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Vol 7, Issue 3, 2015

TABLE OF CONTENTS

Review Article(s)

THE APPLICATION OF COMMUNICATION TECHNOLOGY IN HOSPITAL AND COMMUNITY PHARMACIES: A BRIEF REVIEW 1-5

Pei Lin Lua, Umar Idris Ibrahim

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [559 Downloads] 259 Views

ROLE OF GREEN TEA AND ITS CONSTITUENT EPIGALLOCATECHIN-3-GALLATE IN THE HEALTH MANAGEMENT 6-12

Arshad H. Rahmani, Yousef H Aldebasi, Salah M Aly

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [401 Downloads] 509 Views


CURRENT TREATMENTS FOR TYPE 2 DIABETES, THEIR SIDE EFFECTS AND POSSIBLE COMPLEMENTARY TREATMENTS 13-18

Arshpreet Kalsi, Shivangi Singh, Nancy Taneja, Samiksha Kukal, Shalini Mani

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [305 Downloads] 365 Views

SULFONES: AN IMPORTANT CLASS OF ORGANIC COMPOUNDS WITH DIVERSE BIOLOGICAL ACTIVITIES 19-27

Irshad Ahmad, Shagufta .

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [408 Downloads] 404 Views

PROMISING NEUROPROTECTIVE PLANTS FROM NORTH-EAST INDIA 28-39

Pankaj Phukan, Meenakshi Bawari, Mahuya Sengupta

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [476 Downloads] 1467 Views

CANCER NANOTECHNOLOGY: NANOPARTICULATE DRUG DELIVERY FOR THE TREATMENT OF CANCER 40-46

Ksy Hemant, Abhay Raizaday, Praveen Sivadasu, Swati Uniyal, S Hemanth Kumar

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [296 Downloads] 273 Views

Original Article(s)

SCREENING OF ANTIBACTERIAL AND ANTIFONGICAL ACTIVITY IN MARINE MACROALGAE AND MAGNOLIOPHYTEA FROM THE COAST OF TUNISIA 47-51

Rihab Ben Abdallah Kolsi, Doneyez Frikha, Imed Jribi, Asma Hamza, Lotfi Fekih, Karima Belgith

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [399 Downloads] 463 Views

DEVELOPMENT AND EVALUATION OF DENTAL FILMS CONTAINING AN ANTIBACTERIAL AGENT FOR THE TREATMENT OF PERIODONTITIS 52-59

Vineetha V C, Manju Maria Mathews

[Abstract](#) || [View PDF](#) ||  [Download PDF](#) [320 Downloads] 322 Views

CYTOTOXICITY STUDIES OF TETRAPRELYLTOLUQUINONE, A PRENILATED HYDROQUINONE FROM GARCINA COWA ROXB ON H-460, MCF-7 AND DU-145 60-63

Fatma Sri Wahyuni, Johnson Stanslas, Nordin Hj Lajis, Dachriyanus Hamidi	221 Views
Abstract View PDF Download PDF [270 Downloads]	
PHYSICOCHEMICAL CHARACTERIZATION AND FLAVONOID CONTENTS OF ARTISANAL BRAZILIAN GREEN PROPOLIS	64-68
Flávio Júnior Barbosa Figueiredo, Marcus Vinícius Dias-Souza, Evandro Afonso Nascimento, Leonardo Ramos Paes De Lima	400 Views
Abstract View PDF Download PDF [258 Downloads]	
EVALUATION OF ANXIOLYTIC ACTIVITY OF METHANOL EXTRACT OF TRACHYSPERMUM AMMI L	69-71
Muhammad A. Rajput, Rafeeq A. Khan, Zeeshan Feroz	246 Views
Abstract View PDF Download PDF [266 Downloads]	
CONDUCTOMETRIC DETERMINATION OF THE ANTIHISTAMINIC DIPHENHYDRAMINE HYDROCHLORIDE USING SILVER NITRATE AS A TITRANT	72-76
Mohamed Al Bratty, Hisham Hashem, Amal Noureldeen, Gunasekar Manoharan, Fawaz Towhari	308 Views
Abstract View PDF Download PDF [487 Downloads]	
FORMULATION AND STORAGE STABILITY OF COCONUT FLOUR AND DIETARY FIBRE ISOLATE	77-81
Manikandan Arumugam, Meera Raman, Kannan Eagappan	268 Views
Abstract View PDF Download PDF [257 Downloads]	
EVALUATION AND PHARMACIST'S INTERVENTION FOR IMPROVING ADHERENCE AMONG RENAL FAILURE PATIENTS	82-85
Venkateswararao S, Asha Sara Stephen, Kshama Indoria, Rama P	182 Views
Abstract View PDF Download PDF [332 Downloads]	
XANTHINE OXIDASE INHIBITORY AND ANTIHYPERURICEMIC ACTIVITIES OF ANREDERA CORDIFOLIA (TEN) STEENIS, SONCHUS ARVENSIS L, AND ITS COMBINATION	86-90
Kurnia Dwi Widayari, Elin Yulinah Sukandar, Irda Fidrianny	347 Views
Abstract View PDF Download PDF [321 Downloads]	
AN ORAL FEED STRATEGY IN SHEEP FOR IMPROVED DIGESTIBILITY, NUTRIENT UTILIZATION AND METHANE MITIGATION	91-94
P. K. Pankaj, D. B. V. Ramana, Ch. Srinivasa Rao, K. Ravikanth, Rita Rani, M. Nikhila, Adarsh Chaudhary	195 Views
Abstract View PDF Download PDF [172 Downloads]	
IN VITRO ANTIOXIDANT AND ANTICANCER POTENTIAL OF FLOWERS OF TODDALIA ASIATICA (RUTACEAE)	95-99
Krithiga Thangavelu, Narayanan Ravisankar, Abubakker Siddiq, Jerrine Joseph	178 Views
Abstract View PDF Download PDF [294 Downloads]	
EFFECT OF CRUDE SULPHATED POLYSACCHARIDE FROM MARINE BROWN ALGAE IN TPA INDUCED INFLAMMATION ON POLY MORPHONUCLEAR LEUKOCYTES	100-102
Lavanya R, Seethalakshmi S, Gopal V, Chamundeeswari D	110 Views
Abstract View PDF Download PDF [187 Downloads]	
INTERACTION BETWEEN CHOLINERGIC AND OPIOID SYSTEMS IN RELAPSE TO ALCOHOL DEPENDENCE	103-106
Kusnandar Anggadiredja, Julianri Sari Lebang	

Abstract View PDF Download PDF [167 Downloads]	97 Views
INVESTIGATION OF BIOGENIC SILVER NANOPARTICLES GREEN SYNTHESIZED FROM CARICA PAPAYA	107-110
Reshma S Konjari, Anju A Jacob, S Jayanthi, C Ramalingam, Anita Sagadevan Ethiraj	
Abstract View PDF Download PDF [312 Downloads]	559 Views
STANDARDIZATION OF FRIABLE CALLUS DEVELOPMENT IN CATHARANTHUS ROSEUS (LINN.) G. DON	111-113
Sairam Veerabathini, Sarang S, Shalini S, Deepa Sankar P	
Abstract View PDF Download PDF [262 Downloads]	398 Views
IMPACTS OF COUNSELING ON ADHERENCE TO PRESCRIBED MEDICATIONS AND BLOOD PRESSURE OF HYPERTENSIVE PATIENTS IN FOUR INDONESIAN PRIMARY HEALTH CENTERS	114-117
Nasution A, Khairunnisa ., Tanjung H R	
Abstract View PDF Download PDF [215 Downloads]	219 Views
EVALUATION OF ANTI-THYROID ACTIVITY OF FICUS RACEMOSA LINN BARK IN MALE RATS	118-122
Mohammed Azharuddin, Mohammed Atif, Mohammed Ibrahim Ahmed, Syed Asadulla Bakhtiyari, Mohammed Ibrahim	
Abstract View PDF Download PDF [214 Downloads]	152 Views
MOLECULAR BEACON PROBE BASED PROMOTER MOTIFS VALIDATION IN ANOXIA RESPONSIVE DIFFERENTIALLY EXPRESSED GENES AND THEIR IN SILICO INTERACTION STUDIES WITH AP2/EREBP TF IN RICE (ORYZA SATIVA L.)	123-130
Gopal Kumar Prajapati, Dev Mani Pandey	
Abstract View PDF Download PDF [178 Downloads]	276 Views
ANTIMICROBIAL ACTIVITY OF OLIGOMERIC B-DEFENSIN ISOLATED FROM HEART TISSUE OF CAPRA HIRCUS	131-135
Devjani Banerjee, Tejas Gohil, Sunil Trivedi	
Abstract View PDF Download PDF [162 Downloads]	63 Views
INFLUENCE OF CARBON AND NITROGEN SOURCE ON GROWTH, DON AND NIV PRODUCTION BY TWO SPECIES OF FUSARIUM ISOLATED FROM FINGER MILLETS	136-139
P. Shilpa, V. Koteswara Rao, K. Narasimha Rao, S. Girisham, S. M. Reddy	
Abstract View PDF Download PDF [167 Downloads]	128 Views
CHARACTERIZATION OF POLYHYDROXYBUTYRATE SYNTHESIZED BY BACILLUS CEREUS	140-144
A Brinda Devi, C. Valli Nachiyar, T. Kaviyarasi, Antony V. Samrot	
Abstract View PDF Download PDF [271 Downloads]	195 Views
EFFECT OF VISCOSITY, SURFACTANT TYPE AND CONCENTRATION ON PHYSICOCHEMICAL PROPERTIES OF SOLID LIPID NANOPARTICLES	145-153
Mamdouh Ghorab, Ahmad Gardouh, Shadeed Gad	
Abstract View PDF Download PDF [233 Downloads]	326 Views
ANTIDIABETIC EFFECT AND PHYTOCHEMICAL SCREENING OF ETHANOLIC EXTRACT OF POLYALTHIA CERASOIDES STEM BARK IN STREPTOZOTOCIN INDUCED DIABETIC ALBINO RATS	154-158
Bhargavi G, Josthna P, Naidu Cv	

Abstract View PDF Download PDF [267 Downloads]	280 Views
DEVELOPMENT OF CULTIVATION MEDIUM FOR HIGH YIELD KEFIRAN PRODUCTION BY LACTOBACILLUS KEFIRANOFACIENS	159-163
Daniel J. Dailin, Elsayed A. Elsayed, Nor Zalina Othman, Roslinda Abd Malek, Solleh Ramli, Mohamad R. Sarmidi, Ramlan Aziz, Mohamad A. Wadaan, Hesham A. Elenshasy	
Abstract View PDF Download PDF [199 Downloads]	263 Views
BIO-POTENTIATION AND IN VIVO PHARMACOKINETICS STUDIES OF POLYPHENOLS FROM PUNICA GRANATUM BY FORMING PHOSPHOLIPID CONJUGATES FOR BETTER ANTIOXIDANT & ANTI-INFLAMMATORY ACTIVITY	164-172
Deep Bhattacharya, Priscilla M D'mello, Tarunkumar Dasgupta	
Abstract View PDF Download PDF [150 Downloads]	111 Views
INVESTIGATION OF ANALGESIC ACTIVITY OF XANTHINE OXIDASE INHIBITOR ALLOPURINOL: AN EXPERIMENTAL STUDY	173-176
Pise H N, Padwal SI, Motghare Vm, Deshmukh Vs, Jaykare Sc, Jadhav Ss	
Abstract View PDF Download PDF [149 Downloads]	86 Views
ANTI-INFLAMMATORY ACTIVITIES OF FRUIT AND LEAVES EXTRACT OF LANNEA MICROCARPA ENGLAND K. KRAUS (ANACARDIACEAE)	177-182
Batiano Jean Hubert, Hilou Adama, Compaore Moussa, Coulibaly Y Ahmed, Kiendrebeogo Martin, Nacoulma Odile Germaine	
Abstract View PDF Download PDF [183 Downloads]	187 Views
DRUG UTILIZATION AND ECONOMIC IMPACT OF ANTICOAGULANTS IN UNSTABLE ANGINA/ NON- ST ELEVATION MYOCARDIAL INFARCTION IN KARACHI	183-185
Shazia Alam, Syed Baqir Shyum Naqvi, Maqsood Ahmed	
Abstract View PDF Download PDF [171 Downloads]	179 Views
ANTIFUNGAL SCREENING OF 61 TRADITIONAL MEDICINAL PLANTS OF 305 EXTRACTS AGAINST DERMATOPHYTIC FUNGI TRICHOPHYTON TONSURANS	186-190
P. Shivakumar Singh, G. M. Vidyasagar	
Abstract View PDF Download PDF [163 Downloads]	162 Views
RP-HPLC METHOD FOR SIMULTANEOUS ESTIMATION AND STABILITY INDICATING STUDY OF METFORMIN AND LINAGLIPTIN IN PURE AND PHARMACEUTICAL DOSAGE FORMS	191-197
Mallikarjuna Rao N, Gowri Sankar D	
Abstract View PDF Download PDF [258 Downloads]	642 Views
EXTRACTION, STABILITY AND SEPARATION OF ANTHOCYANINS OF IXORA COCCINEA LINN	198-202
Nital Patil, Ajit Datar	
Abstract View PDF Download PDF [379 Downloads]	336 Views
CITRONELLOL: A POTENTIAL ANTIOXIDANT AND ALDOSE REDUCTASE INHIBITOR FROM CYMBOPOGON CITRATUS	203-209
Ashwini Dinkar Jagdale, Snehalata Prakash Kamble, Megha Laxman Nalawade, Akalpita Umesh Arvindekar	
Abstract View PDF Download PDF [299 Downloads]	274 Views
MOLECULAR DYNAMICS GUIDED 4D QSAR STUDIES ON CHROMENONE BASED DNA-DEPENDENT PROTEIN KINASE INHIBITORS	210-218

Rajesh B Patil, Sanjay D Sawant, Rakhi R Tikhe	
Abstract View PDF Download PDF [190 Downloads]	166 Views
<hr/>	
PHARMACOKINETICS AND BIOEQUIVALENCE STUDIES OF WARFARIN SODIUM 5 MILLIGRAMS TABLET IN HEALTHY THAI SUBJECTS	219-222
Chuleegone Sornsuvit, Narawadee Niamhun, Nutthida Luengpiansamut, Siriluk Sangsrijan, Wirat Niwatananum, Sayam Kaewwichit, Darunee Hongwiset, Nontakan Nuntachit, Songwut Yotsawimonwat	
Abstract View PDF Download PDF [421 Downloads]	207 Views
<hr/>	
NEW FLUORESCENCE QUENCHING BASED METHOD FOR THE DETERMINATION OF TRANDOLAPRIL IN BULK AND CAPSULES	223-227
Amir Alhaj Sakur, Tamim Chalati, Hanan Fael	
Abstract View PDF Download PDF [199 Downloads]	235 Views
<hr/>	
PREPARATION, DEVELOPMENT AND VALIDATION OF UV SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF APIGENIN IN APIGENIN-HYDROGENATED SOY PHOSPHATIDYLCHOLINE (HSPC) COMPLEX	228-231
Arun P, Amol T, T Darshan	
Abstract View PDF Download PDF [223 Downloads]	91 Views
<hr/>	
EFFECT OF TAMSULOSIN A SELECTIVE A1-ANTAGONIST ON GLUCOSE HOMEOSTASIS IN RATS	232-234
Gurudatta Moharir, Shivaprasad K, Ambadasu Bharatha, A A Naikawadi, Wali R. S.	
Abstract View PDF Download PDF [222 Downloads]	214 Views
<hr/>	
EXPLORATION OF IN VITRO ANTIOXIDANT AND ANTIMICROBIAL STUDIES OF MOMORDICA CYMBALARIA FENZL LEAVES EXTRACTSEXPLORATION OF IN VITRO ANTIOXIDANT AND ANTIMICROBIAL STUDIES OF MOMORDICA CYMBALARIA FENZL LEAVES EXTRACTS	235-241
N. Umarani, K. Ilango	
Abstract View PDF Download PDF [164 Downloads]	175 Views
<hr/>	
PREPARATION AND CHARACTERIZATION OF CROSSLINKED POLY (VINYL) ALCOHOL FILMS WITH WATERPROOF PROPERTIES	242-248
Edwin Marin, John Rojas	
Abstract View PDF Download PDF [184 Downloads]	198 Views
<hr/>	
ENVIRONMENTALLY BENIGN SYNTHESIS OF N'-SUBSTITUTED NAPHTHALENE-1-SULFONOHYDRAZIDE UNDER MICROWAVE IRRADIATION AND THEIR BIOLOGICAL EVALUATION	249-253
Sunita Ghiya, Yogesh C Joshi	
Abstract View PDF Download PDF [149 Downloads]	148 Views
<hr/>	
GASTROPROTECTIVE POTENTIAL OF LASIA SPINOSA IN ALBINO RATS	254-257
Mohammed Atif, Mohammed Azharuddin, Syed Baquer Mahmood	
Abstract View PDF Download PDF [157 Downloads]	60 Views
<hr/>	
OPTIMIZATION OF PROCESS PARAMETERS FOR THE PRODUCTION OF ANTIBACTERIAL COMPOUND BY A NEWLY ISOLATED NOCARDIA CYRIACIGEORGICA, KD-15 STRAIN FROM KAZIRANGA NATIONAL PARK OF NORTH EAST INDIA	258-264
Madhumita Talukdar, Ritasree Sarma, Shruti Talukdar, Archana Yadav, Dhruva K Jha, Tarun Chandra Bora	
Abstract View PDF Download PDF [194 Downloads]	157 Views

ANTIBIOTIC SUSCEPTIBILITY PATTERN AND ESBL PREVALENCE IN ESCHERICHIA COLI ISOLATES FROM PUS SAMPLES IN A TERTIARY CARE HOSPITAL	263-264
Sandhiya R, Lakshmi Priya R, Esthermary Selvam	
Abstract View PDF Download PDF [131 Downloads]	99 Views
IN VITRO ANTIBACTERIAL ACTIVITY OF LEAF, SEED, ROOT, POD AND FLOWER EXTRACTS OF CAJANUS CAJAN (L.) MILLSP	265-268
Mahitha Banala, Srikanth Kagithoju, Archana Pamulaparathi, Chamundeshwari Edara, Ramaswamy Nanna	
Abstract View PDF Download PDF [193 Downloads]	154 Views
FREEZE-DRIED POLYMERIC NASAL INSERTS FOR ANTIHYPERTENSIVE DRUG DELIVERY	269-273
Amal Youssef M. Abdel Reheem	
Abstract View PDF Download PDF [195 Downloads]	104 Views
DEVELOPMENT AND VALIDATION OF ZERO AND FIRST ORDER SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF OPIPRAMOL IN BULK AND PHARMACEUTICAL DOSAGE FORM	274-277
Fazil Khan, Bm Gurupadaya, Am Nasefa, Sai Prudhvi	
Abstract View PDF Download PDF [176 Downloads]	132 Views
STRUCTURE AFFINITY RELATIONSHIP AND CHARACTERIZATION OF BENZOPORPHYRINS AS POTENT INHIBITORS OF YAP ONCOPROTEIN THROUGH IN SILICO EXPERIMENTS	278-284
M. Vijey Aanandhi, P. Samuel Gideon George	
Abstract View PDF Download PDF [174 Downloads]	165 Views
ANTITESTICULAR ACTIVITY OF HYDRO-METHANOL EXTRACT OF CUMINUM CYMINUM IN ADULT RAT: A DOSE DEPENDENT STUDY	285-291
Bhabani Prasad Pakhira, Kishalay Jana, Abhinandan Ghosh, Debidas Ghosh	
Abstract View PDF Download PDF [153 Downloads]	72 Views
ANTIARRHEAL ACTIVITY OF THYMUS ALGERIENSIS BOISS AND REUT AQUEOUS EXTRACT ON RATS AND MICE	292-294
Leila Beyi, Mohammed Aziz, Hanane Makrane, Ahmed Karim, Mohammed Bnouham, Abderrahim Ziyat	
Abstract View PDF Download PDF [193 Downloads]	137 Views
IN SILICO STUDIES ON NEW INDAZOLE DERIVATIVES AS GSK-3B INHIBITORS	295-299
Namachivayam Balakrishnan, Joseph Santhana Raj, Naresh Kandakatia	
Abstract View PDF Download PDF [193 Downloads]	165 Views
THE EFFECT OF HANDING MERCURY DROP ELECTRODE ON DIFFERENTIAL PULSE POLAROGRAPHIC BEHAVIOR AND DETERMINATION OF ROSUVASTATIN IN PURE AND PHARMACEUTICAL TABLETS	300-307
Abdul Aziz Ramadan, Hasna Mandil, Nour Ghazal	
Abstract View PDF Download PDF [124 Downloads]	92 Views
OPTIMIZATION, CHARACTERIZATION AND STABILITY OF ESSENTIAL OILS BLEND LOADED NANOEMULSIONS BY PIC TECHNIQUE FOR ANTI-TYROSINASE ACTIVITY	308-312
Thananya Nantararat, Sunee Chansakaow, Pimporn Leelapornpisid	
Abstract View PDF Download PDF [581 Downloads]	446 Views

<p>COMPARATIVE STUDY OF AQUEOUS EXTRACT OF MOMORDICA CHARANTIA SEEDS WITH SYNTHETIC INSULIN SENSITIZERS ON BLOOD GLUCOSE LEVELS AND BODY WEIGHT IN ALBINO RATS</p> <p>Guruprasad Nb, Dasaraju Rajesh, Thejaswini Muppala</p> <p>Abstract View PDF Download PDF [225 Downloads]</p>	313-316	158 Views
<p>CYTOTOXICITY AND ANTIMICROBIAL ACTIVITY OF MONO-, DI- AND TRINUCLEAR RUTHENIUM(II) POLYPYRIDINE COMPLEXES</p> <p>Senthamarai Kannan Balakrishnan, Suresh Kumar Dass, Host Antony David Rajendran, Ignacimuthu Savrimuthu</p> <p>Abstract View PDF Download PDF [192 Downloads]</p>	317-321	424 Views
<p>ANTIMICROBIAL ACTIVITIES OF CHLORHEXIDINE GLUCONATE AND CETRIMIDE AGAINST PATHOGENIC MICROORGANISMS ISOLATED FROM SLAUGHTER HOUSES IN RIVERS STATE, NIGERIA</p> <p>Oluwayemisi Agnes Agnes Olorode, Emmanuel A Bamigbola, Ofonime M Ogba</p> <p>Abstract View PDF Download PDF [181 Downloads]</p>	322-328	137 Views
<p>IN VITRO EFFECT OF ACETYLSALICYLIC ACID ON CALCIUM OXALATE CRYSTALLIZATION: AN APPROACH TO ANTLITHIASIS</p> <p>Bouatia Mustapha, Benzeid Hanane, Idrissi M.O.B, Benramdane Laarbi, Draoui Mustapha</p> <p>Abstract View PDF Download PDF [168 Downloads]</p>	329-331	194 Views
<p>METHOD DEVELOPMENT FOR SIMULTANEOUS ANALYSIS OF MARKER SCOPOLETINE, ANDROGRAPHOLIDE, QUERCETIN, AND LUTEOLIN IN ANTIHYPERTENSION JAMU FORMULATION USING RP-HPLC</p> <p>Ummi Rukoyah, Irda Fidrianny, Komar Ruslan W</p> <p>Abstract View PDF Download PDF [313 Downloads]</p>	332-336	161 Views
<p>LEAD IDENTIFICATION AND OPTIMIZATION OF PLANT INSULIN-BASED ANTIDIABETES DRUGS THROUGH MOLECULAR DOCKING ANALYSES</p> <p>Shabana Bibi, Saima Kalsoom, Hamid Rashid, Katsumi Sakata</p> <p>Abstract View PDF Download PDF [192 Downloads]</p>	337-343	180 Views
<p>INSIGHTS INTO THIORIDAZINE FOR ITS ANTI-TUBERCULAR ACTIVITY FROM MOLECULAR DOCKING STUDIES</p> <p>A Jerad Suresh, R Devi, K M Noorulla, P R Surya</p> <p>Abstract View PDF Download PDF [179 Downloads]</p>	344-346	180 Views
<p>FORMULATION AND EVALUATION OF NATURAL HAIR COLORANTS</p> <p>Rashmi Mallya, Padmini Ravikumar</p> <p>Abstract View PDF Download PDF [622 Downloads]</p>	347-349	400 Views
<p>EVALUATION OF MYRISTICA FRAGRANS AS A PENETRATION ENHANCER IN TRANSDERMAL GEL FORMULATION</p> <p>Sunetra Kaustubh Patwardhan, Manali Anant Bhide</p> <p>Abstract View PDF Download PDF [187 Downloads]</p>	350-355	132 Views
<p>EFFECT OF MOMORDICA CHARANTIA FRUITS ON STREPTOZOTOCIN-INDUCED DIABETES MELLITUS AND ITS ASSOCIATED COMPLICATIONS</p> <p>Arvind Kumar Srivastava, Arvind Mishra, Sudeep Gautam, Savita Pal, Akansha Mishra, Arun Rawat Kumar, Rakesh Maurya</p> <p>Abstract View PDF Download PDF [213 Downloads]</p>	356-363	224 Views

EFFECT OF CAFFEINE IN EXPERIMENTAL MODEL OF RHEUMATOID ARTHRITIS IN RATS	364-367
Krishna Shah, Paresh Dadhaniya, Vandit R Trivedi	
Abstract View PDF Download PDF [230 Downloads]	173 Views
ANTI-ULCER AND ANTIOXIDANT ACTIVITY OF NELUMBO NUCIFERA GAERTN STALKS IN RATS	368-373
Amit Kumar Srivastava, Umesh Kumar Patil, Ashish Singhai, Manish Kumar	
Abstract View PDF View PDF View PDF Download PDF [188 Downloads]	338 Views
EVALUATION OF HEPATOPROTECTIVE AND ANTIOXIDANT ACTIVITY OF PELLIONIA HEYNEANA WEDD. LEAF ETHANOLIC EXTRACT ON CARBON TETRACHLORIDE INDUCED LIVER DAMAGE IN WISTAR RATSEVALUATION OF HEPATOPROTECTIVE AND ANTIOXIDANT ACTIVITY OF PELLIONIA HEYNEANA WEDD LEAF E	374-378
V Vilash, S R Suja, P G Latha, S Rajasekharan	
Abstract View PDF Download PDF [161 Downloads]	113 Views
SUB-LETHAL TOXICITY OF POTASSIUM CYANIDE ON NILE TILAPIA (OREOCHROMIS NILOTICUS): BIOCHEMICAL RESPONSE	379-382
Bangeppagari Manjunatha, Juan Ortiz Tirado, Mariadoss Selvanayagam	
Abstract View PDF Download PDF [244 Downloads]	301 Views
SPECTROSCOPIC ANALYSIS AND ANTIBACTERIAL EFFICACY OF BIOACTIVE COMPOUNDS FROM LIMONIA ACIDISSIMA L. FRUIT EXTRACT AGAINST CLINICAL PATHOGENS	383-389
Srinivasan Ponnuraj, Dineshbabu Jagathanan, Manimekalai Kanagarajan, Teepica Priya Darsini Deivamarudachalam	
Abstract View PDF Download PDF [276 Downloads]	228 Views
ANTI ISCHEMIA REPERFUSION EFFECT OF SODIUM THIOSULFATE IN LLC PK1 CELLS	390-396
Gino A Kurian	
Abstract View PDF Download PDF [137 Downloads]	150 Views
SYNTHESIS, CHARACTERIZATION AND EVALUATION OF ANTICANCER ACTIVITY OF SOME NEW SCHIFF BASES OF 1, 3, 4-THIAZOLE DERIVATIVES	397-402
Arup Naskar, Tanushree Singha, Tanmoy Guria, Jagadish Singh, Ashok Kumar B, Tapan Kumar Maity	
Abstract View PDF Download PDF [222 Downloads]	284 Views
ANTIARRHOEAL ACTIVITY OF AQUEOUS AND ALCOHOLIC EXTRACTS OF HEMIDESMUS INDICUS ROOT	403-406
Rajan Suyambu, Shalini R	
Abstract View PDF Download PDF [168 Downloads]	165 Views
TRADITIONAL MEDICINAL PLANTS USED BY LOCAL PEOPLE OF KAILASAKONA- A SACRED GROVE OF CHITTOOR DISTRICT, ANDHRA PRADESH, INDIA	407-411
Nataru Savithamma, Pulicherla Yugandhar, Damai Suhlulatha	
Abstract View PDF Download PDF [226 Downloads]	272 Views
EFFECT OF BLACK CUMIN (NIGELLA SATIVA) POWDER ON SERUM LIPID PROFILE, MALONDIALDHYDES, NITRITES, SICAM-1 AND SVCAM-1 IN EXPERIMENTALLY INDUCED ATHEROSCLEROSIS	412-417
Elsayed Abdelhady Elmorsy, Basem H Elesawy, Hatim A El-Baz	

[Abstract](#) || [View PDF](#) || [Download PDF](#) [175 Downloads] 120 Views

FORMULATION, IN-VITRO RELEASE KINETICS AND STABILITY INTERPRETATION OF SUSTAINED RELEASE TABLETS OF METFORMIN HYDROCHLORIDE 418-422

Sudipta Das, Arnab Samanta, Himadri Sekhar De

[Abstract](#) || [View PDF](#) || [Download PDF](#) [163 Downloads] 152 Views

ENHANCEMENT OF SOLUBILITY AND DISSOLUTION RATE OF TELMISARTAN BY TELMISARTAN-OXALIC ACID CO-CRYSTAL FORMATION 423-426

Fikri Alatas, Hestiar Ratih, Sundani Nuroso Soewandhi

[Abstract](#) || [View PDF](#) || [Download PDF](#) [339 Downloads] 396 Views

IMPACT OF TAI IN TRANSLATIONAL DYNAMICS OF HOMO SAPIENS GENES IN ESCHERICHIA COLI GENOME 427-432

Binata Halder, Supriyo Chakraborty

[Abstract](#) || [View PDF](#) || [Download PDF](#) [142 Downloads] 160 Views

MULTIPARTICULATE DRUG DELIVERY SYSTEM FOR COLON TARGETING 433-436

Sangeeta Mohanty, Amit Kumar Panigrahi

[Abstract](#) || [View PDF](#) || [Download PDF](#) [190 Downloads] 151 Views

COMPARISON OF POST-LICENSURE SAFETY SURVEILLANCE OF BIVALENT AND QUADRIVALENT HUMAN PAPILLOMAVIRUS VACCINES IN HEALTHY MUMBAI WOMEN 437-442

Vikrant C. Sangar, B B Ghongane, Rajan Gupte, Rohan Kesarkar, Karthik Kalyan, Abhay Chowdhary

[Abstract](#) || [View PDF](#) || [Download PDF](#) [150 Downloads] 184 Views

DEVELOPMENT AND VALIDATION OF RP-HPLC METHOD FOR SIMULTANEOUS DETERMINATION OF METHOTREXATE, DEXAMETHASONE AND INDOMETHACIN 443-446

Narendra K. Lariya, G. P. Agrawal

[Abstract](#) || [View PDF](#) || [Download PDF](#) [203 Downloads] 470 Views

DEVELOPMENT, OPTIMIZATION AND EVALUATION OF DIFFERENT HERBAL FORMULATIONS FOR WOUND HEALING 447-452

Fedelic Ashish Toppo, Rajesh Singh Pawar

[Abstract](#) || [View PDF](#) || [Download PDF](#) [276 Downloads] 246 Views

PHARMACOPHORE MODELING AND ATOM-BASED 3D-QSAR STUDIES OF ANTIFUNGAL BENZOFURANS 453-458

Sandeep S. Pathare, Sujit G. Bhansali, Kakasaheb R. Mahadik, Vithal M. Kulkarni

[Abstract](#) || [View PDF](#) || [Download PDF](#) [204 Downloads] 192 Views

Short Communication(s)

GREEN SYNTHESIS AND CHARACTERIZATIONS OF SILVER NANOPARTICLES USING FRESH LEAF EXTRACT OF MORINDA CITRIFOLIA AND ITS ANTI-MICROBIAL ACTIVITY STUDIES 459-461

Asha R Pai, Kavitha S, Shweta Raj S, Priyanka P, Vrinda A, Vivin T S, Silpa Sasidharan

[Abstract](#) || [View PDF](#) || [Download PDF](#) [245 Downloads] 271 Views

ANTIBACTERIAL ACTIVITY OF AEGLE MARMELOS (L) CORREA	462-464
Surender Singh Yadav, Kuldeep Dahiya, Showkat Ahmad Ganie, Surender K. Gulia	
Abstract View PDF Download PDF [256 Downloads]	219 Views
TOTAL POLYPHENOL CONTENT AND ANTIOXIDANT AND CYTOTOXIC ACTIVITIES OF THE SRI LANKAN ENDEMIC PLANT GENUS SCHUMACHERIA	465-467
Chamara Janaka Bandara, D Nedra Karunaratne, Anura Wickramasinghe, D. S. A Wijesundara, B. M. Ratnayake Bandara, Veranja Karunaratne	
Abstract View PDF Download PDF [144 Downloads]	235 Views
ANTI-INFLAMMATORY ACTIVITY OF HMG CO A REDUCTASE INHIBITORS: A COMPARATIVE STUDY	468-469
Rajitha Panonnummal, Joyamma Varkey	
Abstract View PDF Download PDF [137 Downloads]	75 Views
PHYTOCHEMICAL ANALYSIS OF WRIGHTIA TINCTORIA BARK EXTRACT IN WATER USING GC-MS	470-472
Rajendra Singh, Arun Kakkar, Vinod Kumar Mishra	
Abstract View PDF Download PDF [244 Downloads]	236 Views
IN VITRO EVALUATION OF DISSOLUTION PROFILE OF TWO COMMERCIALY AVAILABLE FOLIC ACID PREPARATIONS	473-475
Omar Sarheed, Ramesh Kvrns, Fasiha Shah	
Abstract View PDF Download PDF [379 Downloads]	126 Views
EX VIVO ANTICOAGULANT ACTIVITY OF 1, 3, 4-OXADIAZOLE DERIVATIVES	476-478
B. Vishwanathan, B. M. Gurupadayya	
Abstract View PDF Download PDF [144 Downloads]	79 Views
MOLECULAR IDENTIFICATION OF AEROMONAS SPP ISOLATED FROM PATIENTS WITH DIARRHEA AT SANTA MARIA-RS, BRAZIL	479-480
Adriana Medianeira Rossato, Karoline De Campos Prediger, Cristiane Nascimento Gomes, Carlos Hugo Winckler Dal Priore Neto, Renata Da Silva Pereira, Cibelle De Borba Dallagassa, Roberto Christ Vianna Santos, Cyntia Maria Telles Fadel-Picheth, Bruno Stefanello Vizzotto	
Abstract View PDF Download PDF [150 Downloads]	205 Views
N-PHENYLBENZAMIDE SYNTHESIS BY NUCLEOPHILIC SUBSTITUTION WITH 1,3-DIPHENYLTHIOUREA	481-482
Indah Purnama Sary, Siswandono Siswodihardjo, Tutuk Budiati	
Abstract View PDF Download PDF [302 Downloads]	131 Views

**International Journal of Pharmacy and Pharmaceutical Sciences (IJPPS):
a brief description**

International Journal of Pharmacy and Pharmaceutical Sciences (Int J Pharm Pharm Sci) is peer reviewed, Monthly (Onward April 2014) open access Journal with ISSN 0975 – 1491 (ISSN Number). IJPPS publishes original research work that contributes significantly to further the scientific knowledge in pharmacy and pharmaceutical sciences (Pharmaceutical Technology, Pharmacognosy, Natural Product Research, Pharmaceutics, Novel Drug Delivery, Biopharmaceutics, Pharmacokinetics, Pharmaceutical/Medicinal Chemistry, Computational Chemistry and Molecular Drug Design, Pharmacology, Pharmaceutical Analysis, Pharmacy Practice, Clinical and Hospital Pharmacy, Cell Biology, Genomics and Proteomics, Pharmacogenomics, Bioinformatics, Pharmacoeconomics). Research outcomes from medical sciences/case study and biotechnology of pharmaceutical interest are also considered. IJPPS publishes original research work either as an Original Article or as a Short Communication. Review articles on current topic under mentioned scopes are also considered for publication.

Abstracting and indexing of this journal include Google Scholar, Scopus, Elsevier, EBSCO, EMBASE, SCI mago (SJR), CAS, CASSI (American Chemical Society), Directory of Open Access Journal (DOAJ), Index Copernicus, ICAAP, Scientific commons, PSOAR, Open-J-Gate, Indian Citation Index (ICI), Index Medicus for WHO South-East Asia (IMSEAR), OAI, LOCKKS, OCLC (World Digital Collection Gateway), UIUC, and Chemical Abstracts, Medline, Pubmed, Pubmed Central are under process.

Short Communication

N-PHENYLBENZAMIDE SYNTHESIS BY NUCLEOPHILIC SUBSTITUTION WITH 1,3-DIPHENYLTHIOUREA

INDAH PURNAMA SARY^{a,*}, SISWANDONO^b, TUTUK BUDIATI^b

^aFaculty of Pharmacy, Jember University, Jember, East Java 68121, Indonesia, ^bFaculty of Pharmacy, Airlangga University, Surabaya, East Java 60286, Indonesia.

Email: indahpurnamasary.farmasi@unej.ac.id

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ABSTRACT

Objective: *N*-phenylbenzamides are important and biologically active compounds. *N*-phenylbenzamides have been synthesized by some routes. An attempt has been made to find out the new route to synthesize *N*-phenylbenzamides.

Methods: The reaction was carried out by reacting substituted benzoyl chlorides with 1,3-diphenylthiourea in the presence of triethylamine in THF at 70 °C. After 4hr the product was purified and identified.

Results: An excellent and pure yield of *N*-phenylbenzamides was obtained by reacting substituted benzoyl chlorides with 1,3-diphenylthiourea. The proposed mechanism follows imino alcohol-amide tautomerism and suggests the involvement of rearrangement intermediate.

Conclusion: 1,3-diphenylthiourea is inexpensive commodity chemical and it is found to be the useful reagent for the direct conversion to *N*-phenylbenzamide. The proposed mechanism follows imino alcohol-amide tautomerism and suggests the involvement of rearrangement intermediate. The synthesis gave pure, high yield, and the one and only isolated product.

Keywords: *N*-phenylbenzamide, 1,3-diphenylthiourea, Imino alcohol-amide tautomerism, Rearrangement intermediate.

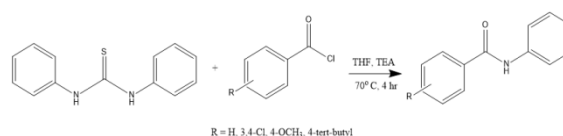
Benzamides are important class of compounds and useful building blocks in organic synthesis [1, 2]. Benzamides, especially *N*-phenyl benzamides, display a wide range of biological activity including antibacterial, antimicrobial, antiviral, antimalarial, anticonvulsant, analgesic, antidiabetic, antiulcer and anti allergy agents [1-7].

N-phenylbenzamides have been synthesized by interconversions of carboxylic acid derivatives, metal-catalyzed amidation of aryl halides, oxidative amidation of aldehyde, aminocarbonilation, and other routes [8]. In this article, we reported a direct route to *N*-phenyl benzamides by conversion of benzoyl chloride derivatives with 1,3-diphenylthiourea.

Reaction of benzoyl chloride derivatives with 1,3-diphenylthiourea presumably and expectedly yielded the targeted compounds, 1,3-dibenzoyl-1,3-diphenylthiourea derivatives [9]. Instead of the targeted compounds, the synthesis gave different compound as high yield (40-93%) and the one and only isolated product, which were *N*-phenylbenzamides.

The synthesis of *N*-phenylbenzamides was carried out by adding dropwise a solution of substituted benzoyl chloride (20 mmol) in tetrahydrofuran (10 ml) to a suspension of 1,3-diphenylthiourea (10 mmol) and triethylamine (10 mmol) in tetrahydrofuran (20 ml),

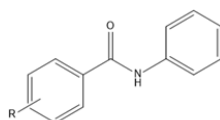
then the reaction mixture was refluxed for 4 hours. The reaction mixture was evaporated, quenched into water and washed by saturated sodium bicarbonate and water again. The paste form-reaction mixture was poured into hot ethanol and the precipitated *N*-phenylbenzamides were recrystallized aqueous ethanol.



Scheme 1: Synthesis of *N*-phenylbenzamides

¹HNMR, ¹³CNMR, IR, and UV-Vis spectra of all synthesis products indicated undesired different results. There was still one unsubstituted amine of 1,3-diphenylthiourea shown by ¹HNMR, ¹³CNMR and IR spectra, giving assumption that the reaction produced 1-benzoyl-1,3-diphenylthioureas. Unpredictably, ¹HNMR and ¹³CNMR spectra showed loss of some protons and carbons of the corresponding 1-benzoyl-1,3-diphenylthiourea compound. Finally, we established the structure of *N*-phenylbenzamides in accordance with the spectra.

Table 1: Derivatives of *N*-phenylbenzamide^a



Compound	R	Mp (°C)	Yield (%)	Formula
1	H	160	82	C ₁₃ H ₁₁ ON
2	3,4-Cl	169	69	C ₁₃ H ₁₀ ONCl ₂
3	4-OCH ₃	159	93	C ₁₄ H ₁₃ O ₂ N
4	4- <i>tert</i> -but	117	40	C ₁₇ H ₁₉ ON

^aThe infrared and nuclear magnetic resonance (¹H and ¹³C) spectra were consistent with the structural assignments.

Herein, we explained the proposed mechanism for the conversion to *N*-phenylbenzamide products. Nucleophilic substitution reaction between 1,3-diphenylthiourea and benzoyl chloride derivatives gave the attachment of one benzoyl ring to one of the amine, then continued with rearrangement to obtain *N*-phenylbenzamides. The

proposed mechanism follows imino alcohol-amide tautomerism. In the case of hydroxide ion the result, after protonation, is an imidic acid (also called an imino alcohol), the tautomer of an amide, and in general less stable than the corresponding amide [10]. It obviously explained the products were amide tautomer.

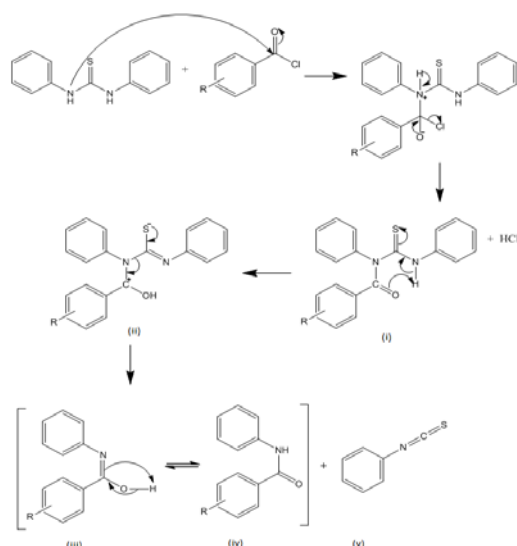


Fig. 1: The proposed mechanism of *N*-phenylbenzamide synthesis

The proposed mechanism (fig. 1) involves further steps that include conversion of 1-benzoyl-1,3-diphenylthiourea (i) to *N*-phenylbenzamide (iv). Based on the observed products, we suggest the bulky size of 1,3-diphenylthiourea to give a hindrance so that only one amine successfully substituted by benzoyl moiety, resulting compound (i). Furthermore, rearrangement occurred due to the bulky steric strain owned by (i), converting it to compound (ii). The unstable compound (ii) converted itself into compound (iii), which was the tautomer of (iv), and release compound (v) at the same time.

We proposed that rearrangement was the key intermediate in the conversions to the *N*-phenylbenzamide products. The rearrangement converting (i) to (ii) gave a possible route to provide compound (iii), the tautomer of an amide (iv). This suggestion was in accordance with the earlier study of proton-transfer tautomerization of benzanilide (*N*-phenylbenzamide) to its imidol form (*N*-phenylbenzimidic acid) reported by Tang [11]. Moreover, the conversions to the *N*-phenylbenzamides also released compound (v), phenylisothiocyanate. Presumably, any byproducts phenylisothiocyanate were lost in the ethanolic phase during workup.

In conclusion, we have found that 1,3-diphenylthiourea is a useful reagent for the direct conversions to the *N*-phenylbenzamides. The proposed mechanism follows imino alcohol-amide tautomerism and suggests the involvement of rearrangement intermediate. The synthesis gave pure, high yield, and the one and only isolated product.

ACKNOWLEDGMENT

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CONFLICT OF INTERESTS

We hereby declare that there is no conflict of interest

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