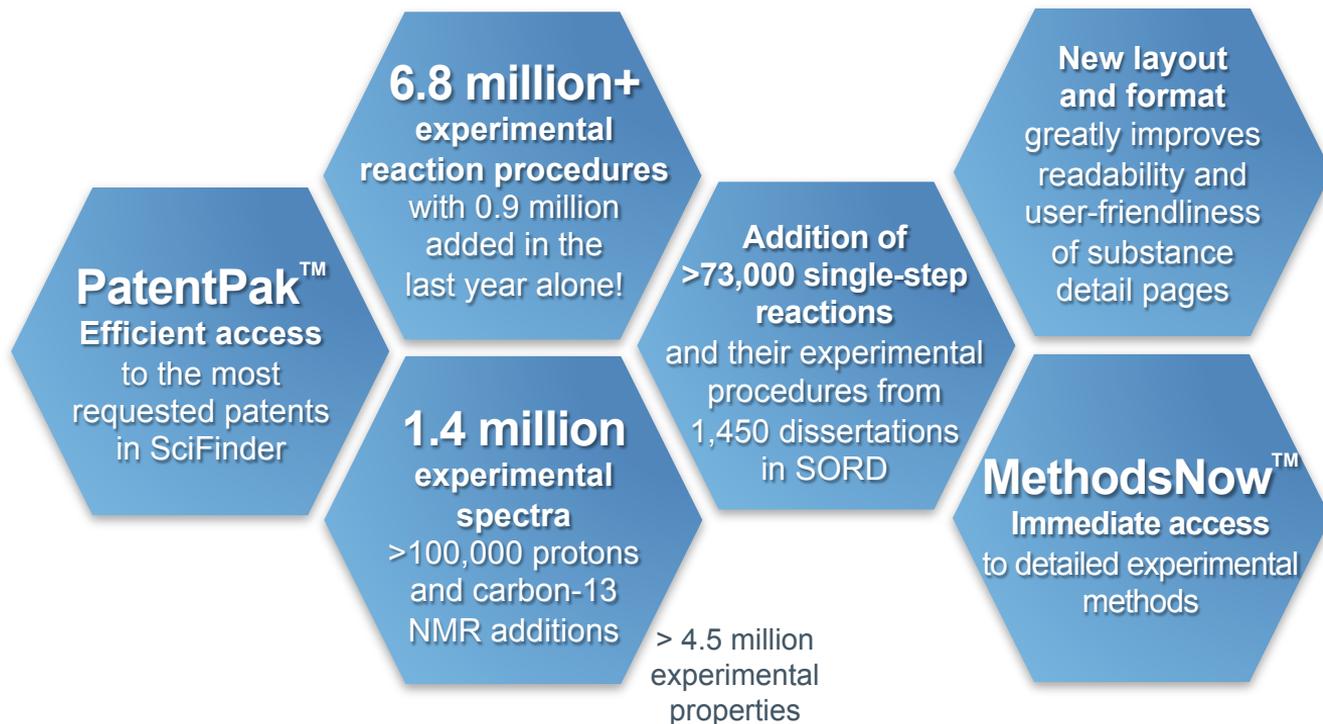
Original publication languages –  
35% not published in English

**CAS analyzes the world's disclosed research to keep SciFinder the most valuable tool supporting your organization's research enterprise**



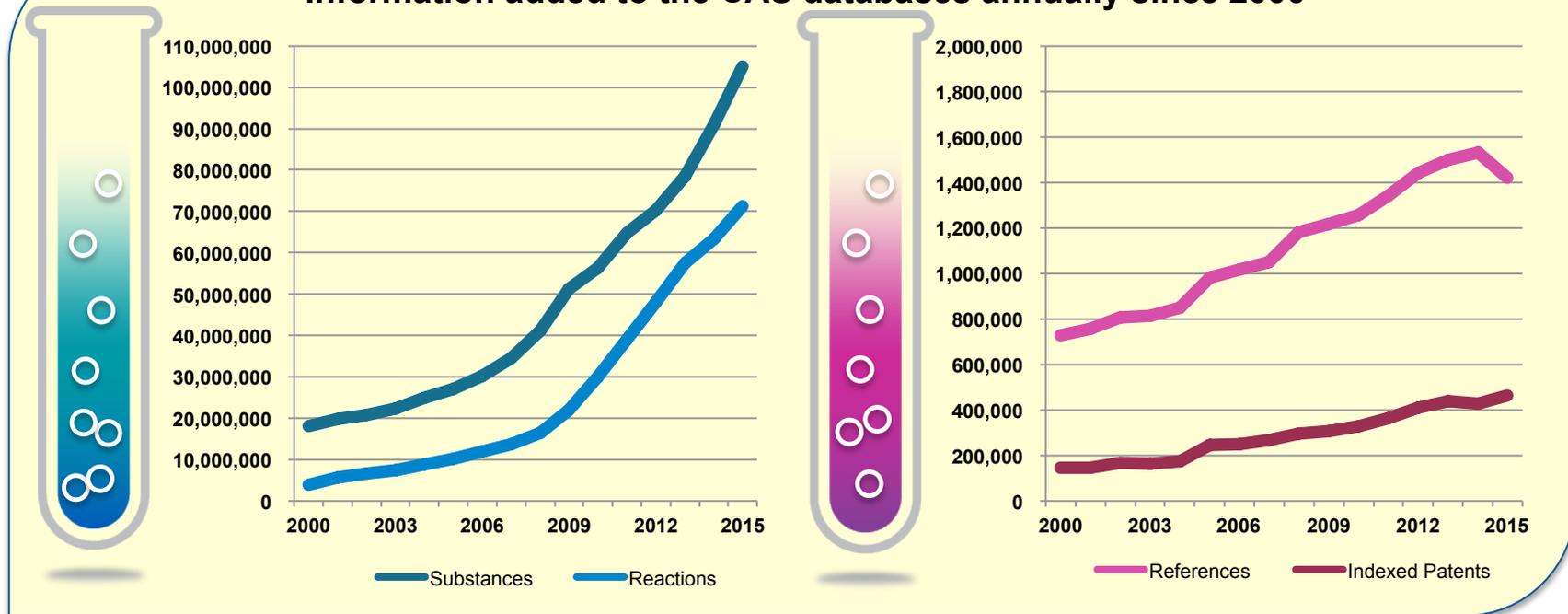
*Data as of March 2016*

# SciFinder saves you time by delivering valuable information to advance your research



# The rapid acceleration of disclosed research drives content growth in the CAS databases

## Information added to the CAS databases annually since 2000



# CAS scientists curate scientific information using standardized terminology to help researchers quickly find the most relevant search results in SciFinder

**SUBSTANCE DETAIL** [Get References](#) [Get Reactions](#) [Get Commercial Sources](#) [Send to SciPlans](#)

[Return](#) [Previous](#) | [Next](#)

**48. CAS Registry Number** 50-23-7

-55,285 -205

**C<sub>21</sub> H<sub>30</sub> O<sub>5</sub>**  
Pregn-4-ene-3,20-dione, 11,17,21-trihydroxy-, (11β)-

**Molecular Weight**  
362.46

**pKa (Predicted)**  
Value: 12.47±0.70 | Condition: Most Acidic Temp: 25 °C

**Melting Point (Experimental)**  
Value: 217-220 °C (decomp)

**Boiling Point (Predicted)**  
Value: 566.4±50.0 °C | Condition: Press: 760 Torr

**Density (Experimental)**  
Value: 1.4 g/cm<sup>3</sup>

**Other Names**  
Cortisol (8CI)  
11β,17,21-Trihydroxypregn-4-ene-3,20-dione  
11β,17,21-Trihydroxyprogesterone  
11β,17α,21-Trihydroxypregn-4-ene-3,20-dione  
11β-Hydroxycortisone  
[View more...](#)

Absolute stereochemistry.

Indexing in other search tools can be

- unpredictable
- less-detailed
- non-existent

▼ **BIOACTIVITY INDICATORS**

Indicators	References
Anti-infective agents (all) > > Antibacterial agents	220
Anti-infective agents (all) > > Antibiotics	338
Anti-infective agents (all) > > Antimicrobial agents	122
Anti-infective agents (all) > > Antiviral agents	179
Anti-infective agents (all) > > Fungicides	226
Anti-inflammatory agents (all) > Antiarthritics	64
Anti-inflammatory agents (all) > Anti-inflammatory agents	871
Anti-inflammatory agents (all) > Antirheumatic agents	98
Anti-inflammatory agents (all) > Nonsteroidal anti-inflammatory drugs	265
Antitumor agents (all) > Antiangiogenic agents	60
Antitumor agents (all) > Antitumor agents	404
Dermatological agents (all) > Dermatological agents	73
Immune agents (pharmaceutical) > Allergy inhibitors	83
Immune agents (pharmaceutical) > > Immunomodulators	117
Immune agents (pharmaceutical) > > Immunosuppressants	213
Natural products, pharmaceutical	89
Nervous system agents (all) > > > Analgesics	216
Nervous system agents (all) > > > Anesthetics	209
Receptor antagonists (all) > > Antihistamines	124
Respiratory system agents (all) > Antiasthmatics	104
Wound healing promoters	55

# Categorize and analyze options make it easy to find relevant information

**Categorize**

1. Select a heading and category.      2. Select index terms of interest.

Category Heading	Category	Index Terms	Selected Terms
All	Substances in biology (221)	<b>Select All</b> <b>Deselect All</b>	
General chemistry	Animal pathology (69)	<input type="checkbox"/> Interferons 7	
Biotechnology	Immunology (72)	<input type="checkbox"/> Antibodies and Immunoglobulins 5	
Synthetic chemistry	Processes & systems (44)	<input type="checkbox"/> Interferons, $\alpha$ 5	
Genetics & protein chemistry	Endocrinology (48)	<input type="checkbox"/> Vaccines 5	
Physical chemistry	Anatomy (27)	<input type="checkbox"/> Interleukin 2 3	
Polymer chemistry	Substances in adverse effects (16)	<input type="checkbox"/> Interleukin 4 3	
<b>Biology</b>		<input type="checkbox"/> Leukotriene B4 3	
Technology		<input type="checkbox"/> RANTES (chemokine) 3	
Analytical chemistry		<input type="checkbox"/> Spleen 3	
Environmental chemistry		<input type="checkbox"/> Tumor necrosis factor $\alpha$ 3	
		<input type="checkbox"/> Anti-HIV agents, vaccines 2	
		<input type="checkbox"/> CD4 antigens 2	
		<input type="checkbox"/> CXC chemokines 2	
		<input type="checkbox"/> Etanercept 2	
		<input type="checkbox"/> High throughput screening 2	

Biology > Immunology

**SUBSTANCES**

Get References   Get Reactions   Get Commercial Sources

Analyze   Refine

Sort by: CAS Registry Number

0 of 1 Substance Selected

Analyze by:

- Target Indicators
- Bioactivity Indicators**
- Commercial Availability
- Elements
- Reaction Availability
- Substance Role
- Transport proteins (all) 1

Show More

1. **28911-01-5**

~2061   ~34

**C<sub>17</sub> H<sub>12</sub> Cl<sub>2</sub> N<sub>4</sub>**  
4H-[1,2,4]Triazolo[4,3-a][1,4]benzodiazepine, 8-chloro-6-(2-chlorophenyl)-1-methyl-

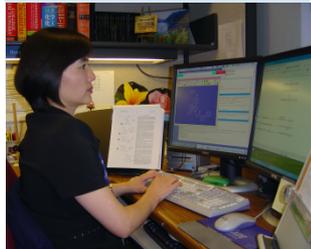
Regulatory Information  
Spectra  
Experimental Properties

# CAS scientists analyze, summarize and make scientific information accessible to colleagues worldwide

Source  
Selection



Document  
Indexing



Reaction  
Indexing



Markush  
Indexing



Authority  
Processing



Proprietary, standardized indexing in CAS databases ensures consistent, comprehensive search results.

# CAS scientists find the chemistry, and save you time!

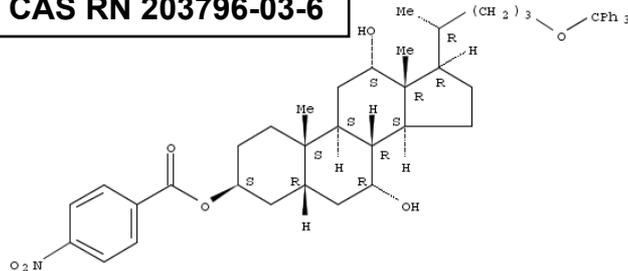
Compound 34: Diisopropyl azodicarboxylate (DIAD) (1.20 mL, 6.08 mmol) was added to triphenylphosphine (1.60 g, 6.08 mmol) in THF (100 mL) at 0 °C. and was stirred for half an hour during which time the yellow solution became a paste.

Compound 14 (2.58 g, 4.06 mmol) and p-nitrobenzoic acid (0.81 g, 4.87 mmol) were dissolved in THF (50 mL) and added to the paste. The resulted mixture was stirred at ambient temperature overnight. Water (100 mL) was added and the mixture was made slightly basic by adding NaHCO<sub>3</sub> solution followed by extraction with EtOAc (3x50 mL). The combined extracts were washed with brine once and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The desired product (2.72 g, 85% yield) was obtained as white powder after

SiO<sub>2</sub> chromatography (Et<sub>2</sub>O/hexanes 1:2). m.p. 207-209 °C.; IR (KBr) 3434, 3056, 2940, 2868, 1722, 1608, 1529, 1489, 1448, 1345 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 8.30-8.26 (m, 2 H), 8.21-8.16 (m, 2 H), 7.46-7.42 (m, 6 H), 7.31-7.18 (m, 9 H) 5.33 (bs, 1 H), 4.02 (bs, 1 H), 3.90 (bs, 1 H), 3.09-2.97 (m, 2 H), 2.68 (td, J=14.95, 2.56 Hz, 1 H), 2.29-2.19 (m, 1 H), 2.07-1.06 (series of multiplets, 24 H), 1.01 (s, 3 H), 0.98 (d, J=6.6 Hz, 3 H), 0.70 (s, 3 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 164.21, 150.56, 144.70, 136.79, 130.77, 64.22, 47.79, 46.79, 42.18, 28.74, 27.71, 26.85, 26.3

(thioglycerol+Na<sup>+</sup> matrix)

CAS RN 203796-03-6



Absolute stereochemistry.



# SciFinder allows you to explore and analyze the breadth of CAS content so you can be more effective in your work

Stay current in your field and keep track of competition

Quickly find and analyze information (research topic, structure, reaction)

Share ideas with colleagues and collaborators

The screenshot displays the SciFinder interface with the following elements:

- Navigation:** 'Explore', 'Saved Searches', 'SciPlanner', 'Save', 'Print', 'Export'.
- Alerts:** 'Keep Me Posted "Hydrogenation of Alkenes"[Apr 16, 2016] (3)'.
- Tools:** 'Get Substances', 'Get Reactions', 'Get Related Citations', 'Tools', 'Create Keep Me Posted Alert', 'Send to SciPlanner'.
- Analysis:** 'Analyze by: Author Name' dropdown menu.
- Results:** '0 of 3 References Selected'. Two results are shown, both titled 'Ph(i-Pr)SiH2: An Exceptional Reductant for Metal-Catalyzed Hydrogen Atom Transfers'. The first result includes a chemical reaction scheme showing an alkene reacting with a metal catalyst (Mn or Fe) and a silane reagent to form a secondary amine.

# Unlike its competitors, CAS covers patents in many languages, with enhanced translations

## 9. Process for the preparation of favipiravir

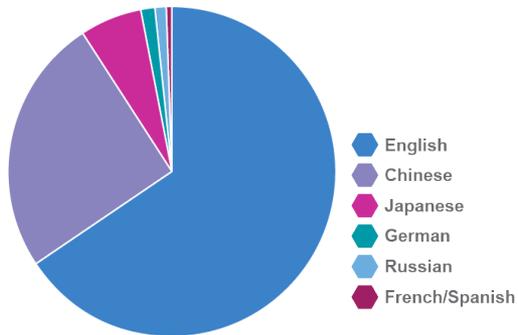
Quick View

PATENTPAK

By Bao, Jinyuan; H  
From Faming Zhua  
Patent No. CN 104496917  
Kind A  
Language Chinese  
A 20150408. | Language: Chinese, Database: CAPLUS

The invention relates to process for the prepn. of favipiravir. For example, favipiravir bromopyrazine-2-carboxylic acid to afford Me 3-amino-6-bromopyrazine-2-carboxylate, which and fluorination to afford Me 3-(benzyloxy)-6-fluoropyrazine-2-carboxylate, which underwent

Original publication languages not in English **35%**



(19) 中华人民共和国国家知识产权局



(12) 发明专利申请

(10) 申请公布号 CN 104496917 A  
(43) 申请公布日 2015.04.08

(21) 申请号 201410769599.5

(22) 申请日 2014.12.15

(71) 申请人 南京华威医药科技开发有限公司  
地址 210012 江苏省南京市仙林大学城纬地路9号

(72) 发明人 包金运 黄辉 梅玉伟 张孝清

(51) Int. Cl.  
C07D 241/24(2006.01)

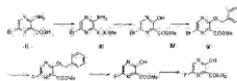
权利要求书12页 说明书6页

(54) 发明名称

一种法匹拉韦的合成方法

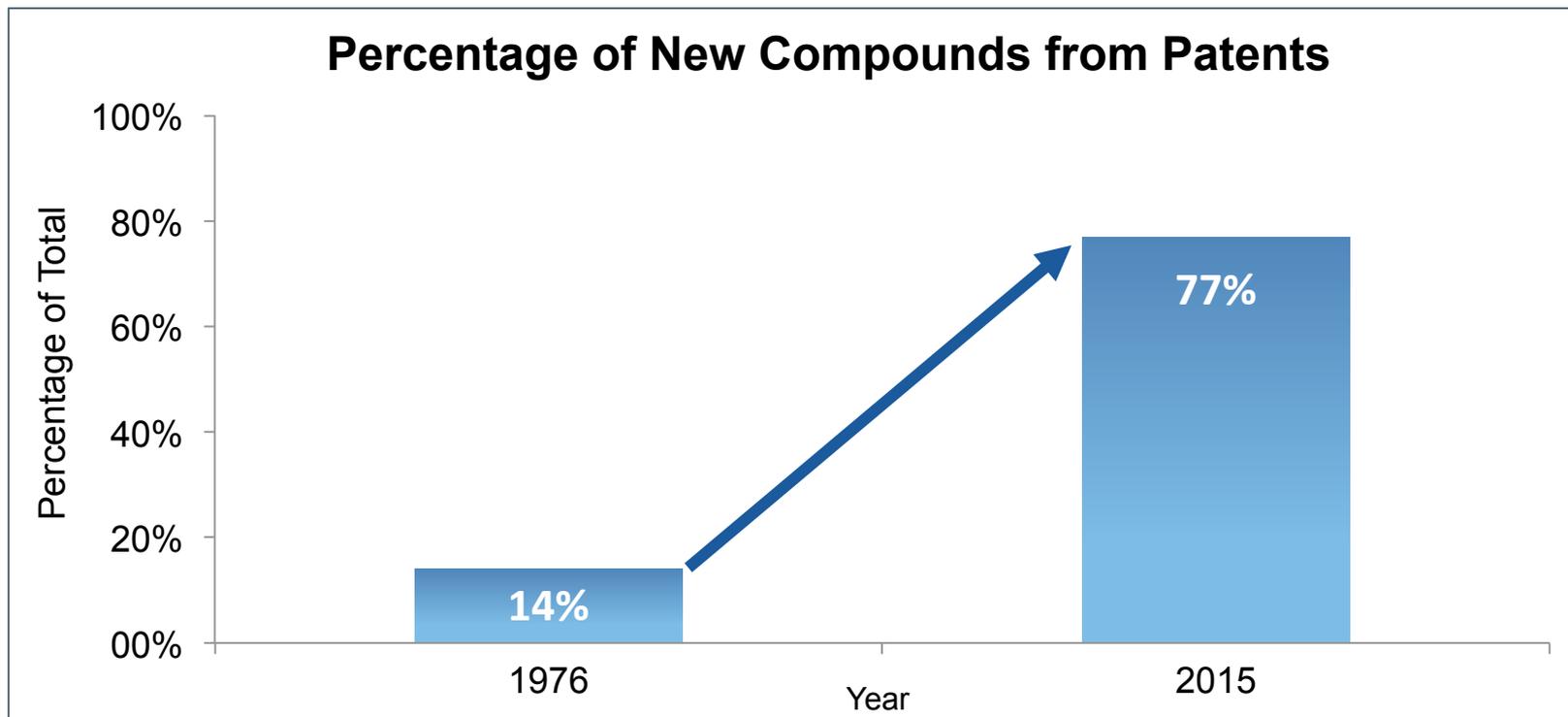
(57) 摘要

本发明属于药物化学领域,具体涉及一种法匹拉韦的合成方法。该方法是以式(II)为原料,经过羧基保护生成化合物(III),在浓硫酸和亚硝酸钠的作用下经重氮水解反应生成化合物(IV),然后经苄基保护反应生成化合物(V),然后在氯化钾和正丁基溴化镁的作用下生成化合物(VI),脱苄基保护基生成化合物(VII),然后加入氯化剂进行氯化反应生成法匹拉韦(式I)。本发明提供的方法反应周期短,操作简便,生产成本低,产品质量好,适合工业化生产。



SCI-FINDER®  
A CAS SOLUTION

## Increasingly, new compounds in the literature are first disclosed in patents





**PATENTPAK**<sup>TM</sup>  
A CAS SOLUTION

- Instant access to **searchable full-text patents** from major **patent offices around the world**
- Patent family coverage in **multiple languages**
- **Substance location mapping**
- **Interactive viewer** with built-in SciFinder search functionality
- **Secure and confidential** patent research
- **Daily updates** 8 mill patents from 31 countries



**SCIFINDER**<sup>®</sup>  
A CAS SOLUTION

# SciFinder PatentPak – Patent Family information

- 57. **Fluorine-containing substituted 5-[2-(pyrid-3-yl)-ethyl]-2,3,4-tetrahydro-1H-pyrido[4,3-b]indoles and hydrochlorides and hydrobromides thereof as agents for reducing uncontrolled protein aggregation in the nervous system, pharmacological agent based thereon and method for preparation and using thereof**

Quick View **PATENTPAK** ▼

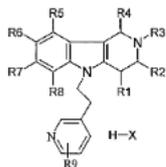
By **Bachurin, S. O.**; Ustyugov, A. A.; Ninkina, N. N.; Sokolov, V. B.; Aksinenko, A. Yu  
From Russ. (2013), RU 2490268 C2 20130820. | Language: Russian, Database: CAPLUS

Present invention refers to org. chem., namely to new fluorine-contg. (C1-C<sub>6</sub>H<sub>4</sub>) alkyl; R<sup>2</sup>, R<sup>3</sup>, R<sup>4</sup>, R<sup>5</sup> = H, F, Cl, Br, (C1-C6) alkyl, (C1-C6) al method of decreasing uncontrolled protein aggregation in the nervous system.

- 61. **Fluorine-containing substituted 5-[2-(pyrid-3-yl)-ethyl]-2,3,4-tetrahydro-1H-pyrido[4,3-b]indoles and hydrochlorides and hydrobromides thereof as agents for reducing uncontrolled protein aggregation in the nervous system, pharmacological agent based thereon and method for preparation and using thereof**

Quick View **PATENTPAK** ▼

By **Bachurin, Sergey Olegovich**; Ustugov, Aleksey Anatolyevich; Ninkina, Natalya N Aleksey Viktorovich  
From PCT Int. Appl. (2013), WO 2013070117 A2 20130516. | Language: Russian, Database: CAPLUS



The invention provides the alkynyl, C<sub>1</sub>-C<sub>6</sub> alkoxy, etc.; R<sup>9</sup> = F, 2F, CHF<sub>2</sub>, CCF<sub>2</sub>, reducing uncontrolled protein aggregation in the nervous system. The invention relates to the field of

## Patent Information

Patent No.	Kind	Language	Date
WO 2013070117	PATENTPAK A2		May 16, 2013
WO 2013070117	PATENTPAK A3	Russian	Jul 18, 2013
RU 2490268	PATENTPAK C2	Russian	Aug 20, 2013
AU 2012336451	A1		Jul 3, 2014
EP 2781518	PATENTPAK A2	English	Sep 24, 2014
CN 104105698	PATENTPAK A	Chinese	Oct 15, 2014
JP 2014533266	PATENTPAK T	Japanese	Dec 11, 2014
US 20150038526	PATENTPAK A1	English	Feb 5, 2015

## Priority Application

RU 2011-145513	A	Nov 10, 2011
WO 2012-RU894	W	Nov 1, 2012

- 62. **Agent for neutralizing toxic effect of tumor necrosis factor on the basis of tetrahydro-pyrido(4,3-b)indole derivatives and method for the treatment of autoimmune diseases**

Quick View **PATENTPAK** ▼

By Alesenko, A. V.; **Bachurin, S. O.**; Grigor'ev, V. V.; Gur'yanova, S. V.; Guss, F. V.; Lukina, G. V.; Lyangus, A. P.; Nasonov, E. L.; Sigidin, Ya. A.  
From Russ. (2013), RU 2477131 C1 20130310. | Language: Russian, Database: CAPLUS

Agent for neutralizing toxic effect of tumor necrosis factor on the basis of tetrahydro-pyrido(4,3-b)indole derivs. and method for the treatment of autoimmune diseases are disclosed. The invention relates to pharmaco. and involves agents for neutralizing toxic action of tumor necrosis factor on the basis of tetrahydro-pyrido(4,3-b)indole derivs. of

US 2008-61046712	P	Apr 21, 2008
WO 2009-US41276	W	Apr 21, 2009
JP 2011-505263	A3	Apr 21, 2009

CAPLUS

**LANGUAGE**

English

**Indexing**

Pharmacology (Section1-12)

Section cross-reference(s): 8, 9, 34, 63

**Concepts**

## Transcription factors

Antennapedia, of Drosophila, conjugates with polydentate ligands; selective high-affinity polydentate ligands of target mols. and methods of making such and uses for diagnosis and therapeutics in relation to delivery of effectors

Biological study, unclassified; Diagnostic use; Pharmacological activity; Therapeutic use; Biological study; Uses

## Viral transcription factor Tat

HIV, protein transduction domain, conjugates; selective high-affinity polydentate ligands of target mols. and methods of making such and uses for diagnosis and therapeutics in relation to delivery of effectors

Biological study, unclassified; Diagnostic use; Pharmacological activity; Therapeutic use; Biological study; Uses

## Histocompatibility antigen HLA-D

HLA-DR, target mols.; selective high-affinity polydentate ligands of target mols. and methods of making such and uses for diagnosis and therapeutics in relation to delivery of effectors

Adverse effect, including toxicity; Biological study, unclassified; Diagnostic use; Biological

**Substances**170908-81-3 

Page 104 in PatentPak™

DOTA-NHS-ester; selective high-affinity polydentate ligands of target mols. and methods of making such and uses for diagnosis and therapeutics in relation to delivery of effectors

Reactant; Reactant or reagent

83-44-3 Deoxycholic acid 495-69-2 Hippuric acid 744-59-2 Hippuryl-L-phenylalanine 15307-86-5 2-[(2,6-Dichlorophenyl)amino]benzeneacetic acid 21811-74-5 26129-32-8 40843-25-2 2-[4-(2,4-Dichlorophenoxy)phenoxy]propanoic acid 54857-86-2 5-(Tetradecyloxy)-2-furoic acid 58594-74-4 63058-21-9 69335-91-7 69806-34-4 74051-80-2 Sethoxydim 76578-12-6 89131-11-3 94588-27-9 

Page 84 in PatentPak™

Page 123 in PatentPak™

Page 123 in PatentPak™

Page 123 in PatentPak™

Page 123 in PatentPak™

Page 84 in PatentPak™

Page 123 in PatentPak™

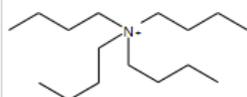
Page 123 in PatentPak™

Page 84 in PatentPak™

Page 123 in PatentPak™

Key Substances in Patent

CAS RN 1643-19-2



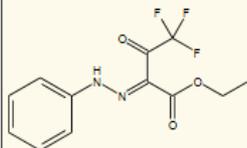
• Br •

[Search in SciFinder](#) | [View Detail](#)

Analyst Markup Locations (1)

[page 9](#)

CAS RN 1494-98-0



[Search in SciFinder](#) | [View Detail](#)

Analyst Markup Locations (2)

[page 10](#)

[page 15](#)

5 см<sup>-1</sup>: 3150, 1625 (NH), 1680 (C=O), 1620, 1540, 1510 (C=N, C=C), 1250-1100 (C-F). Спектр ЯМР <sup>1</sup>H (CDCl<sub>3</sub>, δ, м.д., J/Гц): 1.42 т (3H, OCH<sub>2</sub>Me, <sup>3</sup>J<sub>H,H</sub> 7.1), 4.41 к (2H, OCH<sub>2</sub>CH<sub>3</sub>, <sup>3</sup>J<sub>H,H</sub> 7.1), 7.24-7.26 и 7.43-7.46 оба м (5H, C<sub>6</sub>H<sub>5</sub>), 13.49 уш.с (1H, NH). Найдено, %: C 50.01, H 3.87, F 19.88, N 9.80. C<sub>12</sub>H<sub>11</sub>F<sub>3</sub>N<sub>2</sub>O<sub>3</sub>. Вычислено, %: C 50.01, H 3.85, F 19.77, N 9.72.

10 Пример 2. Этиловый эфир 2-[(4-метилфенил)гидразоно]-3-оксо-4,4,4-трифторбутановой кислоты (2). (Соединение формулы I, где R<sup>1</sup>=CF<sub>3</sub>, R<sup>2</sup>=Et, R<sub>3</sub>=4-Me).  
 Получают аналогично примеру 1 из 1.07 г п-толуидина (10 ммоль) и 1.84 г (10 ммоль) этилового эфира 3-оксо-4,4,4-трифторбутановой кислоты. Выход 2.45 г (81%), желтый  
 15 порошок, т.пл. 75-76°C. ИК спектр (DRA), ν, см<sup>-1</sup>: 3100, 1590 (NH), 1690 (C=O), 1630, 1520, 1500 (C=N, C=C), 1200-1080 (C-F). Спектр ЯМР <sup>1</sup>H (DMCO-d<sub>6</sub>, δ, м.д., J/Гц): 1.28 т (3H, OCH<sub>2</sub>Me, <sup>3</sup>J<sub>H,H</sub> 7.1), 2.28 с (3H, Me), 4.30 к (2H, OCH<sub>2</sub>CH<sub>3</sub>, <sup>3</sup>J<sub>H,H</sub> 7.1), 7.18-7.36 м (4H, C<sub>6</sub>H<sub>4</sub>), 11.65 уш.с (1H, NH). Найдено, %: C 51.89, H 4.59, F 18.58, N 9.15. C<sub>13</sub>H<sub>13</sub>F<sub>3</sub>N<sub>2</sub>O<sub>3</sub>.  
 20 Вычислено, %: C 51.66, H 4.34, F 18.86, N 9.27.

Пример 3. Этиловый эфир 2-(4-метоксифенил)гидразоно-3-оксо-4,4,4-трифторбутановой кислоты (3). (Соединение формулы I, где R<sup>1</sup>=CF<sub>3</sub>, R<sup>2</sup>=Et, R<sub>3</sub>=4-OMe).  
 Получают аналогично примеру 1 из 1.23 г (10 ммоль) п-анизидина и 1.84 г (10 ммоль)  
 25 этилового эфира 3-оксо-4,4,4-трифторбутановой кислоты. Выход 2.70 г (85%), желтые кристаллы, т.пл. 130-131°C. ИК спектр (DRA), ν, см<sup>-1</sup>: 3090 (NH<sup>вал</sup>), 1690 (C=O), 1640,

## MethodsNow™ is a complete CAS solution



- Largest single collection of methods information
  - Addresses core chemistry markets
- CAS-quality indexing and new, value-add templating of key methods from important full-text sources
- Covers both synthetic and analytical researcher needs

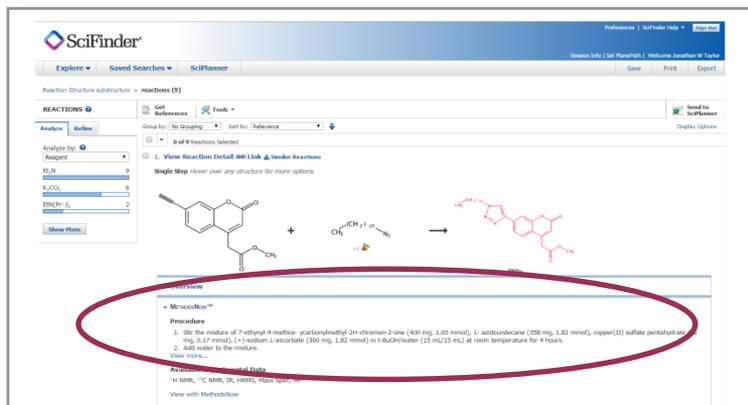
# So what exactly is MethodsNow?

- Largest single collection of methods information
  - Addresses core chemistry markets
- CAS-quality indexing and new, value-add templating of key methods from important full-text sources
- Covers 2+ million synthetic step-by-step protocols from 2000 onwards, and 200,000+ analytical methods focusing in Pharma, Ag, and chemicals
- Details for analytical researchers such as matrix, analyte, instrumentation and comparison capabilities

# One product, two interfaces

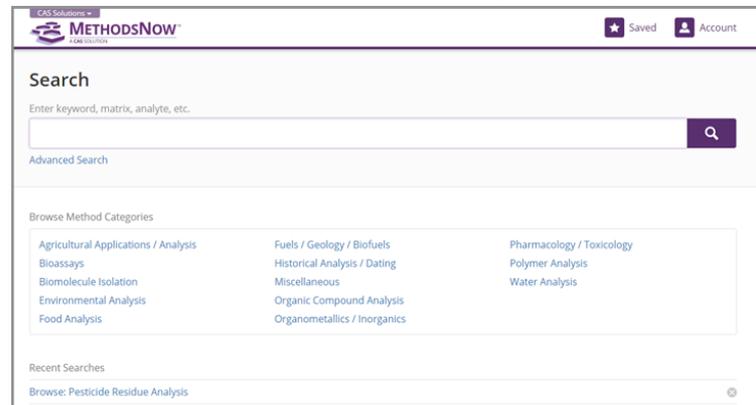
- Research showed that users interested in synthetic methods were often already in SciFinder, but analytical scientists often weren't (though they might be familiar with it)

**Synthetic chemist looking for great methods?  
They are in SciFinder.**



The screenshot shows the SciFinder interface. At the top, there are navigation tabs for 'Explore', 'Saved Searches', and 'SciFinder'. Below this, there's a search bar and a 'Send to SciFinder' button. The main content area displays a chemical reaction scheme with a red oval highlighting the 'Procedure' section below it. The procedure text reads: '1. Stir the mixture of 7-ethyl-4-methoxy-ycarboxymethyl-2H-chromen-2-one (400 mg, 1.05 mmol), 1-azidoundecane (250 mg, 1.82 mmol), copper(II) sulfate pentahydrate (30 mg, 0.17 mmol), (+)-sodium L-ascorbate (360 mg, 1.82 mmol) in t-BuOH/water (15 mL/15 mL) at room temperature for 4 hours. 2. Add water to the mixture. View more...'. Below the procedure, there are sections for 'View with MethodNow' and 'View with MethodNow'.

**Analytical scientist just looking for great methods?  
A new, easy to use interface just for you.**



The screenshot shows the MethodsNow interface. At the top, there are navigation tabs for 'CAS Solutions' and 'METHODSNOW'. Below this, there's a search bar and a 'Send to SciFinder' button. The main content area displays a search bar with the text 'Enter keyword, matrix, analyte, etc.' and a search button. Below the search bar, there's a section for 'Advanced Search' and a section for 'Browse Method Categories'. The categories are listed in a grid:

Agricultural Applications / Analysis	Fuels / Geology / Biofuels	Pharmacology / Toxicology
Bioassays	Historical Analysis / Dating	Polymer Analysis
Biomolecule Isolation	Miscellaneous	Water Analysis
Environmental Analysis	Organic Compound Analysis	
Food Analysis	Organometallics / Inorganics	

Below the categories, there's a section for 'Recent Searches' with the text 'Browse: Pesticide Residue Analysis'.

# MethodsNow– Analysis: A Workflow Solution Powered By Excellent Content

## MethodsNow journal content

<b>Content from years</b>	<b>2000 - present</b>
Number of methods	>220,000
Content coverage	Based on existing CPlus content in the biochemical, applied, analytical CAS sections
Focus	Small molecule-centric
Example journal titles	Food Chemistry, Journal of Chromatography A and B, Journal of Agricultural and Food Chemistry, Talanta, Analytica Chimica Acta
Language	English only

# MethodsNow – Analysis: Main Categories

Method categories	Primary content in these categories
active-pharmaceutical-ingredient	Pharmacology, toxicology, SAR, analysis of drugs
bioassay	Pharmacology, toxicology, biochemical methods
biomolecule-isolation-assay	Biochemical methods, mammalian hormones
bioorganism-isolation-assay	Biochemical genetics
element-detection	Inorganic analytical chemistry
environmental-analysis	Essential oils/cosmetics
food-analysis	Additives, food processing, contaminants, GMO
forensic-analysis	Toxicology
natural-product-isolation-analysis	Analysis of drugs
organic-compound-analysis	Organic analytical chem.
pesticide-residue-analysis	Food, feed, toxicology, agrochemical bioregulators
trace-element-analysis	Inorganic analytical chemistry, benzene
water-wastewater-sludge-analysis	Chemical water analysis (i.e. contaminants in water)

# MethodsNow – Analytical Scientist Interface

CAS Solutions ▾

**METHODSNOW™**  
A CAS SOLUTION

★ Saved    👤 Account

## Search

Enter keyword, matrix, analyte, etc.

Advanced Search

### Browse Method Categories

<a href="#">Agricultural Applications / Analysis</a>	<a href="#">Fuels / Geology / Biofuels</a>	<a href="#">Pharmacology / Toxicology</a>
<a href="#">Bioassays</a>	<a href="#">Historical Analysis / Dating</a>	<a href="#">Polymer Analysis</a>
<a href="#">Biomolecule Isolation</a>	<a href="#">Miscellaneous</a>	<a href="#">Water Analysis</a>
<a href="#">Environmental Analysis</a>	<a href="#">Organic Compound Analysis</a>	
<a href="#">Food Analysis</a>	<a href="#">Organometallics / Inorganics</a>	

### Recent Searches

[hplc lycopene analysis](#) ✕

# Specify one or many advanced search fields

CAS Solutions **METHODSNOW**  
A CAS SOLUTION

★ Saved Account

[← Return to Home](#)

## Advanced Search

Keyword

AND  Matrix

AND  Analyte

Add Search Criteria

CAS Solutions **METHODSNOW**  
A CAS SOLUTION

★ Saved Account

[← Return to Home](#)

## Advanced Search

Publication Name

Keyword

Analyte

Matrix

Method Category

Technique

CAS Method Number

Publication Name

- Keyword
- Analyte
- Matrix
- Method Category
- Technique
- CAS Method Number
- Publication Name

## Search

Enter keyword, matrix, analyte, etc.



[Advanced Search](#)

### Browse Method Categories

[Agricultural Applications / Analysis](#)

[Bioassays](#)

[Biomolecule Isolation](#)

[Environmental Analysis](#)

[Food Analysis](#)

[Fuels / Geology / Biofuels](#)

[Historical Analysis / Dating](#)

[Miscellaneous](#)

[Organic Compound Analysis](#)

[Organometallics / Inorganics](#)

[Pharmacology / Toxicology](#)

[Polymer Analysis](#)

[Water Analysis](#)

### Recent Searches

carbamazepine 

sabinene 

CAS Solutions - METHODS NOW™ A CAS SOLUTION

carbamazepine

Results (488) Sort Relevance

Return to Home

**Analyte**

- Carbamazepine (291)
- Diclofenac (89)
- Acetaminophen (70)
- Metoprolol (69)
- Propranolol (63)
- [View All](#)

**Matrix**

- Blood plasma (140)
- Wastewater (62)
- Pharmaceutical tablets (41)
- Surface waters (39)
- Blood serum (32)
- [View All](#)

**Method Category**

**Technique**

**Year**

**Analysis of Carbamazepine in Blood plasma by HPLC**  
CAS MN: 1-101-CAS-714

[View Details & Instructions](#) [Add to Compare](#)

Analyte **Carbamazepine**; Carbamazepine 10,11-epoxide; *trans*-10,11-Dihydroxy-10,11-dihydrocarbamazepine

Matrix Blood plasma

Other Materials Material: 0.45 µm regenerated cellulose membrane filter; analytical column (250 mm x 4.6 mm; 5 µm); cartridges (30 mg,1 mL, particle size 30 µm)

Method Category Active Pharmaceutical Ingredient and Metabolite Analysis

Technique Solid phase extraction; HPLC

Equipment Used HPLC system; Vacuum Manifold

Source **Development and validation of a solid phase extraction-HPLC method for the determination of carbamazepine and its metabolites, carbamazepine epoxide and carbamazepine trans-diol, in plasma**  
Dzodic, Predrag; Zivanovic, Ljiljana; Protic, Ana; Ivanovic, Ivana; Velickovic-Radovanovic, Radmila; Spasic, Mirjana; Lukic, Stevo; Zivanovic, Slavoljub  
Journal of the Serbian Chemical Society (2012), 77 (10), 1423-1436, Serbian Chemical Society

[Document Sources](#)

[Abstract](#)

**Analysis of Carbamazepine in Pharmaceutical tablets by Reversed-phase HPLC**  
CAS MN: 1-101-CAS-13062

[View Details & Instructions](#) [Add to Compare](#)

Analyte **Carbamazepine**

Matrix Pharmaceutical capsules; Pharmaceutical tablets

Return to Home

**Analyte**

- Carbamazepine (38)
- Carbamazepine 10,11-epoxide (20)
- Phenytoin (17)
- Oxcarbazepine (10)
- Phenobarbital (10)
- [View All](#)

**Matrix**

- Wastewater (54)
- Blood plasma (38)
- Surface waters (35)
- Pharmaceutical tablets (22)
- Blood serum (21)
- [View All](#)

**Method Category**

**Technique**

- HPLC (17)
- Liquid-liquid extraction (9)
- Solid phase extraction (8)
- HPLC-tandem mass spectrometry (4)
- Liquid chromatographic UV detectors (4)
- [View All](#)

**Year**

[← Return to Home](#)

^ **Analyte**

- Carbamazepine (8)
  - Carbamazepine 10,11-epoxide (4)
  - Oxcarbazepine (3)
  - trans*-10,11-Dihydroxy-10,11-dihydrocarbamazepine (3)
  - Eslicarbazepine acetate (2)
- [View All](#)

^ **Matrix**

- Blood plasma (8)
  - Brain (1)
  - Drinking waters (1)
  - Kidney (1)
  - Liver (1)
- [View All](#)

∨ **Method Category**

^ **Technique**

- HPLC (17)
  - Liquid-liquid extraction (9)
  - Solid phase extraction (8)
  - HPLC-tandem mass spectrometry (4)
  - Liquid chromatographic UV detectors (4)
- [View All](#)

∨ **Year**

## Results (8)

Sort Relevance -



[Compare \(0/3\)](#)

**Analysis of Carbamazepine in Blood plasma by HPLC**

CAS MN: 1-101-CAS-714

[View Details & Instructions](#)

[Add to Compare](#)

Analyte **Carbamazepine**; Carbamazepine 10,11-epoxide; *trans*-10,11-Dihydroxy-10,11-dihydrocarbamazepine

Matrix Blood plasma

Other Materials Material: 0.45 µm regenerated cellulose membrane filter; analytical column (250 mm x 4.6 mm; 5 µm); cartridges (30 mg, 1 mL, particle size 30 µm)

Method Category Active Pharmaceutical Ingredient and Metabolite Analysis

Technique Solid phase extraction; HPLC

Equipment Used HPLC system; Vacuum Manifold

Source **Development and validation of a solid phase extraction-HPLC method for the determination of carbamazepine and its metabolites, carbamazepine epoxide and carbamazepine trans-diol, in plasma**

Dzodic, Predrag; Zivanovic, Ljiljana; Protic, Ana; Ivanovic, Ivana; Velickovic-Radovanovic, Radmila; Spasic, Mirjana; Lukic, Stevo; Zivanovic, Slavoljub

Journal of the Serbian Chemical Society (2012), 77 (10), 1423-1436. Serbian Chemical Society

[Document Sources](#)

[Abstract ∨](#)

**Analysis of Phenobarbital in Blood plasma by Solid phase extraction**

CAS MN: 1-101-CAS-66910


[← Return to Results](#)

## Method Detail (2 of 8)

### Analysis of Phenobarbital in Blood plasma by

CAS MN: 1-101-CAS-66910

Method Category: Active Pharmaceutical Ingredient and Metabolite Analysis

Technique: Solid phase extraction; UV radiation; HPLC

Materials	Role
trans-10,11-Dihydroxy-10,11-dihydrocarbamazepine	analyte
<b>Carbamazepine</b>	analyte
Lamotrigine	analyte
Licarbazepine	analyte
Phenobarbital	analyte
Carbamazepine 10,11-epoxide	analyte
Primidone	analyte
Oxcarbazepine	analyte
Phenytoin	analyte
Blood plasma	matrix
C18 column (55 mm × 4 mm; 3 μm particle size)	material
Methanol	reagent
Acetonitrile	reagent
Phosphoric acid	reagent

### Source

First HPLC-UV method for rapid and simultaneous quantification of phenobarbital, primidone, phenytoin, carbamazepine, carbamazepine-10,11-epoxide, 10,11-trans-dihydroxy-10,11-dihydrocarbamazepine, lamotrigine, oxcarbazepine and licarbazepine in human plasma

Serralheiro, Ana; Alves, Gilberto; Fortuna, Ana; Rocha, Marília; Falcao, Amílcar

Journal of Chromatography B: Analytical Technologies in the Biomedical and Life Sciences (2013), 925 (1), 1 - 9. Elsevier B.V.

CODEN: JCBAAI | ISSN: 15700232 | DOI: 10.1016/j.jchromb.2013.02.026

### Abstract

A sensitive and fast high-performance liquid chromatog. method coupled with UV detection is herein reported for the simultaneous determination of human plasma concentration of six antiepileptic drugs frequently used in clin. practice [phenobarbital (PB), primidone (PRM), phenytoin (PHT), carbamazepine (CBZ), lamotrigine (LTG), oxcarbazepine (OXC)] and some of their main metabolites, carbamazepine-10,11-epoxide (CBZ-E), 10,11-trans-dihydroxy-10,11-dihydrocarbamazepine (trans-diol) and licarbazepine (Lic). Sample preparation consisted of a deproteinization step with methanol followed by a solid-phase extraction procedure. Chromatog. separation was achieved in approx. 15 min on a reversed-phase C<sub>18</sub> column using a mobile phase composed by water-methanol-acetonitrile-triethylamine (68.7:25:6:0.3, volume/volume/volume/volume; pH 6.5) pumped isocratically at 1.0 mL/min. The detector was set at 237 nm. Calibration curves were linear with regression coefficients greater than 0.992 over the concentration ranges 0.25-100 μg/mL for PB, 0.4-50 μg/mL for PRM, 0.5-50 μg/mL for PHT, 0.1-50 μg/mL for CBZ, LTG and CBZ-E, 0.1-25 μg/mL for OXC, 0.25-10 μg/mL for trans-diol and 0.15-80 μg/mL for Lic. Inter- and intra-day imprecision did not exceed 12.15% and inaccuracy was within ±14.91%. Absolute mean recoveries ranged from 78.49 to 101.04% and no interferences were observed at the retention times of the analytes and internal standard (ketoprofen). This bioanal. method was successfully applied to real plasma samples from epileptic patients and it seems to be a suitable tool for routine therapeutic drug monitoring and also to support other clin. pharmacokinetic-based studies.

### Equipment Used

Liquid chromatograph, BAS-480

### Conditions

#### Chromatographic

Mobile phase - water:methanol:acetonitrile:TEA (68.7:25:6:0.3, v/v/v/v) adjust at pH 6.5 with ortho-phosphoric acid 85%. Isocratic elution flow rate - 1.0 mL/min, wavelength detection - 237 nm



## Instructions

### Preparation of standard solution

1. Prepare stock solutions of Phenobarbital (PB 20 mg/mL), Primidone (PRM 10 mg/mL), Phenytoin (PHT 10 mg/mL), **Carbamazepine** (CBZ 10 mg/mL), Lamotrigine (LTG 10 mg/mL), Oxcarbazepine (OXC 5 mg/mL), **Carbamazepine**-10,11-epoxide (CBZ-E 10 mg/mL), *trans*-diol (10 mg/mL), Licarbazepine 20 mg/mL and IS (1 mg/mL) individually by dissolving appropriate amounts of each compound in methanol.
2. Dilute these solutions adequately with methanol to obtain the corresponding working solutions.
3. Mix stock and working solutions of drugs and metabolites properly to afford six combined spiking solutions with final concentrations of 6.25, 12.5, 50, 100, 250 and 500 µg/mL for PB; 10, 20, 50, 125, 500 and 1250 µg/mL for PRM; 12.5, 25, 50, 125, 500 and 1250 µg/mL for PHT; 2.5, 5, 12.5, 50, 250 and 1250 µg/mL for CBZ, LTG and CBZ-E; 2.5, 5, 12.5, 25, 125 and 625 µg/mL for OXC; 6.25, 12.5, 25, 50, 100 and 250 µg/mL for *trans*-diol and 3.75, 7.5, 20, 100, 500 and 2000 µg/mL for Lic.

### Solid phase extraction

1. Add an aliquot of human plasma (500 µL) of 20 µL of the IS working solution and mix with 1 mL of methanol in order to precipitate plasma proteins.
2. After centrifuging at 13,400 rpm for 10 min, evaporate the resulting supernatant under a gentle nitrogen stream at 80 °C for 10 min.
3. Dilute the residual volume of supernatant with 1.5 mL of water and vortex-mix for 30 s.
4. Subject the pre-treated sample to a solid-phase extraction (SPE) on the Oasis® HLB (30 mg, 1 mL) cartridge (Waters, Milford, MA, USA), which is previously conditioned with 1 mL of methanol, 1 mL of acetonitrile and 1 mL of water-acetonitrile (95:5, v/v).
5. Submit the loaded cartridge subsequently to -60 kPa and wash four times with 1 mL of water.
6. After drying the sorbent under airflow for 5 min, elute the analytes with 1 mL of ethyl acetate using gentle vacuum.
7. Evaporate the eluate to dryness at 45 °C under a gentle stream of nitrogen gas and reconstitute with 500 µL of mobile phase by vortexing and ultrasonication.
8. Inject 20 µL of the final mixture into the chromatographic system.

### Method or Procedure

1. Perform the chromatographic analysis on a BAS-480 liquid chromatograph equipped with a PM-80 pump, a Rheodyne manual injector with a 20 µL loop, a BAS UV-116 UV-vis detector, a BAS DA-5 chromatography control and a data system interface (Bioanalytical Systems, West Lafayette, IN, USA)
2. Achieve the data acquisition by means of BAS Chromgraph Control and Chromgraph Report software version 2.30.
3. Perform the chromatographic analysis of all the six drugs, metabolites and IS at room temperature.
4. Carry out the separation on reversed-phase LiChroCART® Purospher Star® C18 column (55 mm x 4 mm; 3 µm particle size) purchased from Merck KGaA (Darmstadt, Germany).
5. Apply an isocratic elution at a flow rate of 1.0 mL/min with a mobile phase composed of water-methanol-acetonitrile-TEA (68.7:25.6:0.3, v/v/v/v) and adjust at pH 6.5 with ortho-phosphoric acid 85%.
6. Set the wavelength detection at 237 nm.

## Validation

Linearity Range	0.40 - 50 µg/mL, Primidone
	0.10 - 50 µg/mL, Lamotrigine
	0.25 - 10 µg/mL, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine
	0.25 - 100 µg/mL, Phenobarbital
	0.15 - 80 µg/mL, Licarbazepine
	0.10 - 50 µg/mL, <b>Carbamazepine</b> -10,11-epoxide

Limit of Quantitation	0.40 µg/mL, Primidone 0.10 µg/mL, Lamotrigine 0.25 µg/mL, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine 0.25 µg/mL, Phenobarbital 0.15 µg/mL, Licarbazepine 0.10 µg/mL, <b>Carbamazepine</b> -10,11-epoxide 0.10 µg/mL, Oxcarbazepine 0.50 µg/mL, Phenytoin 0.10 µg/mL, <b>Carbamazepine</b>
Recovery	92.24 ± 3.99%, 80.17 ± 2.84%, 94.47 ± 2.80% spiked in 1.2 µg/mL, 25 µg/mL, 45 µg/mL respectively, Primidone 84.87 ± 5.89%, 84.16 ± 3.20%, 85.96 ± 2.41% spiked in 0.3 µg/mL, 25 µg/mL, 45 µg/mL respectively, Lamotrigine 83.57 ± 3.94%, 83.13 ± 2.57%, 88.68 ± 2.48% spiked in 0.75 µg/mL, 5 µg/mL, 9 µg/mL respectively, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine 89.77 ± 4.77%, 81.35 ± 2.60%, 83.80 ± 2.60% spiked in 0.75 µg/mL, 50 µg/mL, 90 µg/mL respectively, Phenobarbital 101.04 ± 3.83%, 87.45 ± 3.05%, 91.42 ± 2.67% spiked in 0.45 µg/mL, 40 µg/mL, 72 µg/mL respectively, Licarbazepine 89.43 ± 6.02%, 85.27 ± 3.05%, 90.46 ± 2.67% spiked in 0.3 µg/mL, 25 µg/mL, 45 µg/mL respectively, <b>Carbamazepine</b> -10,11-epoxide 82.88 ± 1.95%, 86.96 ± 2.98%, 89.01 ± 2.47% spiked in 0.3 µg/mL, 12.5 µg/mL, 22.5 µg/mL respectively, Oxcarbazepine 84.19 ± 7.42%, 82.81 ± 3.63%, 78.49 ± 2.60% spiked in 1.5 µg/mL, 25 µg/mL, 45 µg/mL respectively, Phenytoin 88.89 ± 9.37%, 88.46 ± 3.55%, 86.37 ± 2.57% spiked in 0.3 µg/mL, 25 µg/mL, 45 µg/mL respectively, <b>Carbamazepine</b>
Accuracy	7.38% (bias, Inter-day); 1.94% (bias, Intra-day), Primidone 5.78% (bias, Inter-day); 16.43% (bias, Intra-day), Lamotrigine 5.46% (bias, Inter-day); 6.09% (bias, Intra-day), 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine -1.58% (bias, Inter-day); -13.93% (bias, Intra-day), Phenobarbital -5.28% (bias, Inter-day); 5.47% (bias, Intra-day), Licarbazepine 9.24% (bias, Inter-day); 10.87% (bias, Intra-day), <b>Carbamazepine</b> -10,11-epoxide 6.26% (bias, Inter-day); 0.43% (bias, Intra-day), Oxcarbazepine 1.22% (bias, Inter-day); -0.17% (bias, Intra-day), Phenytoin 10.88% (bias, Inter-day); 14.74% (bias, Intra-day), <b>Carbamazepine</b>
Precision	13.48% (%CV, Inter-day); 3.31% (%CV, Intra-day), Primidone 14.95% (%CV, Inter-day); 14.29% (%CV, Intra-day), Lamotrigine 9.6% (%CV, Inter-day); 2.42% (%CV, Intra-day), 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine 18.27% (%CV, Inter-day); 8.09% (%CV, Intra-day), Phenobarbital 17.93% (%CV, Inter-day); 7.62% (%CV, Intra-day), Licarbazepine 10.22% (%CV, Inter-day); 10.41% (%CV, Intra-day), <b>Carbamazepine</b> -10,11-epoxide 12.4% (%CV, Inter-day); 7.04% (%CV, Intra-day), Oxcarbazepine 12.05% (%CV, Inter-day); 4.66% (%CV, Intra-day), Phenytoin 12.31% (%CV, Inter-day); 14.85% (%CV, Intra-day), <b>Carbamazepine</b>

**Analysis of Phenobarbital in Blood plasma by Solid phase extraction**

CAS MN: 1-101-CAS-66910

[View Details & Instructions](#)

[Remove from Compare](#)

Analyte *trans*-10,11-Dihydroxy-10,11-dihydrocarbamazepine; **Carbamazepine**; Lamotrigine; Licarbazepine; Phenobarbital; Carbamazepine 10,11-epoxide; Primidone; Oxcarbazepine; Phenytoin

Matrix Blood plasma

Other Materials Reagent: Methanol; Acetonitrile; Phosphoric acid  
Material: C18 column (55 mm × 4 mm; 3 μm particle size)

Method Category Active Pharmaceutical Ingredient and Metabolite Analysis

Technique Solid phase extraction; UV radiation; HPLC

Equipment Used Liquid chromatograph

Source **First HPLC-UV method for rapid and simultaneous quantification of phenobarbital, primidone, phenytoin, carbamazepine, carbamazepine-10,11-epoxide, 10,11-trans-dihydroxy-10,11-dihydrocarbamazepine, lamotrigine, oxcarbazepine and licarbazepine in human plasma**

Serralheiro, Ana; Alves, Gilberto; Fortuna, Ana; Rocha, Marília; Falcao, Amílcar

Journal of Chromatography B: Analytical Technologies in the Biomedical and Life Sciences (2013), 925 (1), 1-9. Elsevier B.V.

[Document Sources](#)

[Abstract](#) ▾

**Analysis of Carbamazepine in Blood plasma by Solid phase extraction**

CAS MN: 1-101-CAS-133183

[View Details & Instructions](#)

[Remove from Compare](#)

Analyte **Carbamazepine**

Matrix Drinking waters; Urine; Blood plasma

chromatographic analysis

Source **Solid-phase extraction combined with dispersive liquid-liquid microextraction as an efficient and simple method for the determination of carbamazepine in biological samples**

Rezaee, Mohammad; Mashayekhi, Hossein Ali

Analytical Methods (2012), 4 (9), 2887-2892. Royal Society of Chemistry

[Document Sources](#)

[Abstract](#) ▾

**Analysis of Carbamazepine in Blood plasma by Reversed-phase HPLC**

CAS MN: 1-101-CAS-60070

[View Details & Instructions](#)

[Remove from Compare](#)

Analyte Licarbazepine; Oxcarbazepine; Eslicarbazepine acetate; *trans*-10,11-Dihydroxy-10,11-dihydrocarbamazepine; Carbamazepine 10,11-epoxide; **Carbamazepine**

Matrix Blood plasma

Other Materials Material: Oasis® HLB (30 mg, 1 ml) cartridges; LiChroCART® Purospher® Star (C<sub>18</sub>, 3 μm, 55 mm × 4 mm)

Method Category Active Pharmaceutical Ingredient and Metabolite Analysis

Technique Solid phase extraction; Reversed-phase HPLC

Equipment Used liquid chromatograph

Source **Development and validation of an HPLC-UV method for the simultaneous quantification of carbamazepine, oxcarbazepine, eslicarbazepine acetate and their main metabolites in human plasma**

Fortuna, Ana; Sousa, Joana; Alves, Gilberto; Falcao, Amílcar; Soares-da-Silva, Patricia

Analytical and Bioanalytical Chemistry (2010), 397 (4), 1605-1615. Springer

[Document Sources](#)

[Abstract](#) ▾

 <b>METHODS</b> <small>A CAS SOLUTION</small>		Preparation	Method	Linearity Range	Limit of Quantitation	Recovery		
<a href="#">Return to Results</a> <b>Compare Meth</b>	<b>Preparation of standa</b> 1. Prepare stock solutions of Phenobarbital, Primidone (PRM), Phenytoin (PHEN), Carbamazepine, Lamotrigine (LMO), Oxcarbazepine, Carbamazepine-10,11-epoxide (CBZ-E) 10 mg/mL, Licarbazepine (LIC) 10 mg/mL and IS standards individually by appropriate amount of compound in 1 mL of acetonitrile with methanol corresponding to the concentration of drugs and measure properly to afford spiking solution concentrations of 250, 100 and 20, 20, 50, 125, 500 and 1250 µg/mL for PRM, 5, 12.5, 50, 250 µg/mL for CBZ, LTG and 12.5, 25, 125 µg/mL for PHEN, 250 µg/mL for PHEN, 3.75, 7.5, 20, 100 µg/mL for LIC.  <a href="#">View Less ^</a>	<b>Solid phase extraction</b> 1. Add an aliquot of human plasma (500 µL) of 20 µL of the working solution and 1 mL of methanol or acetonitrile to precipitate plasma proteins. 2. After centrifuging at 1000 rpm for 10 min, evaporate the solvent, resulting supernatant is dried under gentle nitrogen stream for 10 min. 3. Dilute the residual volume of supernatant with 1.5 mL of acetonitrile and vortex-mix for 30 sec. 4. Subject the pre-treated sample to a solid-phase extraction on the Oasis® HLB (3 µm, 100 Å, Waters, Milford, MA, USA), which is previously conditioned with 1 mL of methanol, 1 mL of acetonitrile and 1 mL of water-acetonitrile (95:5, v/v). 5. Submit the loaded cartridge subsequently to -60 K for 4 hours and wash four times with water. 6. After drying the sorbent under airflow for 5 min, elute analytes with 1 mL of acetonitrile using gentle vortexing and ultrasound. 7. Evaporate the eluate under a gentle nitrogen gas and reconstitute with 500 µL of mobile phase. 8. Inject 20 µL of the final sample into the chromatography system. 9. Perform the chromatography using a 150 x 4.6 mm, 5 µm, C18 column.	0.40 - 50 µg/mL, Primidone, 0.10 - 50 µg/mL, Lamotrigine, 0.25 - 10 µg/mL, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine, 0.25 - 100 µg/mL, Phenobarbital, 0.15 - 80 µg/mL, Licarbazepine, 0.10 - 50 µg/mL, Carbamazepine-10,11-epoxide, 0.10 - 25 µg/mL, Oxcarbazepine, 0.50 - 50 µg/mL, Phenytoin, 0.10 - 50 µg/mL, Carbamazepine  0.40 µg/mL, Primidone, 0.10 µg/mL, Lamotrigine, 0.25 µg/mL, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine, 0.25 µg/mL, Phenobarbital, 0.15 µg/mL, Licarbazepine, 0.10 µg/mL, Carbamazepine-10,11-epoxide, 0.10 µg/mL, Oxcarbazepine, 0.50 µg/mL, Phenytoin, 0.10 µg/mL, Carbamazepine  92.24 ± 3.99%, 80.17 ± 2.84%, 94.47 ± 2.80% spiked in 1.2 µg/mL, 25 µg/mL, 45 µg/mL respectively, Primidone, 84.87 ± 5.89%, 84.16 ± 3.20%, 85.96 ± 2.41% spiked in 0.3 µg/mL, 25 µg/mL, 45 µg/mL respectively, Lamotrigine, 83.57 ± 3.94%, 83.13 ± 2.57%, 88.68 ± 2.48% spiked in 0.75 µg/mL, 5 µg/mL, 9 µg/mL respectively, 10,11- <i>trans</i> -Dihydroxy-10,11-dihydrocarbamazepine, 89.77 ± 4.77%, 81.35 ± 2.60%, 83.80 ± 2.60% spiked in 0.75 µg/mL, 50 µg/mL, 90 µg/mL respectively, Phenobarbital, 101.04 ± 3.83%, 87.45 ± 3.05%, 91.42 ± 2.67% spiked in 0.45 µg/mL, 40 µg/mL, 72 µg/mL respectively, Licarbazepine, 89.43 ± 6.02%, 85.27 ± 3.05%, 90.46 ± 2.67% spiked in 0.3 µg/mL, 25 µg/mL, 45 µg/mL respectively,	2.5-500 µg/L (Urine), 5.0-500 µg/L (Plasma)  0.10 µg/mL, trans-diol, 0.10 µg/mL, Lic, 0.10 µg/mL, CBZ-E, 0.05 µg/mL, OXC, 0.15 - 4.0 µg/mL, ESL, 0.05 - 30 µg/mL, CBZ  0.10 µg/mL, trans-diol, 0.10 µg/mL, Lic, 0.10 µg/mL, CBZ-E, 0.05 µg/mL, OXC, 0.15 µg/mL, ESL, 0.05 µg/mL, CBZ  96.0% in 5 µg/L spiked sample (Tap water), 91.0% in 20 µg/L spiked sample (Urine), 86.0% in 10 µg/L spiked sample (Plasma)  78.21 ± 4.18, 81.44 ± 5.61 and 78.44 ± 3.16% (CV 5.35, 6.88 and 4.03%) in 0.24, 5 and 9 µg/mL, trans-diol, 88.67 ± 8.31, 91.16 ± 6.15 and 86.02 ± 2.06% (CV 9.38, 6.74 and 2.39%) in 0.24, 30 and 54 µg/mL, Lic, 85.72 ± 2.34, 91.52 ± 6.05 and 86.08 ± 1.99% (CV 2.72, 6.61 and 2.31%) in 0.24, 15 and 27 µg/mL, CBZ-E, 84.34 ± 5.32, 91.03 ± 5.87 and 85.98 ± 1.80% (CV 6.31, 6.45 and 2.10%) in 0.14, 10 and 18 µg/mL, OXC, 73.77 ± 4.98, 84.76 ± 7.08, 79.54 ± 3.21% (CV 6.77, 8.35 and 4.03%) in 0.44, 2 and 3.6 µg/mL, ESL, 79.99 ± 2.82, 91.87 ± 4.90 and 85.72 ± 2.29% (CV 3.52, 5.32 and 2.67%) in 0.14, 15 and 27 µg/mL, CBZ  <a href="#">View Less ^</a>				
Title	CAS Method Number	Method Category	Technique	Analyte	Matrix	Other Materials	Equipment Used	Conditions
water:methanol:acetonitrile:TEA (68.7:25:6:0.3, v/v/v/v) adjust at pH 6.5		237 nm; mobile phase: mixture of water, acetonitrile and methanol						

## MethodsNow synthetic chemistry content sources

<b>Content from years</b>	<b>2000 - present</b>
Number of protocols	2.1 million; 3 million later this year
Content Coverage	Small molecule synthesis
Source Focus	180+ journals titles including new coverage from Wiley, RSC and Elsevier in addition to ACS, Springer, Taylor&Francis, WO patents (2010-present)
Example journal titles	Organic Letters, Catalysis Letters, Journal of Coordination Chemistry, Journal of Medicinal Chemistry, Journal of the American Chemical Society, Angewandte Chemie, Tetrahedron, Chemical Science
Language	English only

# MethodsNow – Synthetic Chemist Interface

The screenshot displays the MethodsNow interface. At the top, there are navigation options: "Get References" and "Tools". Below this, a search filter shows "Group by: No Grouping" and "Sort by: Relevance". A summary indicates "0 of 36 Reactions Selected". The main content area shows a reaction titled "1. View Reaction Detail" with a "Link" and "Similar Reactions" option. The reaction is labeled as a "Single Step" and includes a note: "Hover over any structure for more options." The reaction scheme shows the synthesis of a chromone derivative. The starting materials are 7-ethynyl-4-methoxycarbonylmethyl-2H-chromen-2-one and 1-azidodecane. The product is 7-(10-azidodecyl)-4-methoxycarbonylmethyl-2H-chromen-2-one, with a yield of 79%. Below the reaction, the "Overview" section is expanded to show the "METHODSNOW™ Procedure":

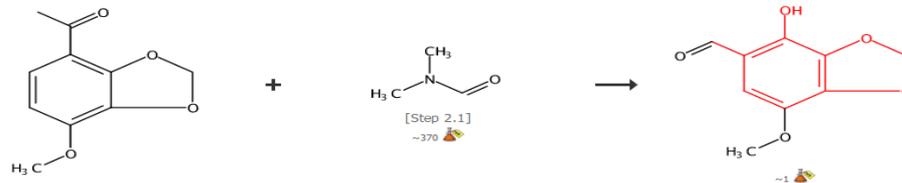
**Procedure**

1. Stir the mixture of 7-ethynyl-4-methoxycarbonylmethyl-2H-chromen-2-one (400 mg, 1.65 mmol), 1- azidoundecane (358 mg, 1.82 mmol), copper(II) sulfate pentahydrate (42 mg, 0.17 mmol), (+)-sodium L-ascorbate (360 mg, 1.82 mmol) in t-BuOH/water (15 mL/15 mL) at room temperature for 4 hours.
2. Add water to the mixture.

[View more...](#)

# SciFinder has the largest collection of experimental procedures for reactions

2 Steps Hover over any structure for more options.



## Overview

### Steps/Stages

- 1.1 R: Na<sub>2</sub>HPO<sub>4</sub>, R: mCPBA, S: CH<sub>2</sub>Cl<sub>2</sub>, cooled; 1 h, rt
- 1.2 R: KOH, S: MeOH, 2 h, rt
- 1.3 R: HCl, S: H<sub>2</sub>O, acidify
- 2.1 R: POCl<sub>3</sub>, S: DMF, 15 min, 5°C; 5°C → rt; 20 min, rt
- 2.2 rt; rt → 75°C; 2 h, 75°C; 75°C → 0°C
- 2.3 R: H<sub>2</sub>O, 5°C

### Notes

1) Baeyer-Villiger oxidation (stage 1), 2) regioselective, Vilsmeier reaction, Reactants: 2, Reagents: 6, Solvents: 4, Steps: 2, Stages: 6, Most stages in any one step: 3

### References

Total Synthesis of Bulbophylol-B

Quick View [Other Sources](#)

By Lin, Jinshun et al

From Journal of Natural Products, 71(11), 1938-1941; 2008

## Experimental Procedure



### Step 1

**4-Methoxy-2,3-methylenedioxyphenyl Acetate (8)**. To a suspension of **7** (5.0 g, 25.5 mmol) and anhydrous Na<sub>2</sub>HPO<sub>4</sub> (4.7 g, 33.2 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (50 mL) was added *m*-CPBA (85%, 23.4 g, 127.5 mmol), in portions and in an ice-water bath, and the mixture was stirred at room temperature for 1 h. The resulting mixture was refluxed overnight, then cooled and filtered. The filter cake was washed with CH<sub>2</sub>Cl<sub>2</sub> (3 × 30 mL). Evaporation of the solvent *in vacuo* gave a residue, which was directly used in the next reaction. **4-Methoxy-2,3-methylenedioxyphenol (9)**, KOH (1.4 g, 25 mmol) in H<sub>2</sub>O (10 mL) was added to the crude **8** (5.5 g, 25.8 mmol) in MeOH (20 mL), and the mixture was stirred for 2 h at room temperature. The mixture was concentrated to 10 mL and acidified with 2 M HCl (5 mL). The aqueous layer was extracted with CHCl<sub>3</sub> (3 × 20 mL), washed with H<sub>2</sub>O (2 × 20 mL) and brine (20 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The residue was purified by column chromatography (CC) (*n*-hexane/EtOAc, 3:1) to give **9** (4.38 g); two steps total yield 78%) as a white solid: **4-Methoxy-2,3-methylenedioxyphenol (9)**, yield 4.38 g, 78% mp 103-105 °C (lit.<sup>12</sup> mp 100-101 °C); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 6.43 (1H, s, H-6), 6.42 (1H, s, H-5), 5.99 (2H, s, OC<sub>2</sub>H<sub>4</sub>O), 4.48 (1H, s, OH), 3.85 (1H, s, OC<sub>2</sub>H<sub>5</sub>).

### Step 2

**2-Hydroxy-3,4-methylenedioxy-5-methoxybenzaldehyde (10)**. POCl<sub>3</sub> (5.5 mL, 59.5 mmol) was added dropwise to DMF (10 mL, 129.4 mmol) over 15 min at 5 °C, then stirred at room temperature for 20 min followed by addition of **9** (2.5 g, 14.9 mmol) in portions. The mixture was slowly heated to 75 °C and then stirred at this temperature for 2 h. The resulting mixture was cooled to 5 °C and poured into H<sub>2</sub>O (50 mL). After filtration, the filter cake was purified by CC (*n*-hexane/CHCl<sub>3</sub>, 1:1) to give **10** (2.3 g, 79%) as a white solid: **2-Hydroxy-3,4-methylenedioxy-5-methoxybenzaldehyde (10)**, yield 2.3 g, 79% mp 181-182 °C (lit.<sup>11</sup> mp 179-180 °C); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 10.83 (1H, s, CHO), 9.73 (1H, s, OH), 6.75 (1H, s, Ar-H), 6.18 (2H, s, OCH<sub>2</sub>O), 3.93 (3H, s, OCH<sub>3</sub>).

It is our most popular feature, but you've told us we can do more



SciFinder®

A CAS SOLUTION

Analyze Refine

Group by: No Grouping Sort by: MethodsNow

Analyze by: MethodsNow

MethodsNow Available 26

MethodsNow Not Available 10

Show More

0 of 36 Reactions Selected

1. View Reaction Detail Link

4 Steps Hover over any structure for more options.

[Step 2.1] ~59

[Step 4.1] ~145

Overview

METHODSNow™

Procedure

1. Add lithium hydroxide monohydrate(327 mg, 7.80 mmol) to 4-methoxycarbonylmethyl-7-(1-undecyl-1H- 1,2,3-triazol-4-yl)-2H-chromen-2-one (343 mg, 0

## MethodsNow

### 7-Triazolylcoumarin-based fluorescent tag system for stepwise, comparative assessment of small molecule microarrays

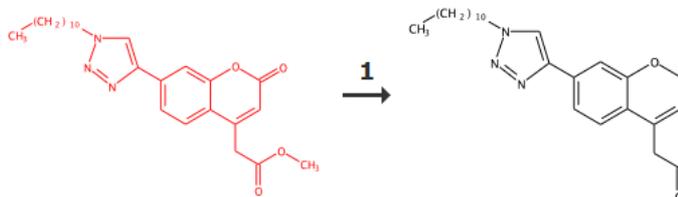
By Jeon, Moon-Kook; Kang, Myoung-Ku; Park, Koon Ha

From Tetrahedron, 68(30), 6038-6053; 2012

Published by Elsevier Ltd.

#### Reaction Steps

1 2 3 4



<b>Products</b>	2H-1-Benzopyran-4-acetic acid, 2-oxo-7-(1-undecyl-1H-1,2,3-triazol-4-yl)-1,2,3-triazolo[4,5-b]pyridin-4(1H)-one, CAS RN: 1384966-77-1
<b>Reactants</b>	2H-1-Benzopyran-4-acetic acid, 2-oxo-7-(1-undecyl-1H-1,2,3-triazol-4-yl)-1,2,3-triazolo[4,5-b]pyridin-4(1H)-one, CAS RN: 1384966-75-9
<b>Reagents</b>	Hydrochloric acid, CAS RN: 7647-01-0 Lithium hydroxide, CAS RN: 1310-65-2
<b>Solvents</b>	Water, CAS RN: 7732-18-5 Tetrahydrofuran, CAS RN: 109-99-9

## MethodsNow

### Procedure

1. Add lithium hydroxide monohydrate(327 mg, 7.80 mmol) to 4-methoxycarbonylmethyl-7-(1-undecyl-1H-1,2,3-triazol-4-yl)-2H-chromen-2-one (343 mg, 0.780 mmol) in THF/water(25 mL/25 mL) at room temperature.
2. Stir the reaction mixture for 3 hours at room temperature.
3. Adjust pH 3-4 to the reaction mixture by adding 1 N hydrochloric acid.
4. Partition the reaction mixture between ethyl acetate and water.
5. Extract the aqueous layer with ethyl acetate.
6. Dry the combined organic layer over magnesium sulfate.

### Scale

milligram

### <sup>1</sup>H NMR

<sup>1</sup>H NMR (300 MHz, acetone- d<sub>6</sub>): δ = 7.83 (s, 1H), 8.58 (s, 1H), 7.92 (d, J = 8.1 Hz, 1H), 7.84 (d, J = 8.1 Hz, 1H), 6.47 (s, 1H), 4.50 (t, J = 7.2 Hz, 2H), 3.99 (s, 2H), 2.00 (quintet, J = 7.2 Hz, 2H), 1.32-1.43 (m, 4H), 1.22-1.32 (m, 12H), 0.87 ppm (t, J = 6.8 Hz, 3H).

### <sup>13</sup>C NMR

<sup>13</sup>C NMR (125 MHz, DMF-d<sub>7</sub>, 60 °C): δ = 161.0, 155.1, 154.2, 146.5, 136.0, 127.2, 123.7, 122.1, 120.4, 115.3, 113.5, 51.1, 32.8, 29.9, 27.3, 23.5, 18.7, 14.7 ppm (decarboxylation occurred to give the corresponding 4-methyl derivative).

### IR

IR (ATR, neat): ν = 3423, 2922, 2851, 1702 (2C=O, overlapped), 1619, 1561, 1375, 1154, 936, 852, 809 cm<sup>-1</sup>.

### HRMS

HRMS (EI): m/z calculated for C<sub>24</sub>H<sub>31</sub>N<sub>3</sub>O<sub>4</sub>: 425.2315 [M<sup>+</sup>]; found: 425.2315.

### Mass Spec

MS (ESI): m/z: 426 [M+H<sup>+</sup>].

### MP

235.5±0.8 °C.

### CAS Method Number

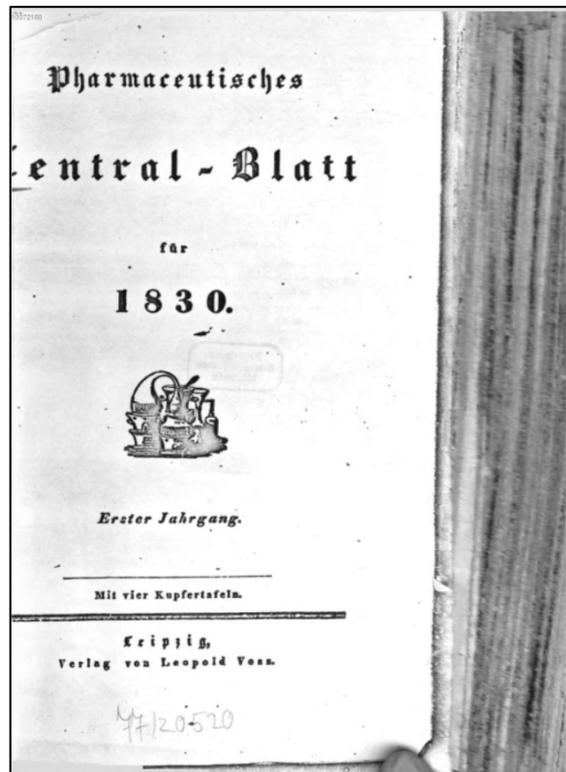
3-352-CAS-78415

Print/Export

Close

# Chemisches Zentralblatt predates the introduction of Chemical Abstracts by almost 80 years

- Founded in 1830 to abstract chemistry-related literature
- The only abstract journal until 1907
- Published from 1830-1969
- Chronicles the birth of chemistry as a science
  - Before 1800, chemistry was more alchemy (i.e. how to turn lead into gold) than actual science



First Issue:  
Note, the name changed several times before 1856



## Why is Chemisches Zentralblatt so important?

- It covers the literature from an historically important era for chemistry
- Many authors covered by this publication formulated the theories and hypotheses that shaped the foundation of today's chemistry

- Mortiz Traube
  - alcohol fermentation by enzymes
- Louis Pasteur
  - microbial fermentation
- Niels Bohr
  - Bohr model
- Albert Einstein
  - theory of relativity
- Eilhard Mitscherling
  - selenic acid
- Heinrich Rose
  - Rediscovered, named Niobium
- Ernest Rutherford
  - alpha and beta radioactivity

## ChemZent™ in SciFinder® makes this literature newly discoverable

- Covers chemical literature from 1830 to 1969
  - The first and oldest abstracts journal in chemistry (>3 million abstracts)
  - The only comprehensive abstract journal available until 1907
- German is translated to and searchable in English
  - Substances and concepts are indexed from the German abstracts
  - Structures and keywords are searchable
  - Markers at the start of German abstracts can be displayed and printed
- Now integrated into SciFinder and available for purchase
  - Completes and extends the comprehensive content in SciFinder
  - Provides easy access to a valuable chemistry collection via a familiar CAS solution

# ChemZent is available via subscription in SciFinder

The screenshot displays the SciFinder web interface. At the top, there is a navigation bar with 'CAS Solutions' and 'SciFinder A CAS SOLUTION' logos, along with 'Preferences', 'SciFinder Help', and 'Sign Out' links. Below this is a secondary bar with 'Explore', 'Saved Searches', and 'SciPlanner' options, and 'Save', 'Print', and 'Export' buttons. The main content area shows a search for 'cholesterol' refined to 'CHEMZENT' (3410). A 'REFERENCES' section is active, showing 0 of 555655 references selected. Three results are visible:

- 1. Compounds containing the provitamin Dz are related. 4. Part 3-Methyl-cholesterin and the corresponding provitamin (3-methyl provitamin Dz)**  
By Strating, J.  
From *Chemisches Zentralblatt* (1955), 126(47), 10999-11000. | Language: German, Database: CHEMZENT  
Original Source *Recueil Trav. chim. Pays-Bas* 71, 822-30, Aug. 1952. Groningen, Univ., Labor, of Organ. Chem.  
(3, see *Chemisches Zentralblatt* 1952, 2180.) By effect of CH<sub>3</sub>MgI on ZIS-Cholestenon- (3) is 3-methyl **cholesterol** (I) represented, the suspected of the same pattern. Configuration (Z $\delta$ ) such as **cholesterol** itself, since it also a Digitonid forms. By bromination of the I-Benzoats with Brom-succinimid and subsequent HBr-Abspaltung was 7-dehydro-3-methyl **cholesterol** (II) -benzoat (3-Methylprovitamin-Dz-benzoat) obtained by treatment with LiAlH<sub>4</sub> II was produced. Irradiated II in rat test only approximately 1/80 the effect of vitamin D<sub>3</sub>. — Bestrahtes 7-dehydro thio **cholesterol** (cf. STRATING and baka...
- 2. Hypocholesterolemie agents. 2. Part 2SS-halogen-5 <x>-androstane**  
By Counsell, P. D. Kilmstraund R. E.  
From *Chemisches Zentralblatt* (1954-), 135(1), 120-121. | Language: German, Database: CHEMZENT  
Original Source *J. med. pharmaco. Chem.* 5, 1216-24, 1962; Chicago, Ill., G. D. Searle & Co., Div. of Chem. Res.; engl.  
Starting from 2 <x>.3 <x>-epoxy-5a-androstan-17-one (I) Synthesized authors 2SS-fluorine (II), 2SS-chloro-(III), 2SS-bromine (IV) and 2- $\delta$ -iodine-3TX-hydroxy-5 <x>-androstan-17-one (V). The pivoting, II from I by reaction with BF<sub>3</sub> etherate in benzene/Ae. to obtain Mainly led to 2-0-[2 <x>-fluorine-17-oxo-5- $\delta$ -androstan-3 $\beta$ -yl]-2 $\delta$ .3ix-dihydroxy-5 $\delta$ -androstan-17-one (C<sub>3</sub>H<sub>6</sub>F<sub>2</sub>O<sub>4</sub>, Melting Point 267-269 °; [a] D<sub>24</sub> -91 °). By reduction with Al-tert-butoxide. LiH Revealed II-V the CORRESPONDING 2SS-halogen-5 <x>-androstan-3 (x,17 $\delta$ -diols; Reducers Verse, Na<sup>+</sup> B<sup>4</sup>H led in III to give 2- $\delta$ -chloro-5 <x>-androstan-3a...
- 3. The effect of o, p<sup>-</sup>DDD therapy on plasma cholesterol in addition to renal carcinoma**  
By Molnar, George D.; Nunn, Stewart L.; Tauxe, W. Newlon  
From *Chemisches Zentralblatt* (1964), 135(1), 136. | Language: German, Database: CHEMZENT  
Original Source *Proc. Staff Meetings Mayo Clin.* 36, 618 - 20, 22/11.1961; Rochester, Minn., Mayo Clinic, Section of Med. u. Clin. Pathol.; engl.  
At 4 of 7 patients in kidney of -carcinomen be during treatment with o, p<sup>-</sup>DDD (2-[2' -chlorophenyl -2]-1<sup>-</sup>chlorophenylV\ -1.1-dichloro-ethane) to increase in **Cholesterol** Determined. The patients hypercholesterolemia occurred were against the DDD-Therapie resistant. Experiments with 14C-acetate in patients thesis Showed That in the former group is propagated 14C-**cholesterol** in the plasma to find what, in the other not.

Explore | Saved Searches | SciPlanner

Research Topic  
  
 A CAS SOLUTION

REFERENCES  
 Explore | Saved Searches | SciPlanner

Research Topic "cholesterol" > references (555655) > refine "CHEMZENT" (3410) > Partial synthesis of chole

REFERENCE DETAIL | Get Substances | Get Related Citations | View with CHEMZENT

Return

## 2. Partial synthesis of cholesterol (Partialsynthese von Cholesterin)

By: Kessar, S. V.; Rampal, A. L.; Mangat, S.; Gupta, Y. P.

Michael-Addition of 1-Nitro-4-methylpentane of *cis*-3/3-Hydroxy-16-oxo- $\Delta^5$ -17-pregnadiene provides 3 $\beta$ -Hydroxy-reduction with hydrazine hydrate is **cholesterol**. Attempts: 1-nitro-4-methylpentane, C<sub>8</sub>H<sub>13</sub>NO<sub>2</sub>, Kp.40 98–100 hydroxy 20-oxo- $\Delta^5$ , 6-diene [of Huang-Minlon and Ching-Tungshun, Tetrahedron letters [London] 1961, 667]; 70 mg. — 3 $\beta$ -Hydroxy-16,22-dioxo-Ab-cholesten, C<sub>27</sub>H<sub>42</sub>O<sub>3</sub>, melting point 154–157° (acetic ester/Pae.) from hydrochloride in pyridine. — **cholesterol**, melting point 144–146° (acetic ester), from the g above cpd., Zn-Am

### Indexing

#### Concepts

amine melting point

### Tags

0 Tags | Edit Tags

### Comments

From Chemisches Zentralblatt (1969), 140(49/50), 140. | Language: German, Database: CHEMZENT  
 Original Source Chem. pharmac. Bull. [Tokyo] 16 (1968) 11, 2123–29; Nanakuma, Fukuoka, Fukuoka Univ., Fac.

By gas chromatography, the free steroids and triterpenoids and as methyl ester of the acid stigmasterol, A1-u. A22-stigmasterol, ot-Spinasterin, 8-Sito-sterin, cc-u. 8-Amyrin, lupeol, Me

5. Black lipid films. A new type of Grenz-flächenadsorptions-Phänomenen

Q Quick View CHEMZENT

By Tien, H. T.; Coone, S.; Dawidowicz, E. A.

Nr. 51/52-1339

C-4. Naturstoffe

1969

**15 $\beta$ -Hydroxydigitoxigenin** [C<sub>28</sub>H<sub>44</sub>O<sub>6</sub>, F. 245–247°]. Die **Acetylierung** von **B** führt zu **15 $\alpha$ -Hydroxy-14 $\alpha$ -digitoxigenin**, Hydrat [I, C<sub>28</sub>H<sub>44</sub>O<sub>6</sub>·H<sub>2</sub>O, F. 130–140°; [a]<sub>D</sub><sup>25</sup> +31.5° (CHCl<sub>3</sub>)]. **Acetylierung** von **B** bzw. **I** liefert **14 $\alpha$ -Digitoxigenin**, **3 $\beta$ -15 $\alpha$ -O-O-Diacetylderiv.** [C<sub>27</sub>·H<sub>42</sub>O<sub>6</sub>, F. 232–234°; [a]<sub>D</sub><sup>25</sup> +38,3° (CHCl<sub>3</sub>)]. Saure Hydrolyse von **II** gibt **15-Oxiddigitoxigenin** [C<sub>28</sub>H<sub>42</sub>O<sub>6</sub>, F. 215–217°; [a]<sub>D</sub><sup>25</sup> +12,7° (CHCl<sub>3</sub>)]. — IR- u. UV-Maxima vgl. Original. K.-P. Hilgetag 3650 Δ

**1339 Heteroc. sche Steroide**, 10. Mitt. **2-Aza-4-Oxasteroid-Analo** D. M. Piatak und E. Caspi. (J. heterocyclic Chem. 1, 4 (1967) 1, 8–11; Worcester Found. for Experim. l. l., Shrewsbury, Mass.; engl.) — 9. vgl. J. org. Chemistry **81** (1966) 4225. — **VI**, synthetisieren aus **5 $\beta$ -Acetyl-1.5-seco-2.3.4-tris-noröstran-17 $\beta$ -ol-1-säure** eine Reihe von **2-Aza-4-oxa-Steroiden**.

**Versuche:** 1.5-Seco-2.3.4-trianoröstran-17 $\beta$ -nitrat-1-säuren: **5 $\beta$ -Hydroxy-**, C<sub>18</sub>H<sub>22</sub>NO<sub>6</sub>, F. 206 bis 208° (Methanol/CHCl<sub>3</sub>), durch Kochen von 370 mg **5 $\beta$ -Acetoxy-1.5-seco-2.3.4-tris-noröstran-17 $\beta$ -nitrat-1-säuremethylster** mit 2 n NaOH; 315 mg (Ausb.). — **5 $\beta$ -Acetoxy (II)**, C<sub>17</sub>H<sub>22</sub>NO<sub>6</sub>, F. 195–199° (Essigester/Pentan), durch Acetylieren von 300 mg vorst. Verb. mit Acetanhydrid/Py.; 300 mg. — **5 $\beta$ -Acetoxy, Amid**, C<sub>17</sub>H<sub>22</sub>N<sub>2</sub>O<sub>6</sub>, F. 176–178° (Aceton/Pentan), durch Kochen von 100 mg vorst. Verb. mit Oxalylchlorid u. Rk. in CHCl<sub>3</sub> mit NH<sub>3</sub>; 70 mg. — **5 $\beta$ -Hydroxy-, Amid**, C<sub>17</sub>H<sub>24</sub>N<sub>2</sub>O<sub>6</sub>, F. 202–204°, durch Kochen vorst. Verb. mit 2 n methanol. NaOH. — **Östrane: 2-Aza-5 $\beta$ -hydroxy-acetoxy-2.5-seco-3.4-bienor-**, C<sub>18</sub>H<sub>23</sub>NO<sub>6</sub>, F. 182–191° (Essigester), durch Zugabe von LiAlH<sub>4</sub> zu 200 mg vorvorst. Verb. in THF u. Acetylieren des erhaltenen **2-Aza-5 $\beta$ -17 $\beta$ -dihydroxy-2.5-seco-3.4-bisnoröstrans (I)**; 180 mg. — **2-Aza-3-oxo-4-oxo-17 $\beta$ -acetoxy-5 $\alpha$ -**, C<sub>18</sub>H<sub>27</sub>NO<sub>6</sub>, F. 219 bis 222° (Essigester/Pentan), durch Zugabe von Phosgen in Bzl. zu 210 mg **I** in CHCl<sub>3</sub>/abs. Py. u. Acetylieren; 55 mg. — **2-Aza-2-acetyl-4-oxo-17 $\beta$ -acetoxy-5 $\alpha$ -**, C<sub>20</sub>H<sub>31</sub>NO<sub>6</sub>, F. 144–147° (Essigester/Pentan), durch Kochen von 100 mg **I** u. Paraformaldehyd in Bzl. unter Abdest. von W. u. Acetylieren; 55 mg. — **1-Oxo-2-aza-5 $\beta$ -hydroxy-4-nor-3.5-seco-, 17 $\beta$ -Nitrat**, C<sub>18</sub>H<sub>26</sub>N<sub>2</sub>O<sub>6</sub>, F. 160 bis 166° (Aceton-Dioxan), durch Dk. von 500 mg **II** mit

**Versuche:** **Cholestane: 3 $\beta$ -Hydroxy-6-[cyanmethyl]-**, C<sub>28</sub>H<sub>44</sub>NO, F. 83–85° (Methanol), aus Na, **I** u. **3 $\beta$ -Hydroxacholestan-6-on**; [a]<sub>D</sub><sup>22</sup> –24° (c = 3,5; (CHCl<sub>3</sub>); 51% (Ausb.). — **3 $\beta$ -Hydroxy-7-[cyanmethyl]-**, C<sub>28</sub>H<sub>44</sub>NO, F. 100–102° (Methanol), aus 2 g **3 $\beta$ -Hydroxycholestan-7-on analog vorst.**; [a]<sub>D</sub><sup>22</sup> –38° (c = 1,62; CHCl<sub>3</sub>); 1,85 g. — **3 $\beta$ -Acetoxy-7-[cyanmethyl]-**, C<sub>28</sub>H<sub>44</sub>NO<sub>2</sub>, F. 108–109° (Methanol), aus 500 mg vorst. Verb. u. Acetanhydrid/Py.; [a]<sub>D</sub><sup>22</sup> –12,7° (c = 4,07; CHCl<sub>3</sub>); 534 mg. — **3.6-Bis-[cyanmethyl]-**, aus Cholestan-3.6-dion u. **I**. — **3.7-Bis-[cyanmethyl]-**, C<sub>28</sub>H<sub>42</sub>N<sub>2</sub>, F. 162 bis 164° (Methanol), aus 0,44 g Cholestan-2.7-dion u. 1,28 g **I**; [a]<sub>D</sub><sup>22</sup> –318° (c = 2,47; CHCl<sub>3</sub>); 0,45 g. — **2-[Cyanmethyl]-**, C<sub>28</sub>H<sub>42</sub>N, aus Cholestan-2-on; 72%. — **3.7.12-Tris-[cyanmethyl]-cholansäuremethylster**, C<sub>28</sub>H<sub>42</sub>N<sub>3</sub>O<sub>2</sub>, F. 232–234° (Methanol), aus Dehydrocholansäuremethylster u. **I**; [a]<sub>D</sub><sup>22</sup> +64° (c = 2,9; CHCl<sub>3</sub>); 56%. — **17 $\beta$ -Acetoxy-4-[cyanmethyl]-androstan**, C<sub>22</sub>·H<sub>32</sub>NO<sub>6</sub>, F. 140–144° (Methanol), aus 17 $\beta$ -Androstan-4-on u. **I**; [a]<sub>D</sub><sup>22</sup> +11,5° (c = 2,11; CHCl<sub>3</sub>); 72%. — **3-[Cyanmethyl]-17 $\alpha$ .20;20.21-bis-methylenedioxy- $\Delta^4$ -pregnen-4-on**, C<sub>22</sub>H<sub>34</sub>NO<sub>6</sub>, F. 280–282° (Aceton/Hexan), aus 17 $\alpha$ .20;20.21-Bis-methylenedioxykortison u. **I**; [a]<sub>D</sub><sup>22</sup> +140° (c = 0,4; CHCl<sub>3</sub>); 97%. — **3-[Cyanmethyl]- $\Delta^4$ .21-lanostadien**, C<sub>28</sub>H<sub>42</sub>N, F. 115–116° (99% (A.)), aus 1,4 g  $\Delta^4$ .21-lanostadien-3-on u. **I**; [a]<sub>D</sub><sup>22</sup> +26° (c = 2,32; CHCl<sub>3</sub>); 0,42 g. K.-P. Hilgetag 3700 Δ

**1341 Partialsynthese von Cholesterin**, S. V. Kessar, A. L. Rampal, S. Mangat und Y. P. Gupta. (Indian J. Chem. 4 (1967) 2, 501–03; Dept. of Chem., Panjab Univ., Chandigarh; engl.) — **Michael-Addition** von 1-Nitro-4-methylpentan an *cis*-3 $\beta$ -Hydroxy-16-oxo- $\Delta^5$ -17-pregnadien liefert **3 $\beta$ -Hydroxy-16-oxo-22-nitro- $\Delta^5$ -cholesten**, das durch Nel-Rk. in **3 $\beta$ -Hydroxy-16.11-dioxo- $\Delta^5$ -cholesten** übergeführt wird. Nachfolgende Red. mit Zn-Amalgam u. HCl sowie weitere Red. mit Hydrazinhydrat gibt **Cholesterin**.

**Versuche:** **1-Nitro-4-methylpentan**, C<sub>8</sub>H<sub>13</sub>NO<sub>2</sub>, Kp. 98–100°, aus 10,6 g Isohexyljodid, **AgNO<sub>2</sub>**, abs. Ae. bei 0° unter Lichtausschluss; 4 g (Ausb.). — ***cis*-3 $\beta$ -Hydroxy-16-oxo- $\Delta^5$ -17-pregnadien**, F. 171–172°, aus 3 $\beta$ -Hydroxy-20-oxo- $\Delta^5$ -16-pregnadien [nach Huang-Minlon, China. Chem. Pharm. Sin. 1962, 3, 100].



## Veli-Pekka Hyttinen

Regional Marketing Manager  
Central and Eastern Europe

[vhyttinen@cas.org](mailto:vhyttinen@cas.org)

## Tetiana Khristova

Regional Marketing Manager  
Ukraine

[tkhristova@acsi.info](mailto:tkhristova@acsi.info)

Connect with SciFinder



*Thank you !!!*