



ANKARA UNIVERSITY
FACULTY OF PHARMACY



I S O P S 13th International SYMPOSIUM ON
PHARMACEUTICAL SCIENCES

Book of Abstracts

JUNE 22-25, 2021
ANKARA, TURKEY





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Dear Participants and Guests,

I would like to express my sincere appreciation for the valuable contributions of all the participants of 13th International Symposium on Pharmaceutical Sciences (ISOPS). As we all know, the COVID-19 pandemic is still ongoing and the vaccination programmes are proceeding throughout the world. However, during 2021 we continue to face travel bans, governmental and contract restrictions in many countries. Therefore, the symposium was organized as a virtual event for the first time in its history.

ISOPS, initiated in 1989, has successfully brought international scientists, researchers, students together from pharmaceutical sciences and related areas. This symposium was organized biannually until 1997 and then every three years.

Ankara University, Faculty of Pharmacy is the first faculty of pharmacy in Turkey and was established in nineteen sixty (1960). Since the establishment, the institution rapidly progressed and now has very advanced scientific and physical infrastructure. Pharmaceutical science refers to a category of scientific fields and has followed important development processes, mainly in line with the developments in Biotechnology, Nanotechnology and Health Technologies, which are among the priority of the technology fields of today. While realizing the modern requirements, our Faculty has a 5-year undergraduate education programme since 2005 and besides Turkish; it provides an instruction programme in English language since 2015. Our faculty has 6689 graduates since its establishment and the current number of students is 1267. Present educational and scientific resources allow a total of 138 faculty members, 45 professors, 22 associate professors, 5 assistant professors, 51 research assistants in our faculty. Moreover, 66 administrative staff members and other personnel are working at different offices.

The mission of 13th International Symposium of Pharmaceutical Sciences was to perform a broad scientific perspective by the invitation of distinguished scientists having national / international reputation in their areas, so most recent advances were discussed interactively, and to empower the knowledge-based drug research development and multidisciplinary collaborations. It was our intention to make this symposium a memorable event.

This year, scientists from 24 countries registered to ISOPS-13. Our programme consisted of 40 plenary lectures, 212 oral and 200 poster presentations. Excellent research works were presented in different sessions. The speakers in the programme were uniquely placed in accordance to their area of expertise.

I would like to refer also to other initiatives that took place in our symposium. A workshop on "Employability of the Graduates of the Faculty of Pharmacy in Europe" was held with the contribution of Prof. Luciano Saso, Prof. Claire Anderson, Prof. Lilian M. Azzopardi, Prof. Sibel Süzen, Prof. İlkay Erdogan Orhan and Pharm. Nilhan Uzman. This workshop was interesting in terms of discussing the priorities and developments on this topic from local, regional and international respects.

On June 25, our panel on "University-industry-public sector cooperation in drug and vaccine development processes" was carried out by Prof. Dr. Asuman BOZKIR. The heads and *senior representatives of relevant institutions* including; Prof. Hasan Mandal, Assoc. Prof. Tolga Karakan, Pharm. Dr. Nihan Burul Bozkurt, Prof. Erhan Akdoğan, Assoc. Prof. Rabia Çakır Koç, Prof. Mayda Gürsel, Prof. Rana Sanyal, Prof. Hülya Ayar Kayalı, Dr. Süha Taşpolatoğlu, Dr. Hasan Ersin Zeytin, and Pharm. Dr. Ferhat Farşı were with us. This event has been a great platform to discuss the existing practices and requirements, and to propose solutions.

On behalf of the Organizing Committee, I would like to mention my gratitude to the President of Ankara University who gave full support for the Symposium Organization. ISOPS-13 was organized successfully, without any professional support, with the contribution of all our faculty members, especially our symposium secretary Assoc. Prof. Zerrin Sezgin-Bayındır. I congratulate the organizing committee and all the other committees with all my heart, as well as all academic and managing personnel because of their extensive work.

Prof. Dr. Asuman BOZKIR

Chair of ISOPS-13

Honory President of the Symposium

Prof. Dr. Necdet ÜNÜVAR

President of Ankara University

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CONTENTS

PL1:	DUAL BENEFITS OF MELATONIN ANALOGUES AS AROMATASE INHIBITORS AND OXIDATIVE STRESS MODULATORS IN BREAST CANCER	2
	¹ Suzen, S., ² Shirinzadeh, H., ¹ Öztürk-Ceylan Ö., ³ İnce-Ergüç E., ^{BR.} , ⁴ Taşçıoğlu-Aliyev A., ⁴ Entezari B., ⁵ Akdemir, A., ⁴ Gurer-Orhan H.	
PL2:	MACHINE LEARNING MODELS FOR PREDICTING DRUG SYNERGY AND SIDE EFFECTS.....	2
	Çiçek, E.	
PL3:	THE ERA OF mRNA VACCINES.....	2
	Diken, M.	
PL4:	CYCLODEXTRIN POLYMER COATINGS FOR DRUG DELIVERY: FROM NANOPARTICLES TO HYDROGELS	3
	Amiel, C.	
PL5:	MESOPOROUS SILICA FOR ADVANCED DRUG DELIVERY APPLICATIONS	3
	Rosenholm, JM.	
PL6:	NOVEL DRUG TARGETING FOR LOCALIZED-DIRECT TREATMENT OF LUNG DISEASES	4
	Yıldız-Peköz, A.	
PL7:	MODULATION OF OXIDATIVE STRESS AS A PHARMACOLOGICAL STRATEGY	4
	Saso, L.	
PL8:	MODULATION OF BETA ADRENERGIC SYSTEM FOR EXACERBATED INFLAMMATION	5
	Ibanez, B.	
PL9:	DRUG DESIGN AND BIOLOGICAL ACTIVITY OF COMPOUNDS TARGETING HUMAN AND/OR PATHOGENIC PROTEASES	5
	Micale, N.	
PL10:	DESENSITIZATION OF β_3 -ADRENOCEPTORS: COMPARISON OF cAMP AND BIASED SIGNALING	5
	Okeke, K., Michel-Reher, M. B., Michel, M. C.	
PL11:	GRK2 INHIBITION FOR HEART FAILURE - NEARING TRANSLATION	6
	Koch, W.J.	
PL12:	NANOBIOSENSORS FOR POINT-OF-CARE DIAGNOSTICS APPLICATIONS	6
	Merkoçi, A.	
PL13:	BUILDING NEW ANALYTICAL PLATFORMS BASED ON CARBON NANOMATERIALS FOR BIOMARKERS BIOSENSING	7
	Rivas, G.	
PL14:	VACCINE DESIGN AND INNATE IMMUNE NETWORK.....	7
	Engin, ED.	
PL15:	TARGETED NANOPARTICLES FOR BRAIN DELIVERY OF DRUGS	8
	¹ Mészáros, M., ^{1,2} Porkoláb, G., ¹ Szecskó, A., ¹ Veszelka, S., ¹ Deli, M.A.	
PL16:	USE OF MAGNETIC-INDUCED HYPERTERMIA FOR CANCER TREATMENT	8
	Carvalho, F.	
PL17:	PROGRESS AND TRENDS IN POTENTIAL UTILIZATION OF NATURAL COMPOUNDS AS DRUGS PRENYLATED PHENOLICS.....	8
	¹ Šmejkal, K., ² Mašek J.	
PL18:	THE ROLE OF CHITOSAN-COLLAGEN BINDING IN DRUG TARGETING TO FIBROTIC DISEASES	9
	Tammam, S.	

PL19:	IN VITRO TOPICAL TOXICITY TESTING OF MEDICAL DEVICES IN LINE WITH THE NEW ISO 10993-23	10
	Kandarova, H.	
PL20:	PLANT CHEMOPHENETIC STUDIES FROM ASTERACEAE TO ZOSTERACEAE.....	10
	Zidorn, C.	
PL21:	THE IMPORTANCE OF BIOMONITORING OF GENOTOXICITY BIOMARKERS IN OCCUPATIONAL SETTINGS.....	10
	Başaran, N.	
PL22:	TOWARD MULTOMICs BIOELECTROANALYTICAL PROFILING FOR PERSONALIZED MEDICINE.....	11
	¹ Campuzano, S., ² Barde拉斯, R., ¹ Povedano, E., ¹ Torreto-Rodríguez, R.M., ¹ Gamella, M., ² Montero-Calle, A., ² Solís-Fernández, G., ¹ Pedrero, M., ¹ Pingarrón. J.M.	
PL23:	PRE-CONCENTRATION AND SELECTIVE ADSORPTION OF PLANT SECONDARY METABOLITES BY SOLID SORBENTS.....	11
	Epifano, F.	
PL24:	HYDROGEN SULFIDE PATHWAY ROLE IN CARDIOVASCULAR SYSTEM: A POTENTIAL THERAPEUTIC TARGET	12
	Sorrentino, R.	
PL25:	ENANTIOSELECTIVE LIQUID CHROMATOGRAPHY IN A TRANSLATIONAL CHEMISTRY PERSPECTIVE.....	13
	Sardella, R.	
PL26:	CANNABINOIDs AGAINST CISPLATIN NEUROTOXICITY.....	13
	Erol, K.	
PL27:	MICROSAMPLING IN BIOANALYSIS: NEW CHALLENGES AND PERSPECTIVES ...	14
	Mercolini, L.	
PL28:	TOWARD PHARMACOLOGICAL MODULATION OF SERCA FUNCTION: WHY AND HOW.....	14
	Zaza, A.	
PL29:	THE IN VITRO ANTIMICROBIAL-ACTIVITY APPROACH AGAINST MDR BACTERIA USING METAL/METAL-OXIDE NANOPARTICLES	15
	¹ Kosalec, I., ² Vukoja, D., ¹ Rak, J., ³ Rezić, I., ⁴ Vlainić, J.	
PL30:	STRATEGIES FOR RE-DISCOVERY OF CNS DRUGS FROM AFRICAN PLANTS	16
	Adejare, A.	
PL31:	INHIBITION OF NUCLEOSIDE DIPHOSPHATE KINASES AS A NOVEL THERAPEUTIC OPTION IN THE TREATMENT OF CARDIOVASCULAR DISEASES	16
	Wieland, T.	
PL32:	BACTERIOPHAGE THERAPY: PAST, PRESENT, FUTURE	17
	Chanishvili, N.	
PL33:	DRUG-INDUCED HYPERSENSITIVITY: OPPORTUNITIES TO EXPAND NON-ANIMAL MODEL FOR THE IDENTIFICATION OF SENSITIZATION TO DRUGS	17
	Corsini, E.	
PL34:	POSITIVE OR NEGATIVE EFFECTS OF RECREATIONAL SCUBA DIVING - CAN WE ADAPT TO A CHALLENGING ENVIRONMENT?.....	17
	Dumic, J.	

PL35:	DEVELOPING, IMPLEMENTING AND EVALUATING ADVANCED PHARMACY SERVICES WORKING WITH PRACTITIONERS AND POLICY MAKERS	18
	<i>Anderson, C.</i>	
PL36:	POTENTIAL MECHANISMS UNDERLYING THE PROTECTIVE EFFECTS OF ANTHOCYANINS IN METABOLIC SYNDROME AND RELATED DISORDERS.....	18
	<i>Cimino, F.</i>	
PL37:	ROLE OF CENTELLA ASIATICA, AEROBIC EXERCISE AND ITS COMBINATION IN WOMEN WITH MILD COGNITIVE IMPAIRMENT	18
	<i>¹Adnyana, IK., ¹Anggadiredja K., ²Fitriana, LA., ³Setiawan</i>	
PL38:	MEDICATION DYSPHAGIA: FOCUS GROUP PILOT STUDY ON PHARMACISTS' KNOWLEDGE, ATTITUDES & PRACTICES MEDICATION DYSPHAGIA: FOCUS GROUP PILOT STUDY ON PHARMACISTS' KNOWLEDGE, ATTITUDES & PRACTICES.....	19
	<i>Chan, SY., Loh, JHT., Yap, KZ., Tan, PL.</i>	
PL39:	PROMISING INHIBITORS TARGETING MPRO: SELENIUM BASED COMPOUNDS WITH ANTI-SARS-COV-2 ACTIVITY	20
	<i>Santi, C., Mangiavacchi, F., Liviabella, D., Menichetti, E., Scimmi, C., Begnoli, L., Rosati, O., Sancinetto, L., Marini, F.</i>	
PL40:	INNOVATIVE DIHYDROORotate DEHYDROGENASE (HDHODH) CLINICAL READY INHIBITORS AS PAN-CORONAVIRUS (SARS-COV-2) ANTIVIRALS: TARGETING THE UNEXPECTED WITH INNOVATION	21
	<i>M. L. Lolli^{*1}, S. Sainas¹, A. Luganini², A. Calistri², G. Sibille², B. Mognetti², V. Conciatori³, C. Del Vecchio³, M. Giorgis¹, A. C. Pippone¹, R. Bagnati⁵, A. Passoni⁵, P. Circosta⁴, V. Gaidano⁴, A. Cignetti⁴, G. Saglio⁴, C. Parolin³, D. Boschi¹ and G. Gribaudo²</i>	
OP001:	DEVELOPMENT AND OPTIMIZATION OF SULPHAMETHOXAZOLE NANOSUSPENSION FORMULATIONS.....	23
	<i>¹Ugur Kaplan, AB., ¹Cetin, M.</i>	
OP002:	STABILITY ENHANCEMENT OF S-ADENOSY-L-METHIONINE THROUGH NANOFORMULATION APPROACH	23
	<i>¹Ergin, AD., ²Sezgin-Bayındır, Z., ²Yüksel, N.</i>	
OP003:	MESOPOROUS SILICA-BASED NANOCARRIER FOR TARGETED CANCER THERAPY	24
	<i>¹Leggio, A., ¹De Santo, M., ¹Fava M., ¹Morelli, C., ²Pasqua, L.</i>	
OP004:	DEVELOPMENT AND <i>IN VIVO</i> EVALUATION OF A PULSATILE-RELEASE CAFFEINE FORMULATION	24
	<i>^{1,2}Arslan, A., ^{1,3}Yerlikaya, F.</i>	
OP005:	CELLULAR UPTAKE OF POLYMERIC TUBULAR NANOCARRIERS	25
	<i>¹Algan, AH., ¹Karatas, A., ²Besikci, A.</i>	
OP006:	PREPARATION AND CHARACTERIZATION OF FAST-DISSOLVING DESLORATADINE ORAL FILM FOR GERIATRIC USE	26
	<i>¹Al-Oran, AYF., ²Yenilmez, E.</i>	
OP007:	PREPARATION AND CHARACTERIZATION OF BERBERINE LOADED CHITOSAN MICROPARTICLES.....	26
	<i>¹Gungor Ak, A., ²Karatas, A.</i>	
OP008:	THE EFFECT OF GEL PROPERTIES OF CYCLODEXtin BASED NANOgEL ON THE RELEASE AND STABILITY STUDIES	27
	<i>^{1,2}Oktay, AN., ¹Ilbasmis-Tamer, S., ^{1,3}Celebi, N.</i>	

OP009: BSA LOADED POLY(ISOBUTYL-METHYL GLYCOLIDE) NANOPARTICLES FOR DRUG DELIVERY SYSTEMS.....	28
¹ <i>Vardar, A., </i> ² <i>Erdebil, Ö., </i> ^{1,2} <i>Mert, O., </i> ²⁻⁴ <i>Mert, S.</i>	
OP010: MICROFLUIDIC APPROACH FOR SURFACE MODIFICATION OF MESOPOROUS SILICA NANOPARTICLES	28
^{1, 2} <i>Küçüktürkmen, B., </i> ² <i>Rosenholm, JM.</i>	
OP011: POLYELECTROLYTE COMPLEX NANOPARTICLES-FILLED ENTERIC-COATED CAPSULES FOR ORAL INSULIN DELIVERY.....	29
¹ <i>Devrim, B., </i> ² <i>Arpaç, B., </i> ¹ <i>Küçüktürkmen, B., </i> ³ <i>Özakça, I., </i> ¹ <i>Bozkır, A.</i>	
OP012: FORMULATION AND CHARACTERIZATION OF RESVERATROL LOADED SELF-MICROEMULSIFYING DRUG DELIVERY SYSTEMS (SMEDDS) FOR TOPICAL DRUG DELIVERY	30
¹ <i>Samancı, B., </i> ¹ <i>Yener, FG., </i> ² <i>Değim, İT.</i>	
OP013: DEVELOPMENT OF A PRINTABLE COATING FILAMENT FOR 3D COLON-TARGETING TABLETS	30
¹ <i>Duran, C., </i> ¹ <i>Sarısaltık Yaşın, D., </i> ² <i>Takka, S.</i>	
OP014: NICLOSAMIDE LOADED NIOSOME FOR TOPICAL APPLICATIONS: DEVELOPMENT AND IN VITRO CHARACTERIZATION	31
¹ <i>Yetgin, C., </i> ² <i>Citlak, H., </i> ^{1,3} <i>Coban, O.</i>	
OP015: PRECLINICAL DEVELOPMENT OF AN INJECTABLE MULTIPURPOSE PREVENTION TECHNOLOGY (MPT) FORMULATION	31
^{1,2} <i>Haeck, C.M., </i> ¹ <i>Boyd, P., </i> ³ <i>Dimant, N., </i> ³ <i>Desjardins, D., </i> ^{1,*} <i>Malcolm, R.K.</i>	
OP016: HOW USEFUL ARE MICROSCOPIC TECHNIQUES TO PREDICT DRUG RELEASE PROFILE FROM THE LIPID MICROPARTICLES	32
¹ <i>Wolska, E., </i> ¹ <i>Sznitowska, M.</i>	
OP017: PICKERING EMULSIONS STABILIZED BY CYCLODEXTRIN DERIVATIVES FOR TREATMENT OF ATOPIC DERMATITIS: OPTIMIZATION AND <i>IN VITRO</i> CHARACTERIZATION.....	33
¹ <i>Aydilek, N., </i> ¹ <i>Kahraman, E., </i> ¹ <i>Güngör, S.</i>	
OP018: DEVELOPMENT AND IN-VITRO IN-VIVO EVALUATION OF HYDROPHILIC GEL FORMULATIONS FOR TREATMENT OF KERATOCONUS BY NON-INVASIVE TECHNIQUE	33
¹ <i>Aytekin, E., </i> ² <i>Polat, HK., </i> ¹ <i>Bozdag Pehlivan, S., </i> ¹ <i>Çalış, S.</i>	
OP019: PREPARATION AND CHARACTERIZATION OF BIGEL SYSTEMS CONTAINING CICLOPIROX AND UREA	34
¹ <i>Kodan, E., </i> ¹ <i>Tırnaklı, F.</i>	
OP020: STABILITY STUDIES ON MEDICAL DEVICE PREPARED BY VANCOMYCIN-LOADED BONE CEMENT	35
¹ <i>Zanbak Çotaoğlu, EM., </i> ¹ <i>Köse Özkan, C., </i> ¹ <i>Eşim, O., </i> ² <i>Kıymacı, ME., </i> ² <i>Ünal, N., </i> ¹ <i>Savaşer A., </i> ¹ <i>Özkan, Y.</i>	
OP021: PREPARATION AND IN VITRO EVALUATION OF APO-E MODIFIED SOLID LIPID NANOPARTICLES FOR DELIVERY OF HUMANIN PEPTIDE	35
¹ <i>Topal, GR., </i> ² <i>Mészáros, M., </i> ² <i>Porkoláb, G., </i> ² <i>Szecskó, A., </i> ³ <i>Küçüktürkmen, B., </i> ³ <i>Öz, UC., </i> ² <i>Deli, MA., </i> ² <i>Veszelka, S., </i> ³ <i>Bozkır, A.</i>	
OP022: DESIGN, FABRICATION AND CHARACTERIZATION OF SURFACE MODIFIED HALLOYSITE/POLYMER NANOCOMPOSITE AND ITS 5-FLUOROURACIL CONJUGATES.....	36
¹ <i>Üner, G., </i> ² <i>Karakus, G., </i> ³ <i>Kaplan Can, H.</i>	

OP023:	CHEMOSENSITIVE EVALUATION OF METHOTREXATE LOADED NIOSOMES ON BURKITT LYMPHOMA CELLS	36
	¹ <i>Demirbolat, GM.,</i> ² <i>Ergul, M.</i>	
OP024:	DESIGN AND EVALUATION OF SEMI-SOLID LIPID NANOPARTICLES AS NOVEL NANOCOSMECEUTICALS	37
	<i>Amasya, G.</i>	
OP025:	ROBUST FORMULATION DESIGN USING COMPACTION SIMULATOR AND QBD APPROACH.....	37
	¹ <i>Özalp, Y.,</i> ¹ <i>Khamis, H.,</i> ¹ <i>Jiwa, N.,</i> ² <i>Mesut, B.,</i> ³ <i>Aksu, B.</i>	
OP026:	LIPOSOMAL ANTIAGING FORMULATION STUDIES CONTAINING SOME PROBIOTIC COMBINATION.....	38
	^{1,2,3} <i>Aslan, I.</i>	
OP027:	PRODUCTION AND CHARACTERIZATION OF OMEPRAZOLE LOADED NA ALGINATE/POLYVINYLPYRROLIDONE FILMS BY ELECTROSPINNING AND SOLVENT CASTING TECHNIQUES.....	39
	<i>Ortaso, JB., Uner, B., Tas, C.</i>	
OP028:	DETECTION OF REACTIVE OXYGEN SPECIES IN SKIN WITH MICRONEEDLES ...	40
	<i>Ozturk Atar, K.</i>	
OP029:	PREPARATION OF SILK FIBROIN NANOPARTICLES FROM BOMBYX MORI COCOONS BY DOE APPROACH.....	40
	<i>Birer, M., Yıldız, A., Acartürk, F.</i>	
OP030:	<i>IN VITRO INCORPORATION STUDIES OF</i> ^{99m} Tc-IBANDRONATE SODIUM <i>ON BONE CANCER CELL LINE.....</i>	41
	<i>Ekinci, M., İlem-Özdemir, D., Özgenç, E., Gündoğdu, E.</i>	
OP031:	DEVELOPMENT OF THE MICELLAR BASED OCULAR <i>IN SITU</i> GELLING SYSTEMS OF POSACONAZOLE WITH QUALITY BY DESIGN (QbD) APPROACH ...	41
	<i>Durgun, ME., Mesut, B., Güngör, S., Özsoy, Y.</i>	
OP032:	^{99m} Tc-LABELED, COLISTIN ENCAPSULATED, THERANOSTIC LIPOSOMES.....	42
	¹ <i>Karpuz, M.,</i> ² <i>Ozgenc, E.,</i> ² <i>Atlihan-Gundogdu, E.,</i> ³ <i>Senyigit, Z.</i>	
OP033:	DEVELOPMENT OF PLGA NANOPARTICLES TO PROMOTE ALVEOLAR BONE REGENERATION	43
	^{1,2} <i>İlhan, M.,</i> ¹ <i>Kılıçarslan, M.,</i> ³ <i>Alcigir, ME.,</i> ⁴ <i>Bagis, N.,</i> ⁵ <i>Ekim, O.,</i> ⁶ <i>Orhan, K.</i>	
OP034:	A NEW ORODISPERISIBLE TABLET FORMULATION OF AN ANTIHYPERTENSIVE DRUG.....	43
	^{1,2} <i>Gultekin, Y.,</i> ³ <i>Ozturk, N.,</i> ⁴ <i>Sahin, G.,</i> ² <i>Pezik, E.,</i> ⁵ <i>Kara, A.,</i> ² <i>Vural, I.</i>	
OP035:	DEVELOPMENT AND CHARACTERIZATION OF ERLOTINIB-RANDOMLY METHYLATED- β -CYCLODEXTRIN COMPLEX FOR THE TREATMENT OF NON-SMALL LUNG CANCER	44
	¹ <i>Erdoğan, N.,</i> ¹ <i>Akkın, S.,</i> ² <i>Varan, G.,</i> ¹ <i>Bilensoy, E.</i>	
OP036:	DESIGN OF DEXPANTHENOL LOADED ORALLY DISINTEGRATING FILMS.....	45
	^{1,2} <i>Kalfa, N.,</i> ¹ <i>İnal, Ö.</i>	
OP037:	EVALUATION AND COMPARISON OF β -CYCLODEXTRIN DERIVATIVES ON AQUEOUS SOLUBILITY OF DESLORATADINE	45
	<i>Çakmakyan, Ö., Tuğcu-Demiröz, F., Teksin, ZS.</i>	
OP038:	OPTIMIZATION OF LIDOCAINE BASE NANOSUSPENSIONS WITH EXPERIMENTAL DESIGN.....	46
	^{1,2} <i>Çulcu, Ö.,</i> ¹ <i>İlbasmis-Tamer, S.,</i> ¹ <i>Tırnaklı, F.</i>	

OP039:	DEVELOPMENT AND IN VITRO CHARACTERIZATION OF PREGABALIN LOADED NANOPARTICULAR SYSTEM	46
	<i>Sevinc Ozakar, R., Ozakar, E.</i>	
OP040:	EVALUATION OF IN VITRO PERMEABILITY OF AN ANTIVIRAL DRUG, FAVIPIRAVIR, FOR BCS CLASSIFICATION	47
	<i>¹Timur, SS., ²Eroglu, H.</i>	
OP041:	ACE2 LOADED CATIONIC LIPOSOMES FOR COVID-19 TREATMENT	48
	<i>¹Arisoy, S., ²Koçtaş, M., ³Çomoğlu, T.</i>	
OP042:	IMMUNOLOGICAL EFFECTS OF A NEW DEVELOPED CYCLOSPORINE A NANOSUSPENSION IN RATS FOR ORAL ADMINISTRATION.....	48
	<i>^{1,2}Gülbağ Pınar, S., ³Tan, Ç., ⁴Atak Yücel, A., ^{1,5}Çelebi, N.</i>	
OP043:	ORGANOTYPIC BRAIN SLICE CULTURES.....	49
	<i>Gulsun, T.</i>	
OP044:	DEVELOPMENT OF AN IN VIVO TEST METHOD FOR THE NASAL DELIVERY OF LYOSPHERES®	50
	<i>^{1,2}Serim, TM., ^{2,3}Lamprecht, A.</i>	
OP045:	CURCUMIN LOADED SEMISOLID SLN DISPERSIONS: FORMULATION OPTIMIZATION AND IN VIVO EVALUATION	50
	<i>¹Sen, A., ²Badilli, U., ³Yegen, G., ⁴Güven, B., ³Aksu, B., ⁴Onay-Besikci, A.</i>	
OP046:	DEVELOPMENT OF THYMOQUINONE LOADED NANO-FORMULATIONS VIA CENTRAL COMPOSITE DESIGN	51
	<i>Öz, UC., Bozkır, A.</i>	
OP047:	HUMAN SERUM ALBUMIN NANOPARTICLES FOR TARGETED CANCER THERAPY	52
	<i>Akdağ, Y., Geyik, ZM.</i>	
OP048:	ELECTROSPUN NANOFIBERS AS ORAL FAST-DISSOLVING DELIVERY SYSTEM OF RISPERIDONE.....	52
	<i>Turanlı, Y., Birer, M., Acartürk, F.</i>	
OP049:	PREPARATION AND CHARACTERIZATION OF TENOFOVIR DISOPROXIL FUMARATE LOADED NANOFIBER FOR VAGINAL DELIVERY.....	53
	<i>¹Dik, Z., ²Saar, S., ²Tuğcu-Demiröz, F.</i>	
OP050:	LABEL-FREE DETECTION of miRNA-34a by CARBON NANOFIBER ENRICHED SCREEN-PRINTED ELECTRODES.....	53
	<i>^{1,2}Eksin, E., ^{1,3}Congur, G., ¹Erdem, A.</i>	
OP051:	POLYETHYLENEIMINE FUNCTIONALIZED CRYOGEL MEMBRANES AS A CONTROLLED RELEASE SYSTEM	54
	<i>¹Çetin, K.</i>	
OP052:	MULTIPLE-TARGETING LIGANDS AGAINST PROSTATE CANCER: EFFECT ON BOTH AKR1C3 ENZYME AND ANDROGEN RECEPTOR.....	55
	<i>¹Pippione, AC., ²Kılıç-Kurt, Z., ¹Sainas, S., ¹Rolando, B., ¹Kovachka, S., ¹Spraklis, F., ³Buschini, A., ³Montalbano, S., ¹Oliaro Bosso, S., ¹Boschi, D., ¹Lolli, ML.</i>	
OP053:	FUNCTIONALIZED NANOPARTICLES AS POTENTIAL ANTIBIOFILM AGENTS	55
	<i>¹Gelain, A., ¹Mori, M., ¹Meneghetti, F., ^{2,3}Molino, P., ^{2,3}Hayes, P., ¹Villa, S.</i>	
OP054:	SYNTHESIS OF NOVEL 1-BENZYL-2-SUBSTITUTED-BENZIMIDAZOLE-5-SULFONAMIDE DERIVATIVES AND INVESTIGATION OF THEIR EFFECTS ON CHOLINESTERASES AND CARBONIC ANHYDRASE ENZYMES.....	56
	<i>¹Er, A., ²Eroglu Y., ³Bozbey, İ., ⁴Türkeş, C.</i>	

OP055:	NEXT GENERATION MONOCLONAL ANTIBODIES: NOVEL APPROACH TO ATTAIN DIAGNOSTIC AND THERAPEUTIC IMMUNOCONJUGATES	56
	¹ Alkhawaja, B., ² Watts, AG., ³ Van Den Elsen, J.	
OP056:	NEW INHIBITORS OF THE INDUCIBLE NITRIC OXIDE SYNTHASE AS ANTICANCER AND ANTIINFLAMMATORY AGENTS.....	57
	¹ Maccallini, C., ¹ Gallorini, M., ² Bellezza, I., ¹ Cataldi, A., ¹ Amoroso, R.	
OP057:	NEUROMODULATORY ACTIVITY ON THE CANNABINOIDERGIC SYSTEM BY NEW PYRAZOLE STYRYLQUINAZOLINONES	58
	¹ Plescia, F., ² Plescia, F., ² Cannizzaro, C., ¹ Raffa, D.	
OP058:	SYNTHESIS OF NEW PYRAZOLINE DERIVATIVES AND THEIR ANTICANCER ACTIVITIES.....	58
	¹ Tok, F., ² Çevik, Ö.	
OP059:	SYNTHESIS AND STRUCTURE ELUCIDATION OF NEW FENAMATE THIOSEMICARBAZIDE	59
	^{1,2} Coşkun, GP.	
OP060:	INVESTIGATION OF THE ANTIBACTERIAL EFFECTS OF SOME SCHIFF BASES COMPOUNDS CONTAINING NAPHTHALENE AND INDOLE RINGS.....	59
	¹ Shirinzadeh, H.	
OP061:	SYNTHESIS OF NOVEL HYDRAZONE DERIVATIVES AND EVALUATION OF THEIR INHIBITORY ACTIVITIES AGAINST MONOAMINE OXIDASES AND β -SECRETASE	60
	¹ Sellitepe, HE., ¹ Aksel, AB.	
OP062:	LIPASE INHIBITOR ACTIVITY AND MOLECULAR MODELLING STUDIES OF NEW PYRIDAZINONE DERIVATIVES	60
	¹ Alagöz, MA., ² Doğan, İS., ³ Şener, SÖ., ¹ Özdemir, Z.	
OP063:	SYNTHESIS AND HUMAN CARBONIC ANHYDRASE INHIBITION STUDIES OF SOME 1,3,4-THIADIAZOLES	61
	¹ Demir-Yazıcı, K., ¹ Güzel-Akdemir, Ö.	
OP064:	SYNTHESIS AND ANTIPROLIFERATIVE ACTIVITY OF SELENIUM CONTAINING COMPOUNDS	62
	¹ Sancinetto, L., ² Krasowska, D., ² Drabowicz, J., ³ Cieślak, M., ⁴ Iraci, N., ¹ Santi, C.	
OP065:	INSIGHT INTO REDOX PROPERTIES OF SOME SELENIDES AND DISELENIDES	62
	¹ Mangiavacchi, F., ¹ Liviabella, D., ¹ Della Rina, L., ¹ Sancinetto, L., ¹ Marini, F., ¹ Santi, C.	
OP066:	SYNTHESIS, ANTINEOPLASTIC ACTIVITY AND MOLECULAR DOCKING STUDIES OF NOVEL INDOLE-THIAZOLIDINEDIONE DERIVATIVES.....	63
	¹ Kısla, MM., ¹ Zengin-Karadayı, F., ¹ Baran, S., ² Dogan, TS., ² Mutlu, P., ¹ Ates-Alagoz, Z.	
OP067:	THIOUREA-BASED INHIBITORS OF <i>MYCOBACTERIUM TUBERCULOSIS</i> GROWTH AND ENOYL ACYL CARRIER PROTEIN REDUCTASE	63
	¹ Doğan, ŞD., ¹ Doğan, H., ² Krishna, VS., ³ Lherbet, C., ² Sriram, D., ⁴ Gündüz, MG.	
OP068:	MANDELIC ACID-BASED NOVEL SPIROTHIAZOLIDINONES: SYNTHESIS, ANTIMYCOBACTERIAL ACTIVITY AND MOLECULAR MODELLING STUDIES	64
	¹ Trawally, M., ¹ Demir-Yazıcı, K., ^{2,3} Dingiş-Birgül, SI., ² Akdemir, A., ¹ Güzel-Akdemir, Ö.	
OP069:	NOVEL 1,2,4-TRIAZOLES FROM IBUPROFEN AS POTENTIAL mPGES-1 INHIBITORS: SYNTHESIS, <i>IN VITRO</i> AND <i>IN SILICO</i> STUDIES	65
	¹ Kulabas, N., ² Bilgin, YN., ³ Çiftçi, G., ³ Yelekçi, K., ⁴ Gürboğa, M., ⁴ Bingöl Özakpinar, Ö., ¹ Küçükgüzel, İ.	

OP070:	DESIGN SYNTHESIS AND IN VITRO BIOLOGICAL ACTIVITIES OF NEW 6,8,9-TRISUBSTITUTED PURINE DERIVATIVES AS PROMISING HSPs INHIBITORS.....	65
	¹ Kul, P., ¹ Tuncbilek, M., ² Ergul, M., ³ Yenilmez Tunoglu, EN., ⁴ Tutar, Y.	
OP071:	IDENTIFICATION OF A POTENT INDOLE N-OXIDE DERIVATIVE HIF PHD2 INHIBITOR THROUGH HYBRIT VIRTUAL SCREENING	66
	¹ Sari, S., ² Tumber, A.	
OP072:	A NOVEL GREEN SYNTESIS OF NANO/MICRO PARTICLES DIRECTLY FROM CITRUS SINENSIS L. PEEL EXTRACTS AND THEIR USE IN BIOMEDICAL APPLICATIONS	66
	¹ Butun Sengel, S., ² Goger, G., ³ Butun, V.	
OP073:	DESIGN, SYNTHESIS, ADME AND MOLECULAR DOCKING STUDIES OF NOVEL UREA AND SULFONAMIDE DERIVATIVES OF ISATINE AS POTENTIAL ANTICANCER AGENTS	67
	¹ Demirel, UU., ^{2,3} Karaman, FE., ¹ Tanol, M., ³ Özden, S., ⁴ Göker, H., ² Ölgen, S*.	
OP074:	DEVELOPMENT OF NON-STEROIDAL AMINOTHIAZOLE ANALOGS ACTIVE ON MCF7 CELL LINE AND AROMATASE ENZYME	68
	¹ Sahin, Z.	
OP75:	THE EFFECT OF SACUBITRIL/VALSARTAN ON PROTEIN EXPRESSION OF DIASTOLIC COMPONENTS IN HFD/STZ INDUCED DIABETIC RAT HEART	68
	^{1,3} Erdogan, BR., ^{2,3} Yesilyurt, ZE., ³ Karaomerlioglu, I., ³ Muderrisoglu, AE., ³ Arioglu Inan, E.	
OP076:	THE SYNERGIC EFFECTS OF AGOMELATINE ON THE ANTICANCER POTENTIAL OF DOXORUBICIN IN MCF-7 BREAST CANCER CELLS	69
	¹ Ozkemahli, G., ² Dincer, B.	
OP077:	THE ROLE OF NEBIVOLOL ON ERECTILE DYSFUNCTION IN RATS WITH HEART FAILURE	70
	¹ Mercanoglu, G., ² Gumrukcu, G., ³ Macit, Ç.	
OP078:	INVESTIGATION OF DRUG-DRUG INTERACTION OF FAVIPIRAVIR WITH ALLOPURINOL AND VERAPAMIL USING PHARMACOKINETIC PARAMETERS.....	70
	¹ Askin Ozek, D., ² Keskin, Z., ³ Yuce, H., ³ Basak Turkmen, N., ³ Unuvar, S., ³ Aslan, S.	
OP079:	ROLE OF TOLL- LIKE RECEPTOR2 SIGNALING IN RAT MODEL OF CAVERNOUS NERVE INJURY-INDUCED ERECTILE DYSFUNCTION.....	71
	¹ Barut, EN., ¹ Engin, S., ^{1,2} Kaya Yaşar, Y., ^{1,2} Sezen, SF.	
OP080:	A DROSOPHILA APPROACH TO STUDY THE EFFECTS OF ATYPICAL ANTIPSYCHOTIC DRUGS	71
	¹ Milani, D., ¹ Forgiarini, A., ² Gumeni, S., ¹ Comai, S., ¹ Guarato, G., ¹ Orso, G.	
OP081:	A DROSOPHILA BASED APPROACH TO DEVELOP SPECIES-SELECTIVE VERTEBRATE DRUGS	72
	¹ Guarato, G., ¹ Forgiarini, A., ¹ Orso, G.	
OP082:	GENDER DIFFERENCES IN β 3-ADRENOCEPTOR-MEDIATED CARDIAC REMODELING	73
	^{1, 2} Kayki Mutlu, G.	
OP083:	THE CONTRIBUTION OF ADRENERGIC AND SEROTONERGIC RECEPTORS IN THE ANALGESIC EFFECT OF QUERCETIN	73
	¹ Jafarova, Z., ¹ Eken, H., ² Bektaş, N., ² Arslan, R.	
OP084:	INVESTIGATION OF EFFECT OF INACTIVE PARAPOXVIRUS IMMUNOMODULATOR ON LEUKOCYTE PROLIFERATION	74
	¹ Zengin, H., ¹ Dağoğlu, G., ¹ Tanyıldızı, S., ¹ Keskin, Z., ² Vezir, Y., ² Ünal, N.	

OP085:	THE EFFECT OF METHYLENE BLUE TREATMENT ON COGNITIVE FUNCTIONS IN THE D-GALACTOSE-INDUCED AGE RELATED DEMENTIA MOUSE MODEL	74
	<i>Kazkayasi, I., Telli, G.</i>	
OP086:	THE EFFECT OF 4-PHENYLBUTYRIC ACID ON HYPERTENSION-INDUCED CARDIAC IMPAIRMENTS	75
	<i>¹Bal, NB., ¹Han, S., ¹Uludag, MO., ²Demirel-Yilmaz, E.</i>	
OP087:	CONTRIBUTION OF CANNABINOID SYSTEM TO THE ANTIHYPERALGESIC EFFECTS OF ANTIPILEPTIC DRUGS	76
	<i>¹Bektaş Turkmen, N., ¹Alyu, F., ²Okçay, Y., ¹Arslan, R.</i>	
OP088:	THE EFFECTS OF HYDROGEN SULFIDE DONORS ON ERK AND WNT SIGNALING PATHWAYS IN AN <i>IN VITRO</i> LIPOPOLYSACCHARIDE-INDUCED AIRWAY INFLAMMATION MODEL IN MICE	76
	<i>¹Karaman, Y., ²Kaya-Yasar Y., ¹Bozkurt, TE., ¹Sahin-Erdemli I.</i>	
OP089:	EFFECT OF FLUVOXAMINE ON THE PHARMACOKINETICS OF PARACETAMOL IN MICE.....	77
	<i>¹Karakoy, Z., ¹Cetin, G., ²Corum, O., ³Uneý, K.</i>	
OP090:	EVALUATION OF PARENTAL KNOWLEDGE, ATTITUDES AND PRACTICES REGARDING ANTIBIOTIC USE IN ACUTE UPPER RESPIRATORY TRACT INFECTIONS IN CHILDREN IN A TERTIARY CARE HOSPITAL IN TURKEY.....	78
	<i>¹Albayrak, A., ²Karakas-Mutlu N., ³Karahalil, B.</i>	
OP091:	OPINIONS OF CANCER PATIENTS TOWARDS THE COVID-19 VACCINE.....	78
	<i>¹Aras Atik, E., ¹Tecen-Yucel, K., ¹Ozdemir, N., ¹Bayraktar-Ekincioglu, A., ²Akin, S.</i>	
OP092:	EVALUATION OF THE PSYCHOLOGICAL BUDERN OF COVID-19 PANDEMIC ON YOUNG ADULT POPULATION	79
	<i>¹Aksoy, N., ²Ongun, E.</i>	
OP093:	ASSESSMENT OF THE PROPER INHALER TECHNIQUE IN ASTHMA AND COPD PATIENTS.....	80
	<i>¹Durmus, M., ²Gok, S., ³Bahcecioglu, OF.,⁴Gun, ZU., ⁵Hacielyagil, SS.</i>	
OP094:	THE EFFECTS OF CARVACROL AND EPIGALLOCATECHIN GALLATE ON DRUG RESISTANCE IN NEUROBLASTOMA CELL LINE	80
	<i>¹Oltulu, C., ²Bakar, E., ³Akinci, M.</i>	
OP095:	THE ANTIANGIOGENIC ACTIVITY OF METFORMIN IN HT29-HUVEC CO-CULTURE: INVOLVEMENT OF miR-21 EXPRESSION	81
	<i>Sevim, Ç.</i>	
OP096:	COMPARATIVE CYSTEINE S-CONJUGATE β -LYASE ACTIVITIES OF DIFFERENT ORGANS TOWARDS PARACETAMOL IN MICE	81
	<i>Karakuş F., Atmaca K., Aladağ B., Orhan H.</i>	
OP097:	SMOKING BEHAVIORS IN COVID 19: AN ONLINE SURVEY AMONG UNIVERSITY STUDENTS	749
	<i>Çelik, FG., Demirel, G.</i>	82
OP098:	EFFECTS OF PERIOSTIN, TENASCIN-C, YKL-40, TETRAHYDROBIOPTERIN ON THE LIVER CANCER CELL LINE	82
	<i>Yüce, H., Türkmen, N., Ünüvar, S.</i>	
OP099:	EVALUATION OF ANTIOXIDANT, ANALGESIC, ANTI-INFLAMMATORY AND ANTISPASMODIC ACTIVITY AND GENOTOXIC EFFECT OF <i>MICROMERIA FRUTICOSA</i> SUBSP <i>BRACHYCALYX</i> <i>IN VITRO</i> AND <i>IN VIVO</i>	83
	<i>¹Celikkol, I., ²Beceran, A., ³Kabasakal, L., ⁴Taskin, T., ⁵Aydemir, S.</i>	

OP100: EVALUATION OF THE IN-VITRO CYTOTOXIC ACTIVITY OF SUNSET YELLOW IN ACUTE AND CHRONIC DOSING SCENARIOS	84
¹ Sönmez, K., ¹ Dural, E., ² Süzen, HS.	
OP101: THE IMPACT OF THE CYTOCHROME P450 3A4 (CYP3A4*22) POLYMORPHISM ON TACROLIMUS DOSE REQUIREMENTS AND EXPOSURE DURING EARLY PERIOD FOLLOWING KIDNEY TRANSPLANTATION.....	84
¹ Demirbugen Oz, M., ² Keven, K., ¹ Süzen, HS.	
OP102: A PRELIMINARY STUDY; IN VITRO ANTICANCER ACTIVITY OF PATULIN ON HEP3B AND A549 CELLS	85
Türkmen Başak, N., Yüce, H., Ünvar, S.	
OP103: DIRECT PEPTIDE REACTIVITY ASSAY (DPRA) FOR MEASURING SKIN SENSITIZATION POTENTIALS OF COSMETIC INGREDIENTS	85
¹ Kavas, P., ¹ Ulker, OC., ² Gokbulut, A., ¹ Esen, B.	
OP104: THE EFFECTS OF DIFFERENT BISPHENOL DERIVATIVES ON OXIDATIVE STRESS, DNA DAMAGE AND DNA REPAIR IN RWPE-1 CELLS: A COMPARATIVE STUDY	86
¹ Kose, O., ^{2,3} Rachidi, W., ^{2,3} Beal, D., ⁴ Erkekoglu, P., ⁵ Fayyad-Kazan, H., ¹ Kocer-Gumusel, B.	
OP105: BIOCOMPATIBILITY OF PULP CAPPING MATERIALS ON L929 MOUSE FIBROBLAST CELLS	87
¹ Chinheya, RM., ³ Yilmaz, M., ² Üstündag, A., ² Ipek, S., ² Duydu, Y., ³ Aydin, C.	
OP106: EVALUATION OF IN VITRO CYTOTOXIC ACTIVITY OF HYDROXYCHLOROQUINE.....	87
¹ Önal, Ş., ¹ Dural, E., ² Süzen, HS.	
OP107: GENISTEIN AND 5-FLUOROURACIL ENHANCES TRAIL MEDIATED APOPTOSIS VIA INHIBITION OF XIAP AND DCR1 IN SW480 CELLS.....	88
¹ Çal, T., ¹ Aydin Dilsiz, S., ² Canpinar, H., ¹ Ündeğer Bucurgat, Ü.	
OP108: POSSIBLE EFFECT OF CHELATION TREATMENT ON METABOLOMIC AND LIPIDOMIC ANALYSIS IN LEAD EXPOSURE	89
¹ Çetin, T., ² Samadi, A., ³ Reçber, T., ² Eser, B., ² Yalcinkaya, A., ² Öztaş, Y., ³ Nemutlu, E., ² Lay, I., ¹ Sabuncuoğlu, S.	
OP109: MALE REPRODUCTIVE SYSTEM TOXICITY OF DI (2-ETHYLHEXYL) PHTHALATE: A COMPARISON IN TERMS OF EXPOSURE TIME AND ROUTE	89
¹ Sur, U., ¹ Balci, A., ^{1,2} Yirun, A., ³ Ozkemahli, G., ⁴ Baysal, E., ⁵ Yersal, N., ⁶ Tan, E., ⁴ Zeybek, ND., ¹ Erkekoglu, P., ⁷ Kocer-Gumusel, B.	
OP110: HEPATOPROTECTIVE EFFECTS OF SIDERITIS CONGESTA AGAINST APAP-INDUCED LIVER INJURY IN HEPG2 CELLS	90
¹ Özhan, Y., ² Güzelmeriç, E., ³ Kan, Y., ¹ Aydin, A., ¹ Sipahi, H.	
OP111: TAMOXIFEN AND SODIUM THIOSULPHATE THERAPEUTIC EFFECT IN RATS WITH LIVER DAMAGE CAUSED BY EXPERIMENTAL <i>Xanthium strumarium</i> POISONING.....	91
¹ Keskin, Z., ¹ Dağoglu, G., ² Eröksüz, Y., ¹ Korkak, FA., ¹ Tanyıldızı, S.	
OP112: DETERMINATION OF THE ELECTROCHEMICAL BEHAVIOUR OF AN ANTICANCER DRUG IN PHARMACEUTICAL AND BIOLOGICAL SAMPLES	91
¹ Cetinkaya, A., ¹ Topal BD., ² Atici, EB., ¹ Ozkan, SA.	
OP113: VOLTAMMETRIC DETERMINATION OF EPIRUBICIN BY A MODIFIED GLASSY CARBON ELECTRODE.....	92
¹ Ates, AK., ² Erk, N.	

OP114: IN VITRO DNA AND BSA INTERACTION OF ANTI VIRAL DRUG TENOFOVIR BY SPECTRAL METHODS	92
Oznur, A., Satana Kara, HE.	
OP115: A SIMPLE AND SENSITIVE ELECTROANALYSIS OF NILOTINIB IN BIOLOGICAL SAMPLES IN THE PRESENCE OF SODIUM LAURYL SULPHATE	93
<u>Dogan-Topal, B., Sener, CE., Ozkan, SA.</u>	
OP116: DEVELOPMENT OF A NEW HPLC METHOD FOR THE DETERMINATION OF MESALAZINE IN HUMAN PLASMA AND APPLICATION TO A PHARMACOKINETIC STUDY	93
¹ Ceylan, B., ² Tekkeli Kepkci, E., ³ Önal, C.	
OP117: COMPARATIVE HPLC-PDA AND LC-MS/MS APPROACHES OSBs LEVELS OF SIMULATED ARTIFICIAL BODY FLUIDS AND SIGNIFICANCE OF RAW DATA FOR CHEMOMETRIC DISCRIMINATION OF OSBs	94
¹ Sengul, A., ² Yengin, C., ³ Egrilmez, S., ¹ Kilinc, E.	
OP118: DESIGN OF A NOVEL NANOSENSOR FOR THE DETERMINATION OF CARDIAC INOTROPE DRUG MILRINONE	95
¹ Unal, DN., ¹ Selcuk, O., ² Süslü, I., ¹ Uslu, B.	
OP119: ASSESSMENT OF ANTIOXIDANT AND ANTICANCER ACTIVITIES OF ACHILLEA PHRYGIA EXTRACT LOADED CHITOSAN NANOPARTICLES	95
¹ Taşkin, D., ² Doğan, M.	
OP120: ELECTROCHEMICAL ANALYSIS OF DAPAGLIFLOZIN USING BORON-DOPED DIAMOND ELECTRODE	96
^{1,2} Ozkan, E., ³ Ozcelikay, G., ³ Cetinkaya, A., ¹ Nemutlu, E., ¹ Kir, S., ³ Ozkan, SA.	
OP121: A NOVEL DESIGN OF GRAPHENE-BASED ELECTROCHEMICAL NANOSENSOR FOR THE DETECTION OF ANTIMETABOLITE ANTICANCER AGENTS	97
Er, E.	
OP122: POLY (HPBAs) FOR VOLTAMMETRIC DETERMINATION OF FLUORIDE IN DENTAL FORMULATIONS (DFs); PCA APPROACH.....	97
¹ Der, FG., ² Yalcin, G., ³ Ozcan Bulbul, E., ⁴ Ileri, H., ¹ Kilinc, E.	
OP123: ELECTROCHEMICAL DETERMINATION OF ANTINEOPLASTIC DRUG IN HUMAN PLASMA BY MODIFIED GLASSY CARBON ELECTRODE	98
¹ Mehmandoust, M., ¹ Erk, N., ² Tiris, G.	
OP124: AN ELECTROCHEMICAL PLATFORM BASED ON MAGNETIC/CHITOSAN NANOMATERIALS FOR DETERMINATION OF THE DAPAGLIFLOZIN IN DIFFERENT MATRICES	98
¹ Ozcelikay, G., ^{2,3} Ozkan, E., ¹ Cetinkaya, A., ² Nemutlu, E., ² Kir, S., ¹ Ozkan, SA.	
OP125: A SENSITIVE ELECTROCHEMICAL NON-ENZYMATİC HYDROGEN PEROXİDE SENSOR USING AuNPs-ERGO/POLY(INDOLE-5-CARBOXYLIC ACİD) NANOCOMPOSİTE	99
¹ Aydoğdu Tiğ, G., ² Zeybek, B.	
OP126: FABRIC PHASE SORPTİVE EXTRACTION FOLLOWED BY HPLC-PDA DETECTION FOR THE MONITORİNG OF PİRİMİCARB AND FENİTROTHİON PESTİCİDE RESİDİÜS.....	99
¹ Ulusoy, Hİ., ¹ Koseoglu, K., ² Kabir, A., ³ Ulusoy, S., ⁴ Locatelli, M.	
OP127: A NOVEL METHOD FOR ANALYTICAL DETERMINATION OF COVID-19 DRUG, FAVIPIRAVİR, İN TABLETS	100
¹ Evcil, I., ¹ Caglar-Andac, S., ² Pehlivanoglu, H.	

OP128:	SIMULTANEOUS DETERMINATION OF FEBUXOSTAT AND MONTELUKAST IN HUMAN PLASMA BY USING FABRIC PHASE SORPTIVE EXTRACTION AND HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	101
	¹ Gazioglu, I., ¹ Kepekci Tekkeli, SE., ² Kabir, A., ¹ Aslan, C.	
OP129:	SYNTHESIS OF COBALT OXIDE NANOPARTICLES FROM PLANT EXTRACT OF DURANTA RIPENS FOR THE SENSITIVE ELECTROCHEMICAL DETERMINATION OF TRAMADOL IN PHARMACEUTICAL FORMULATION	101
	¹ Palabıyık, İM., ² Memon, SA., ² Hassan, D., ² Buledi, JA., ² Solangi, AR., ³ Memon, SQ.	
OP130:	BIOINSPIRED DESIGN OF POROUS MOLECULARLY IMPRINTED NANOFILM FOR SELECTIVE AND SENSITIVE SENSING OF AN ANTICANCER DRUG RUXOLITINIB.....	102
	^{1,2} Corman, ME, ¹ Cetinkaya, A., ¹ Ozcelikay, G., ³ Ozgür, E., ⁴ Atici, EB., ⁵ Uzun, L., ¹ Ozkan, SA.	
OP131:	QUANTITATIVE PYRROLIDONYL ARYLAMIDASE ASSAY FOR GROUP A STREPTOCOCCUS PYOGENES DETECTION WITH IMAGE ANALYSIS	103
	¹ Eryilmaz, M., ² Boyaci, IH., ¹ Tamer, U.	
OP132:	MICROSAMPLING AND HRMS FOR THE ANALYSIS OF TRYPTOPHAN-DERIVED BIOMARKERS IN A MURINE MODEL OF AMYOTROPHIC LATERAL SCLEROSIS.	103
	¹ Protti, M., ² Volpi, C., ¹ Mercolini, L.	
OP133:	FABRICATION OF 2D-G-C ₃ N ₄ /SDS/GNPs AS AN ELECTROCHEMICAL SENSOR FOR BIOMEDICAL APPLICATION.....	104
	<i>Mehmandoust, M., Erk, N.</i>	
OP134:	QSRR-ANN MODELLING IN β-CD-MODIFIED RP-HPLC	104
	<i>Djajić, N., Krmar, J., Otašević, B., Malenović, A., Protić, A.</i>	
OP135:	ELECTROCHEMICAL INVESTIGATION OF SURFACTANT EFFECT ON THE ETODOLAC AND THIOCOLCHICOSIDE SIGNALS	105
	<i>Selcuk, O., Erkmen, C., Bozal-Palabıyık, B., Uslu, B.</i>	
OP136:	USE OF NOVEL BIOCHAR-DERIVATIZED MAGNETIC NANOCOMPOSITE AS MAGNETIC SOLID-PHASE EXTRACTION ADSORBENT FOR PRECONCENTRATION AND DETERMINATION OF SDZ BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY	106
	<i>Avci, NR., Oymak, T.</i>	
OP137:	NEW SCHIFF BASE LIGAND-COMPLEXES AS CARBONIC ANHYDRASE AND CHOLINESTERASE ENZYME INHIBITORS: SYNTHESIS, CHARACTERIZATION AND <i>IN VITRO / IN SILICO</i> EVALUATION.....	107
	<i>¹Tuna Yıldırım, S., ²Gügercin, RS., ³Duran HE., ⁴Türkeş, C.</i>	
OP138:	SPECTROPHOTOMETRIC and HPLC DETERMINATION OF NAFTIFINE HCL.....	107
	<i>Dermis, S., Civelek, Z.</i>	
OP139:	LC-MS/MS AND LC-DAD METHODS FOR ROBUST DETERMINATION OF GLYCEROL PHENYLBUTYRATE IN BIOLOGICAL FLUIDS AND HIGH-RESOLUTION MASS SPECTROMETRIC IDENTIFICATION OF FORCED DEGRADATION PRODUCT	108
	<i>^{1,2}Özcan, S., ^{1,2}Can, NÖ.</i>	
OP140:	DETAILED ELECTROCHEMICAL BEHAVIOR AND THERMODYNAMIC PARAMETERS OF ANTICANCER DRUG REGORAFENIB AND ITS SENSITIVE ELECTROANALYTICAL ASSAY IN BIOLOGICAL AND PHARMACEUTICAL SAMPLES	108
	<i>^{1,2}Doulache, M., ^{3,4}Kaya, SI., ⁴Cetinkaya, A., ³Bakırhan, KN., ²Trari, M., ⁴Ozkan, SA.</i>	

- OP141: EFFECT OF GEOGRAPHICAL DIFFERENCES ON THYMOQUINONE CONTENT AND CYTOTOXICITY OF BLACK CUMIN SEEDS 109
¹Işık, S., ²Yurdakok-Dikmen, B., ¹Garba Usman, A., ²Turker, E., ³Aslan Erdem, S., ¹Altunterim Erkan, S.
- OP142: MICROWAVE-ASSISTED *IN SITU* SYNTHESIS OF A NOVEL DEEP EUTECTIC SOLVENT FOR THE LIQUID-LIQUID MICROEXTRACTION OF BETA BLOCKERS . 110
¹Yıldırım, S., ²Sellitepe, HE.
- OP143: INHIBITION OF TYROSINASE BY NON-STEROIDAL ANTI-INFLAMMATORY DRUG: AN ELECTROCHEMICAL APPROACH..... 110
Kurbanoglu, S., Erkmen, C., Demir, Y., Uslu, B.
- OP144: CHROMATOGRAPHIC DETERMINATION OF IRINOTECAN RESIDUES IN URINE SAMPLES BY USING A NEW SYNTHESIZED SORBENT MATERIAL..... 111
¹Ulusoy, S., ²Tartaglia, A., ³Kabir, A., ⁴Ulusoy, Hİ., ²Locatelli M.,
- OP145: DEVELOPMENT OF ANALYTICAL METHOD FOR SENSITIVE SIMULTANES DETERMINATION OF AMITRIPTYLINE AND VENLAFAXINE BASED ON MAGNETIC PHASE EXTRACTION 111
^{1,2}Morgül, U., ¹Ulusoy, Hİ., ²Palabıyık, İM.
- OP146: THE PRELIMINARY ETHNOBOTANICAL STUDY OF GÖKÇEADA/ÇANAKKALE 112
¹Kızılaşlan-Hançer, Ç., ¹Sevgi, E., ²Eyi, H., ³Sancaklı, G.
- OP147: ESSENTIAL OIL COMPOSITION AND ANATOMICAL STRUCTURE OF *FERULA TINGITANA* L. (APIACEAE)..... 112
Ekşi Bona, G.
- OP148: COMPARATIVE LEAF ANATOMY OF SOME *OPOPANAX* SPECIES..... 113
Gümüşok, S., Kılıç, CS..
- OP149: DEVELOPMENT OF A CELLULAR MEMBRANE AFFINITY CHROMATOGRAPHY COLUMN CONTAINING IMMOBILIZED TRKB RECEPTORS FOR THE IDENTIFICATION OF PHYTOCHEMICALS MIMICKING THE EFFECTS OF BDNF .. 113
¹Arituluk, ZC., ²Maitra, U., ²Ciesla, L.
- OP150: *IN VITRO* BIOLOGICAL EFFECTS OF ENDEMIC ANATOLIAN SPOTTED NEWT DERMAL VENOM: A POTENTIAL ACTIVE PHARMACEUTICAL INGREDIENT (API) FOR DRUG DELIVERY SYSTEMS 114
^{1,2}Karış, M., ³Çimik, A., ⁴Gürel-Gürevin, E., ⁵Öztürk, AA., ⁶Kıyan, HT..
- OP151: PHYTOCHEMICAL ANALYSIS AND BIOLOGICAL ACTIVITY OF *NEPETA CADMEA* BOISS..... 115
¹Gökbulut, A., ¹Kalender, S., ²Deliorman Orhan, D., ²Özüpek, B., ³Yılmaz, G.
- OP152: METABOLOMICS AND ACETYLCHOLINESTERASE INHIBITORY ACTIVITY STUDIES ON *DACTYLIS GLOMERATE* L. AND *HORDEUM MURINUM* L. 115
¹Gonulalan, EM., ²Kahraman, C.
- OP153: A COMPARATIVE ANALYSIS ON ANTIOXIDANT PROPERTIES, PHENOLIC COMPOSITION AND HPTLC EXAMINATION OF *SIDERITIS SCARDICA* SPP. *SCARDICA* INFUSION AND HYDROALCOHOLIC EXTRACT 116
¹Bardakci, H., ²Yıldırım, EB., ¹Barak, TH.
- OP154: *SAMBUCUS EBULUS* L. VERSUS *S. NIGRA* L.: COMPARATIVE ASSESSMENT OF THE PHENOLIC COMPOSITIONS AND BIOACTIVITY PROFILES OF FLOWERS, LEAVES AND FRUITS..... 117
Guzelmeric, E., Celik, C., Yeşilada, E.
- OP155: THE RADICAL SCAVENGING ACTIVITIES AND ENZYME INHIBITION EFFECTS OF THE EXTRACTS FROM *ORIGANUM ONITES* L. 117
Ayaz, F., Eruygur, N.

OP156: IN THE FIGHT AGAINST BACTERIA: AERIAL PARTS OF <i>PEGANUM HARMALA</i> L.....	118
¹ <i>Guragac Dereli, FT.</i> , ² <i>Onem, E.</i> , ³ <i>Ozaydin, AG.</i>	
OP157: INVESTIGATION OF <i>in vitro</i> ANTIMICROBIAL EFFECTS OF <i>TRIPLEUROSPERMUM CALLOSUM</i> (BOISS. & HELDR.) E. HOSSAIN EXTRACTS ON URINARY SYSTEM PATHOGENS and <i>in vivo</i> TOXICITY IN <i>Caenorhabditis elegans</i> MODEL.....	118
¹ <i>Göger, G.</i> , ² <i>Aksoy, D.</i> , ³ <i>Göger, F.</i> , ⁴ <i>Köse, YB.</i> , ^{3,5} <i>Demirci, F.</i>	
OP158: LAMIACEAE MEMBERS USED IN ANATOLIA TRADITIONALLY FOR RESPIRATORY DISEASES FROM THE PERSPECTIVE OF BACTERIAL AND VIRAL INFECTIONS.....	119
<i>Zare, G., Diker NY., Tatlı Çankaya, İİ</i>	
OP159: COMPARATIVE STUDY OF ANTIHYALURONIDASE ACTIVITIES OF HYBRID NANOFLOWERS OF <i>ROSMARINUS OFFICINALIS</i> METHANOL PLANT EXTRACT	119
<i>Durbilmez, GD., Koca Çalışkan, U.</i>	
OP160: 5-LIPOXYGENASE ENZYME INHIBITORY ACTIVITY OF <i>ORIGANUM MINUTIFLORUM</i> O. SCHWARZ ET P.H. DAVIS VARIOUS EXTRACTS.....	120
¹ <i>Yıldız, G.</i> , ² <i>Temel, HE.</i> , ³ <i>Kirimer, N.</i>	
OP161: THE IMPORTANCE OF <i>DROSOPHILA MELANOGLASTER</i> AS A MODEL ORGANISM IN PHYTOCHEMICAL ACTIVITY BIOASSAY FOR NEUROLOGICAL DISEASES	121
<i>Emecen, G.</i>	
OP162: <i>NEPETA TRANSCAUASICA</i> GROSSH.: CHEMICAL COMPOSITION AND ALPHA GLUCOSIDASE INHIBITORY ACTIVITY OF ESSENTIAL OIL AND ANATOMICAL PROPERTIES OF DIFFERENT PARTS OF THE PLANT	121
¹ <i>Yuca, H.</i> , ² <i>Karakaya, S.</i> , ³ <i>Yılmaz, B.</i> , ¹ <i>Guvenalp, Z.</i>	
OP163: <i>IN VIVO</i> ANTI-ANGIOGENIC AND ANTI-INFLAMMATORY POTENTIALS OF R(+) OR S(-) LIMONENE LOADED EUDRAGIT® RS 100 NANOPARTICLES	122
¹ <i>Kıyan, HT.</i> , ² <i>Öztürk, AA.</i>	
OP164: SEARCH OF POTENTIAL MARINE NATURAL PRODUCTS AGAINST COVID-19....	123
¹ <i>Uras, IS.</i> , ^{2,3} <i>Ebada, SS.</i> , ^{4,5} <i>Konuklulgil, B.</i>	
OP165: THREE NEW ANTIMICROBIAL NATURAL COMPOUNDS FROM <i>Scorzonera aucheriana</i>	124
¹ <i>Erik, İ.</i> , ¹ <i>Yaylı, N.</i> , ² <i>Coşkunçelebi, K.</i> , ³ <i>Karaoglu, ŞA.</i>	
OP166: CYTOTOXIC ACTIVITY AND PHYTOCHEMICAL PROFILE of <i>PIMPINELLA ISAURICA MATTHEWS</i> ssp. <i>ISAURICA</i>	124
¹ <i>Taban Akça K.</i> , ¹ <i>Süntar İ.</i> , ² <i>Emerce E.</i> , ¹ <i>Gök HN.</i> , ³ <i>Tugay O.</i> , ⁴ <i>Gürbüz P.</i>	
OP167: POTENTIAL PHYTOCHEMICALS FOR THE TREATMENT OF PULMONARY ARTERIAL HYPERTENSION	125
<i>Yüzbaşıoğlu Baran, M.</i>	
OP168: A SURVEY ON PRACTICE OF DIETARY SUPPLEMENTS AND AROMATHERAPY DURING COVID-19 PANDEMIC IN TURKEY	125
<i>Mancak Karakus, M.</i> , <i>Koca Çalışkan, U.</i>	
OP169: BIOACTIVITY-GUIDED ISOLATION OF CYTOTOXIC COMPOUNDS FROM THE UNDERGROUND PARTS OF <i>VALERIANA ALLIARIIFOLIA</i> ADAMS.....	126
¹ <i>Erdoğan, M.</i> , ² <i>Aru, B.</i> , ³ <i>Taygun, U.</i> , ³ <i>Şimşek C.</i> , ¹ <i>Yeşilada, E.</i> , ² <i>Yanikkaya Demirel G.</i> , ¹ <i>Kırmızıbekmez, H.</i>	

OP170: IDENTIFICATION AND CHARACTERIZATION OF DESCHLORO-CHLOROTHRICIN OBTAINED FROM A LARGE NATURAL PRODUCT LIBRARY TARGETING AURORA A KINASE IN MULTIPLE MYELOMA.....	127
1 ² Özenver, N., ² Abdelfatah, S., ³ Klinger, A., ³ Fleischer, E., ² Efferth T.	
OP171: ANTIMICROBIAL EVALUATION OF JUNIPER BERRY (<i>Juniperus communis L.</i>) ESSENTIAL OIL COMBINATION WITH STANDARD ANTIMICROBIAL COMPOUNDS	127
1 ² Besirk, NS., ² Goger, G.	
OP172: CONSTITUENTS AND BIOLOGICAL ACTIVITY OF ENDEMIC CYNOGLOTTIS CHETIKIANA.....	128
1 ² Gündoğdu, S., ² Yüzbaşıoğlu Baran, M., ¹ Kuruüzüm-Uz, A.	
OP173: DETERMINATION OF CAFFEINE CONTENT IN WORLD COFFEES BY A NEW, VALIDATED HPLC METHOD AND INVESTIGATION OF THE RELATIONSHIP BETWEEN CAFFEINE CONTENT AND LIPASE INHIBITION.....	129
Sener, SO., Ozgen, U.	
OP174: ENZYME INHIBITORY AND PHYTOCHEMICAL STUDIES ON <i>Pistacia terebinthus</i> COLLECTED FROM DIFFERENT LOCATIONS	129
Pekacar, S., Deliorman Orhan, D.	
OP175: QUALITY-CONTROL OF <i>HYPERICUM PERFORATUM L.</i> PREPARATIONS SOLD IN HERBAL DROGSTORES AND PHARMACIES OF ADANA AND INVESTIGATION OF THEIR HYPERICIN AND HYPEROSIDE CONTENT BY HPLC.....	130
1 ² Şerbetçi, T., ² Yüzbaşıoğlu Çepni, E.	
OP176: QUANTIFICATION OF FATTY ACIDS IN CHIA SEED OILS OBTAINED WITH SFE-CO ₂ AND COLD PRESS TECHNIQUES	130
1 ² Darı, Y., ^{2,3} Yur, S., ⁴ Özek, G., ⁵ Uysal, Ü.D., ^{3,4} Özek, T.	
OP177: QUALITATIVE AND QUANTITATIVE ANALYSIS OF ISOORIENTIN IN <i>LINUM ARBOREUM</i> AND <i>LINUM FLAVUM SSP. SCABRINERVE</i>	131
1 ² Torun, Z., ^{1,2} Konuklugil, B.	
OP178: CYTOKINE-BASED ANTI-INFLAMMATORY STUDIES ON <i>Acanthus spinosus-L.</i>	132
Dogan, Z., Sarikaya-Aydin, S., Saracoglu, I.	
OP179: ESSENTIAL AND FIXED OILS OF SICILIAN PLANTS AS PHYTOTHERAPEUTIC SOURCES	132
1 ² Badalamenti, N., ^{1,2} Maurizio, B.	
OP180: ISOLATION OF SALMONELLA BACTERIOPHAGE IN POULTRY AND ITS CHARACTERIZATIONS	133
1 ² Unverdi, A., ² Erol, H.B., ² Kaskatepe, B.	
OP181: THE COMBINATORY ANTIFUNGAL ACTIVITY OF CURCUMIN AND QUERCETIN ON <i>CANDIDA SPP.</i>	134
Simsek, D., Altanlar, N.	
OP182: DETERMINATION OF VIRULENCE AND TRIAZOLE DRUG SUSCEPTIBILITY OF WILD TYPE AND FLUCONAZOLE ADAPTED STRAINS OF <i>MAGNUSIOMYCES CLAVATUS</i>	134
Kaplan, E.	
OP183: ANTIBIOFILM ACTIVITY OF TWO NEW GENERATION DISINFECTANTS	135
1 ² Sad Eldin, E., ¹ Gurpinar, SS., ² Kart, D., ¹ Eryilmaz, M.	
OP184: ANTIBIOFILM AND ANTIBACTERIAL EFFECTS OF METABOLITES OF <i>BACILLUS SP.</i> ISOLATED FROM SOIL.....	135

OP185:	ANTIBACTERIAL AND ANTIBIOFLIM ACTIVITY OF NISIN AGAINST METHICILLIN RESISTANT STAPHYLOCOCCUS AUREUS ISOLATES.....	136
	¹ Savluk, M., ¹ Kiymaci, ME., ² Kaskatepe, B.	
OP186:	ANTIBACTERIAL ACTIVITY OF SOME ANTIDEPRESSANT ACTIVE SUBSTANCES AGAINST CLINICAL ACINETOBACTER BAUMANNII ISOLATES....	136
	¹ Gurpinar, SS., ² Kart, D., ¹ Eryilmaz, M.	
OP187:	IN VIVO EFFECT OF <i>Origanum majorana</i> L. ESSENTIAL OIL ON <i>Galleria mellonella</i> LARVAE	137
	¹ Ozturk, S., ² Erdem, SA.	
OP188:	ROLE OF EXOSOMAL miRNAs IN IMATINIB RESISTANCE OF CHRONIC MYELOID LEUKEMIA.....	137
	¹ Karabay, AZ., ² Özkan, T., ¹ Koç, A., ³ Karadağ, A., ² Hekmatshoar, Y., ² Sunguroglu, A., ¹ Aktan, F., ¹ Buyukbingol, Z.	
OP189:	EVALUATION OF THE EFFECTS OF SIRT5 MODULATORS ON THE APOPTOSIS OF K562 CELLS AND SIRT5 AND CYTOCHROME C PROTEINS	138
	¹ Koc, A., ² Ozkan, T.	
OP190:	INVESTIGATION OF CYTOTOXIC AND APOPTOTIC EFFICACY OF ORCINOL IN SW480 HUMAN COLORECTAL CANCER CELLS	138
	¹ Yanik, B., ² Bakar-Ates, F.	
OP191:	INVESTIGATION OF <i>IN VITRO</i> PHOTODYNAMIC THERAPY EFFECTS OF WATER SOLUBLE Zn (II) PHTHALOCYANINE ON HCT-116 CELLS.....	139
	¹ Barut, B., ² Yalçın, CÖ.	
OP192:	ANGIOTENSIN II INDUCES NLRP1 INFLAMMASOME ACTIVATION IN HCN-2 CELL LINE	139
	¹ Birim, D., ² Armanag, G.	
OP193:	DEVELOPMENT OF LABEL-FREE ELECTROCHEMICAL IMMUNOSENSOR FOR LEPTIN DETERMINATION	140
	¹ Kaman G., ² Koyuncu Zeybek, D.	
OP194:	PRENATAL STRESS MAY INCREASE THE RISK OF DEVELOPING ALZHEIMER-LIKE NEUROPATHOLOGY IN THE HIPPOCAMPUS OF RATS	141
	¹ Turunc Ozoglu, E.	
OP195:	DETERMINATION OF THE FIBRINOGENOLYTIC ACTIVITY OF <i>MONTIVIPERA RADDEI</i> (RADDE'S MOUNTAIN VIPER) VENOM BY POLYACRYLAMIDE GEL ELECTROPHORESIS.....	141
	¹ Atasoy, F., ² İgci, N.	
OP196:	PROPHYLACTIC EFFECT OF MYRICETIN AND APIGENIN AGAINST LIPOPOLYSACCHARIDE-INDUCED ACUTE LIVER INJURY.....	142
	¹ Berkoz, M., ¹ Unal, S., ² Karayakar, F., ³ Yunusoğlu, O., ² Ozkan-Yılmaz, F., ² Ozluer-Hunt, A., ^{1,4} Aslan, A.	
OP197:	TOLUIDINE BLUE O DECREASES TAU PHOSPHORYLATION AT THR181 AND SER202/THR 205 IN N2A MOUSE NEUROBLASTOMA CELLS STABLY EXPRESSING THE HUMAN SWEDISH MUTANT APP695	142
	¹ Onder, S., ¹ Biberoglu, K., ¹ Yuksel, M., ¹ Tacal, O.	
OP198:	INVESTIGATION OF <i>IN VITRO</i> ANTIOXIDANT, CYTOTOXIC AND MUTAGENIC ACTIVITIES OF ESSENTIAL OIL DERIVED FROM <i>Lavandula angustifolia</i> CULTIVATED in TURKEY	143

¹Biltekin, SN., ²Omurtag-Özgen, PS., ³İduğ, T., ⁴Macit, Ç., ⁵Ayran, İ., ⁵Çelik, SA. ⁵Kan, Y.,
⁶Omurtag GZ.

- OP199: BRAIN-DERIVED NEUROTROPHIC FACTOR LEVELS IN BREAST CANCER 144
¹Taskan, T., ²Kurukahvecioğlu, O., ³Karaman, N., ²Noori, F., ¹Gonenc, A.
- OP200: THE EFFECT OF NEOPTERIN ON EPITHELIAL MESENCHYMAL TRANSITION DRIVING GENE EXPRESSIONS AT HCC 144
²Subashi, Y., ¹Najjar, M., ¹Kunter, I.
- OP201: HEALTHCARE SERVICES AND EMPATHY: EXAMPLE OF PHARMACY STUDENTS 145
¹Yaman, U., ²Sözen-Şahne, B.
- OP202: A STUDY ON PATIENT EXPERIENCE IN COMMUNITY PHARMACIES: ISTANBUL PROVINCE SAMPLE 145
¹Akalgan, D., ²Ozcelikay, G..
- OP203: THE STATE OF QUALITATIVE RESEARCH IN PHARMACY LITERATURE: A FOCUSED MAPPING REVIEW AND SYNTHESIS 146
Gülpinar, G.
- OP204: IS THERE ANY DIFFERENCE SINCE 2012: WEB SITES OF PHARMACY SCHOOLS 147
Yumrukaya, L., Sözen-Şahne, B., Yeğenoğlu, Y.
- OP205: CINCHONA BARK AND ITS ALKALOIDS IN THE 4TH PORTUGUESE OFFICIAL PHARMACOPOEIA 147
^{1,2}Semedo, M., ^{1,2}Pita, J.
- OP206: DETERMINATION OF PRESCRIBED MEDICINE BORROWING BEHAVIOR OF INDIVIDUALS 148
Başak H., Arslan, M.
- OP207: COVID-19 ANXIETY OF THE STUDENTS AND ACADEMICIANS OF PHARMACY SCHOOLS IN TURKEY AND ITS EFFECTS ON THEIR PSYCHOLOGICAL WELL-BEING 148
Çalikuşu, M., Özçelikay, G.
- P001: HPLC METHOD DEVELOPMENT AND VALIDATION OF CHLORHEXIDINE GLUCONATE AND BENZYDAMINE HCL FOR BUCCAL DELIVERY 151
Arpa, MD., Yağcılar, AP.
- P002: EVALUATION OF BERBERINE PHYTOSOME STABILITY IN SIMULATED BODY FLUIDS BY HPLC METHOD 151
¹Gungor Ak, A., ²Karatas, A.
- P003: THE EVALUATION OF THE POTENTIAL EUDRAGIT-BASED DELAYED RELEASE NANOFIBERS FOR COLON TARGETING 152
Yıldırım, E., Yıldız, A., Saar, S., Tuğcu-Demiröz, F., Acartürk, F.
- P004: EFFECTS OF PRODUCTION METHOD VARIATIONS ON PARTICLE SIZE DISTRIBUTION OF ETHYL CELLULOSE NANOPARTICLES 152
Tas, B., Akdag, Y., Aytekin, E., Bozdag Pehlivan, S., Oner, L.
- P005: A NOVEL RP-HPLC METHOD TO DETERMINE IRBESARTAN AND HYDROCHLOROTHIAZIDE IN FIXED DOSE COMBINATIONS: METHOD DEVELOPMENT AND VALIDATION 153
^{1,2}Kaval, B., ^{3,4}Özcan, S., ^{2,5}Kaynak, MS.
- P006: PREPARATION AND EVALUATION OF LYSOZYME LOADED POLYCAPROLACTONE MICROPARTICLES USING THE FULL FACTORIAL DESIGN 153

P007:	EVALUATION OF IN VITRO-IN VIVO RELATIONSHIP: BOSENTAN-LOADED LIPID BASED FORMULATION VERSUS COMMERCIAL PRODUCT	154
	^{1,2} Timur, B., ¹ Yilmaz Usta, D., ¹ Teksin, ZS.	
P008:	DoE BASED APPROACH FOR THE DESIGN OF PIROXICAM LOADED POLYMERIC NANOPARTICLES.....	155
	¹ Bayram, B., ² Sengel-Turk, CT.	
P009:	ATORVASTATIN-ENCAPSULATED CORE-SHELL TYPE HYBRID NANOCARRIERS FOR LOCAL THERAPY OF BREAST CANCER: FORMULATION AND OPTIMIZATION STUDIES	155
	¹ Sengel-Turk, CT., ² Bakar-Ates, F.	
P010:	DEVELOPMENT AND OPTIMIZATION OF AN ANTIHYPERTENSIVE FIXED-DOSE COMBINATION USING PLACKETT-BURMAN DESIGN	156
	¹ Sarisaltik-Yasin, D., ² Teksin, ZS.	
P011:	DEVELOPMENT AND OPTIMIZATION OF SELF-NANOEMULSIFYING DRUG DELIVERY SYSTEM OF BOSENTAN USING BOX BEHNKEN DESIGN.....	156
	¹ Yilmaz Usta, D., Teksin, ZS.	
P012:	COMPARISON OF BIORELEVANT DISSOLUTION OF MEDIUM CHAIN MONO AND DIGLYCERIDES BASED BOSENTAN-LOADED SELF-NANOEMULSIFYING FORMULATIONS.....	157
	¹ Yilmaz Usta, D., Teksin, ZS.	
P013:	POLYMERIC MICRONEEDLES FOR NASAL DRUG DELIVERY	158
	^{1,2} Aykaç, K., ¹ Başaran, E.	
P014:	PREPARATION AND OPTIMIZATION OF B-CYCLODEXTRIN INCLUSION COMPLEXES OF ATOMOXETINE HYDROCHLORIDE	159
	^{1,2} Ozyilmaz, ED., ² Comoglu, T.	
P015:	PREPARATION OF LAMOTRIGINE SOLID DISPERSIONS WITH DIFFERENT POLYMERIC AND SURFACTANT CARRIERS TO ENHANCE SOLUBILITY	159
	¹ Pezik, E., ^{1,2} Gultekin, Y., ¹ Gulsun, T., ¹ Sahin, S., ¹ Vural, I.	
P016:	DEVELOPMENT AND VALIDATION OF AN HPLC METHOD FOR DETERMINATION OF LAMOTRIGINE.....	160
	¹ Pezik, E., ^{1,2} Gultekin, Y., ¹ Gulsun, T., ¹ Sahin, S., ¹ Vural, I.	
P017:	POSACONAZOLE LOADED EUDRAGIT® FS 100 NANOPARTICLES	161
	^{1,2} Aykaç, K., ¹ Başaran, E., ¹ Yenilmez, E., ¹ Demirel, M.	
P018:	CAFFEINE LOADED CHITOSAN GEL: FORMULATION AND IN-VITRO EVALUATION	161
	¹ Karapınar, B., ² Yenilmez, E.	
P019:	ANALYTICAL METHOD DEVELOPMENT AND VALIDATION FOR SIMULTANEOUS QUANTIFICATIONS OF BRUSATOL IN DRUG LOADED LIPOSOMES	162
	¹ Bilgili, G., ² Sezgin Bayındır, Z.	
P020:	INCREASING RIBOFLAVIN SOLUBILITY WITH SULFOBUTYL ETHER-B-CYCLODEXTRIN AND FORMATION OF DRUG- CYCLODEXTRIN INCLUSION COMPLEXES.....	163
	¹ Polat, HK., ² Aytekin, E., ² Kurt, N., ² Bozdag Pehlivan, S., ² Çalış, S.	
P021:	THERMODYNAMIC STABILITY TESTING OF KETOCONAZOLE AND CAFFEINE LOADED NANOEMULSION FORMULATIONS FOR DERMAL APPLICATION	163

P022:	DEVELOPMENT&VALIDATION OF HPLC METHOD FOR TOPICAL DELIVERY OF FINASTERIDE USED IN HAIR LOSS TREATMENT.....	164
	¹ Arpa, MD., ² Seçen, İM.	
P023:	LACOSAMIDE LOADED MICRONEEDLES AS NASAL DRUG DELIVERY SYSTEMS	164
	^{1,2} Aykaç, K., ¹ Başaran, E.	
P024:	DEVELOPMENT OF OROMUCOSAL FORMULATIONS - EVALUATION OF THE STRUCTURAL AND MECHANICAL PROPERTIES.....	165
	Centkowska, K., Płaczek, M., Sznitowska, M., Stawecka, M.	
P025:	VALIDATION OF AN HPLC METHOD FOR THE DETERMINATION OF CARFILZOMIB AND NILE RED FROM PLGA NANOPARTICLES	166
	¹ Kaya, MZ., ² Ozturk, M., ¹ Bozdag Pehlivan, S.	
P026:	A NOVEL UV/VIS SPECTROSCOPY METHOD FOR THE DETERMINATION OF ATEZOLIZUMAB: METHOD DEVELOPMENT AND VALIDATION.....	166
	¹ Ekinci, M., ² Akbaba, H., ³ Santos-Oliveira, R., ¹ İlem-Özdemir, D.	
P027:	DEVELOPMENT AND OPTIMIZATION OF R-HPLC METHOD OF OXICONAZOLE NITRATE FOR TOPICAL DRUG DELIVERY	167
	Arpa, MD., Ünükür, MZ.	
P028:	FORMULATION AND CHARACTERIZATION OF TEDIZOLID PHOSPHATE LOADED LIPOSOMAL GEL FORMULATIONS FOR TOPICAL TREATMENT ABSSSIS.....	168
	¹ Kuru, HH. ² Karpuz, M. ¹ Şenyiğit, Z.	
P029:	PREPARATION AND CHARACTERIZATION OF CREAM-GELS BASED ON HYDROXYETHYL ACRYLATE / SODIUM ACRYLOYLDIMETHYL TAURATE COPOLYMER INCORPORATED WITH FIXED OR ESSENTIAL OILS	168
	¹ İlhan, M., Gultekin, HE., Senyigit, Z.	
P030:	DEVELOPMENT OF BIODEGRADABLE NANOPARTICLES FOR THE BRAIN DELIVERY OF FLURBIPROPHENE	169
	Kurt, N., Çopur, T., Bozdag Pehlivan, S., Öner, L.,	
P031:	PREPARATION AND <i>IN VITRO</i> CHARACTERIZATION OF LIPID-COATED NANOPARTICLES CONTAINING CARBOPLATIN AND DECITABINE.....	170
	¹ Eşim, O., ² Hascicek, C.	
P032:	DEVELOPMENT AND CHARACTERIZATION OF BUCCAL FILM CONTAINING HYDROCORTISONE NANOSUSPENSIONS	170
	^{1,2} Çulcu, Ö., ¹ Saar, S., ¹ Tuğcu-Demiröz, F., ¹ Tırmaksız, F.	
P033:	MODELING AND COMPARISON OF <i>IN VITRO</i> DISSOLUTION PROFILES OF NAPROXEN SODIUM TABLETS IN BIORELEVANT MEDIA	171
	¹ Olgac, S., ¹ Yılmaz Usta, D., ² Demirdağ, B., ² Erman, NA., ¹ Teksin, ZS.	
P034:	VALIDATED HPLC METHOD FOR THE DETERMINATION OF TENOFOVIR AND ITS APPLICATION FOR <i>IN-VITRO</i> AND <i>EX-VIVO</i> INVESTIGATIONS OF TENOFOVIR LOADED DOUBLE NANOEMULSION	172
	¹ Olgac, S., ¹ Yılmaz Usta, D., ² Erman, NA., ¹ Inceçayır, T., ¹ Teksin, ZS.	
P035:	EVALUATION OF POLYVINYL ALCOHOL NANOFIBERS AS VAGINAL DRUG DELIVERY SYSTEM.....	172
	Saar, S., Tuğcu-Demiröz, F.	

P036:	PREPARATION AND EVALUATION OF ALPHA TOCOPHEROL/ CYCLODEXTRIN COMPLEXES.....	173
	<i>^{1,2}Adatepe, S., ¹Demirel, M.</i>	
P037:	IN SILICO PREDICTION OF INTESTINAL DISSOLUTION AND ABSORPTION OF CARBAMAZEPINE IN HUMANS	174
	<i>Incecayir, T., Benli, S.</i>	
P038:	EVALUATION OF A BIPHASIC IN VITRO DISSOLUTION TEST FOR LAMOTRIGINE IMMEDIATE RELEASE TABLETS AND CORRELATION TO HUMAN IN VIVO PERFORMANCE.....	174
	<i>Incecayir, T., Demir, ME.</i>	
P039:	DESIGN OF KETOROLAC TROMETHAMINE LOADED NANOPARTICLES AND EVALUATION OF IN VITRO EFFICIENCY FOR BRAIN TUMOR TREATMENT.....	175
	<i>¹Copur, T., ²Yalcin, D., ¹Kurt, N., ¹Pezik, E., ¹Bozdag Pehlivan, S., ¹Oner, L.</i>	
P040:	THE EFFECT OF FREEZE DRYING WITH DIFFERENT CRYOPROTECTANTS ON THE CHARACTERISTICS OF KETOROLAC TROMETHAMINE LOADED NANOPARTICLES.....	176
	<i>¹Copur, T., ²Yalcin, D., ¹Kurt, N., ¹Pezik, E., ¹Bozdag Pehlivan, S., ¹Oner, L.</i>	
P041:	SEMISOLID NLC FORMULATIONS FOR COSMETIC USE: EVALUATION OF MECHANICAL PROPERTIES	176
	<i>¹Cakir, K., ²Inal, Ö., ²Badilli, U.</i>	
P042:	BUDESONIDE LOADED CONTROLLED-RELEASE POLYCAPROLACTONE NANOPARTICLES.....	177
	<i>Turanli, Y., Acartürk, F.</i>	
P043:	EFFECT OF POLYMER ON ONDANSETRON HCl LOADED POLYMERIC NANOPARTICLES.....	177
	<i>^{1,2}Ozdal, ZD., ¹Takka, S.</i>	
P044:	BIOPHARMACEUTICS CLASSIFICATION SYSTEM (BCS) BASED BIOWAIVER APPROACH IN TURKEY.....	178
	<i>Koksal, T., Teksin, ZS.</i>	
P045:	EVALUATIONS OF FASTED AND FED BIOAVAILABILITIES OF SELF DOUBLE EMULSIFYING DRUG DELIVERY SYSTEM OF TENOFOVIR.....	179
	<i>Bektaş, D., Tuğcu-Demiröz, F., Teksin, ZS.</i>	
P046:	CHARACTERISATION AND FORMULATION OF MELATONIN INTRANASAL DELIVERY SYSYSTEM	179
	<i>Görür, FŞ., Uzuner, YY.</i>	
P047:	DETERMINATION OF EFFECTIVE SURFACE MODIFICATIONS OF SILICA NANOPARTICLES AS VEGF-TARGETED SIRNA CARRIERS.....	180
	<i>^{1,2}Ultav, G., ¹Tonbul, H., ²Salva, E.</i>	
P048:	INVESTIGATION OF THE EFFECTIVENESS OF GLYCOPOLYMER BASED THERANOSTIC NANOSISTEMS IN BREAST CANCER.....	181
	<i>¹Yiğit-Erdem, G., ²Omurtag-Özgen, PS., ³Dağ, A.</i>	
P049:	SYNTHESIS OF CISPLATIN AND/OR GEMCITABINE CONTAINING POLYMERIC NANODRUG FORMULATIONS FOR BREAST CANCER TREATMENT	181
	<i>¹Gençoglu, T., ²Cetin, B., ¹Yiğit-Erdem, G., ³Omurtag-Özgen, PS., ⁴Dağ, A.</i>	

P050:	TRANSCRIPTOMIC CHARACTERIZATION OF THE USNIC ACID (UA) EFFECTS ON TRIPLE NEGATIVE BREAST CANCER (TNBC) WITH NEXT GENERATION SEQUENCING TECHNOLOGY.....	182
	¹ Tanman, Ü., ² Türktaş Erken, M. ¹ Cansaran Duman, D.	
P051:	DETERMINATION THE USNIC ACID (UA) THERAPY EFFECTS ON TRIPLE NEGATIVE BREAST CANCER (TNBC) BY PROTEOMIC APPROACHES.....	182
	¹ Tanman, Ü., ¹ Cansaran Duman, D. ² Türktaş Erken, M.	
P052:	SOME NEW 3,5-DISUBSTITUTED 1,3,4-OXADIAZOLE DERIVATIVES WITH IN VITRO ANTI-INFLAMMATORY ACTIVITY	183
	¹ Dedeoğlu-Erdoğan, A., ^{1,2} Dagliyan, İ., ³ Sipahi, H., ¹ Köksal, M.	
P053:	A MONTMORILLONITE CLAY AS AN EFFICIENT AND GREEN CATALYST FOR FUNCTIONAL POLYETHER SYNTHESIS	184
	<i>Belbekiri, H., Meghabar, R., Belbachir, M.</i>	
P054:	SYNTHESIS AND STANDARDIZATION OF AN IMPURITY OF ACETAMINOPHEN, DEVELOPMENT AND VALIDATION OF RELATED ULTRA-HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD	184
	¹ Arikan, CC., ² Küçükgüzel, İ.	
P055:	DEEP EUTECTIC SOLVENTS AS POWERFUL CATALYSTS AND SOLVENTS FOR THE SYNTHESIS OF AMIDES.....	185
	¹ Procopio, D., ² Nardi, M., ² Oliverio, M., ² Procopio, A., ¹ Di Gioia, ML.	
P056:	AFFINITY OF THE POLYETHER IONOPHORE MONENSIN A TO BIND MONOVALENT METAL IONS: A DFT/PCM STUDY.....	185
	<i>Pantcheva, I., Dudev, T., Cheshmedzhieva, D., Stamboliyska, R.</i>	
P057:	FOCUSING ON C-4 POSITION OF 1,4-DIHYDROPYRIDINE RING: SYNTHESIS AND L-/T-TYPE CALCIUM CHANNEL BLOCKING ACTIVITY	186
	¹ Akman, D., ² Huang, S., ² Zamponi, GW., ¹ Gündüz, MG.	
P058:	THE EFFECT OF COX-2 INHIBITORS ON ACETYLCHOLINE ESTERASE IN TREATMENT OF ALZHEIMER'S DISEASE	186
	¹ Kahvecioglu, D., ² Yilmaz, S., ² Yenice-Cakmak, G., ¹ Kocyigit-Kaymakcioglu, B.	
P059:	NEW INSIGHTS INTO COVALENT ENZYMATIC INHIBITION MEDIANATED BY ELECTROPHILIC SELENIUM COMPOUNDS: THE CASE OF THE SARS-CoV-2 MAIN PROTEASE.....	187
	<i>Scimmi, C., Liviabella, D., Mangiacavalli, F., Sancinetto, L., Santi, C.</i>	
P060:	SYNTHESIS OF BENZIMIDAZOLE, BENZOTHIAZOLE, BENZOFURANE AND NAPHTOFURANE DERIVATIVES OF AMINOTHIAZOLES	187
	¹ Akgun, E., ¹ Tok, Bl., ² Caskurlu, A., ¹ Sahin, Z., ³ Yurttaş, L., ¹ Berk, B., ¹ Demirayak, S.	
P061:	PREPARATION OF SOME PURINE DERIVATIVES: USE OF THE 2D NMR ¹ H, ¹⁵ N & ¹ H, ¹³ C HMBC TECHNIQUES AND X-RAY CRYSTALLOGRAPHY IN ASSIGNING REGIOCHEMISTRY	188
	¹ Doganc, F., ² Sahin, E., ¹ Goker, H.	
P062:	SYNTHESIS AND MOLECULAR MODELING STUDIES OF SOME NOVEL BENZOTIAZOLE DERIVATIVES AS ANTI-CANCER AGENTS.....	189
	¹ Yenice-Cakmak, G., ¹ Yilmaz, S., ² Yildiz, I.	
P063:	SYNTHESIS AND ANTICANCER ACTIVITY OF ETODOLAC HYDRAZONES	189
	¹ Koc, HC., ² Atlihan, I., ³ Mega-Tiber, P., ³ Orun, O., ⁴ Kucukguzel, SG.	
P064:	CRYSTAL STRUCTURE OF LITHIUM(I) COMPLEX OF THE ANTIBIOTIC LASALOCID	190

P065:	SYNTHESIS OF SOME NOVEL 4-(1H-BENZIMIDAZOL-1-YL)-N'-BENZYLIDENE BENZOHYDRAZIDE DERIVATIVES	190
	<i>Alp, M., Alp, AS.</i>	
P066:	INDOLE-BENZIMIDAZOLE DERIVATIVES AS ANTIBACTERIAL AGENTS AGAINST HOSPITAL INFECTIONS AND THEIR DOCKING PROFILES.....	191
	<i>¹Zengin-Karadayi, F., ¹Kisla, MM., ²Kaskatepe, B., ¹Ates-Alagoz, Z.</i>	
P067:	SYNTHESIS AND <i>IN VITRO</i> EVALUATION OF THE ANTIOXIDANT ACTIVITY OF IMINES.....	192
	<i>¹Memmou, F., ²Benmehdi, H., ¹Tounsi, A., ²Fellah, Kh.</i>	
P068:	SYNTHESIS OF PLATINUM(II) COMPLEXES WITH 2-SUBSTITUTED BENZIMIDAZOLE LIGANDS	192
	<i>¹Özçelik, AB., ¹Akdağ, M., ²Utku, S.</i>	
P069:	1,4-DIHYDROPYRIDINE-AZOLE HYBRIDS: SYNTHESIS, COMPUTATIONAL STUDIES AND ANTIMICROBIAL ACTIVITY.....	193
	<i>¹Gunduz, MG., ²Dengiz, Ç., ¹Kocak-Aslan, E., ³Skaro-Bogojevicogojevic, S., ³Nikodinovic-Runic, J.</i>	
P070:	SYNTHESIS OF DIHYDROPRIMIDINE DERIVATIVES WITH L/T-TYPE CALCIUM CHANNEL BLOCKING ACTIVITIES.....	193
	<i>¹Gündüz, MG., ²Dengiz, Ç., ³Huang, S., ³Zamponi, GW.</i>	
P071:	DESIGN, SYNTHESIS AND ANTIMICROBIAL EVALUATION OF NOVEL ISOQUINOLIN-UREA HYBRIDE MOLECULES	194
	<i>¹Han, Mİ., ²Dengiz, Ç., ³Doğan, ŞD., ⁴Gündüz, MG., ⁵Özkul, C.</i>	
P072:	STUDIES ON ANTIMICROBIAL PROPERTIES OF SOME BENZOXAZOLES	194
	<i>¹Faydali, N., ²Temiz Arpacı, O., ³Kuyucuklu, G., ⁴Salan, AS.</i>	
P073:	SYNTHESIS AND STRUCTURE ELUCIDATION OF SOME BENZOXAZOLE DERIVATIVES	195
	<i>¹Faydali, N., ²Temiz Arpacı, O.</i>	
P074:	MONONUCLEAR COPPER(II) COMPLEX OF MACROLIDE ANTIBIOTIC TILMICOSIN.....	196
	<i>¹Stamboliyska, R., ¹Petkov, N., ¹Pantcheva, I., ¹Stoykova, S., ²Stoyanova, R., ²Kukeva, R., ³Simova, S.</i>	
P075:	DINUCLEAR COPPER(II) COMPLEXES OF MACROLIDE ANTIBIOTIC TILMICOSIN.....	196
	<i>¹Stamboliyska, R., ¹Petkov, N., ¹Pantcheva, I., ¹Stoykova, S., ¹Tadjer, A., ²Stoyanova, R., ²Kukeva, R., ³Simova, S.</i>	
P076:	SYNTHESIS OF SOME NOVEL SCHIFF BASES INCORPORATED WITH INDAZOLE MOIETY	197
	<i>Kayikci-Pasa, N., Gurkan-Alp, AS.</i>	
P077:	SYNTHESIS OF SOME NOVEL N'-(ARYLMETHYLENE)-1H-INDOLE-5-CARBOHYDRAZIDES	197
	<i>Gurkan-Alp, AS., Avuka, OF.</i>	
P078:	IN SILICO DESIGN AND SYNTHESIS OF NOVEL 2-ACYLHYDRAZONO-5-ARYLMETHYLENE-4-THIAZOLIDINONES AS enoyl-acyl carrier protein reductase INHIBITORS.....	198
	<i>¹Dingiş Birgül, Sİ. ¹Küçükgüzel, İ. ¹Akdemir, A.</i>	

P079:	SYNTHESIS OF SOME NEW 2-PHENOXYACETAMIDE AND 3-PHENOXYPROPANAMIDE DERIVATIVES AND EVALUATION OF THEIR CHOLINESTERASE INHIBITOR ACTIVITIES.....	198
	¹ Shakila, S., ¹ Kılıç, B., ² Aksakal, F., ¹ Doğruer, DS.	
P080:	PREPARATION OF MICROPARTICLES FROM LAVENDER EXTRACT WITH HYDRO/SOLVOTHERMAL SYNTHESIS: CYTOTOXIC AND GENOTOXIC EFFECT ON CANCER CELL LINES	199
	¹ Butun Sengel, S., ² Sengel, T., ³ Butun, V.	
P081:	MOLECULAR DOCKING AND SYNTHESIS OF NOVEL BIPHENYL-CHROMONE DERIVATIVES AS AMPK ACTIVATORS	200
	² Güney, S., ¹ Ceylan-Ünlüsoy, M.	
P082:	DETERMINATION OF NOVEL UREA AND SULFONAMIDE DERIVATIVES OF ISATIN SCHIFF BASES AS POTENTIAL RECEPTOR TYROSINE KINASE INHIBITOR BY MOLECULAR DOCKING STUDIES	200
	¹ Demirel, UU., ² Ölgen, S.	
P083:	INVESTIGATION OF POSSIBLE PROTECTIVE EFFECTS OF MOMORDICA CHARANTIA (BITTER MELON) IN LUNG DAMAGE CAUSED BY METHOTREXATE.....	201
	¹ Ediz, Ç., ¹ Ede, S., ² Özbeyli, D., ¹ Albayrak, Ö., ³ Çevik, Ö., ⁴ Şener, G.	
P084:	INTEGRATING NANOPARTICLE COATED MICROPARTICLES IN THE FIELD OF ELECTROPHYSIOLOGY.....	201
	Alyu, F.	
P085:	OPTOGENETICS COMBINED WITH THE PATCH CLAMP TECHNIQUE	202
	Alyu, F.	
P086:	ANTIHYPERALGESIC EFFECTS OF LEVETIRACETAM INJECTED INTRA-VPL ON CHRONIC CONSTRICKTION INJURY MODEL.....	202
	Alyu, F., Ozturk, Y.	
P087:	PROBIOTICS AND EXPERIMENTAL HYPERLIPIDEMIA	203
	¹ Radeva–Ilieva, M., ¹ Hvarchanova, N., ¹ Georgieva, M., ¹ Stoeva, S., ² Stefanova, N., ¹ Georgiev, K.	
P088:	ANTI-ULCER POTENTIAL OF CATECHIN FRACTION OBTAINED FROM INONOTUS NIDUS-PICI IN RATS.....	203
	¹ Radeva–Ilieva, M., ¹ Georgieva, M., ² Zhelev, I., ¹ Georgiev, K.	
P089:	THE IMPACT OF ANTIMICROBIAL USE ON POTENTIAL MAJOR DRUG-DRUG INTERACTIONS IN THE PEDIATRIC INTENSIVE CARE UNIT PATIENTS	204
	¹ Albayrak, A., ² Akkuzu, E., ³ Karahalil, B.	
P090:	THE CLINICAL OUTCOMES OF KIDNEY TRANSPLANT PATIENTS USED EITHER AZATHIOPRINE OR MYCOPHENOLATE.....	204
	¹ Selcuk, A., ¹ Pehlivanli, A., ² Eyüpoglu, S., ³ Ozcelikay, AT., ² Sengul, S.	
P091:	PATIENT ENGAGEMENT IN THE MANAGEMENT OF MULTIPLE SCLEROSIS.....	205
	¹ Goncuoglu, C., ² Bayraktar-Ekincioglu, A., ³ Acar-Ozen, P., ⁴ Tuncer, A.	
P092:	PERCEPTION OF COVID-19 VACCINATION AMONGST PHYSICIANS: AN ONLINE SURVEY	206
	¹ Dogan, CZ., ¹ Tecen-Yucel, K., ¹ Kara, E., ² Kutsal-Kaynar, E., ¹ Demirkan, K., ³ Unal, S.	

P093:	SUPPORTIVE THERAPY-INDUCED POLYPHARMACY AND DRUG-RELATED PROBLEMS IN CANCER PATIENTS.....	206
	<i>Bayraktar, I., Aras Atik, E., Bayraktar-Ekincioglu, A.</i>	
P094:	IMPROVEMENT OF IMMUNOSUPPRESSIVE MEDICATION ADHRENCE IN NEW RENAL TRANSPLANT PATIENTS.....	207
	<i>¹Tecen-Yucel, K., ¹Aras, E., ¹Ozdemir, N., ¹Bayraktar-Ekincioglu, A., ²Yildirim, T., ¹Demirkan, K., ²Erdem, Y.</i>	
P095:	NEPHROLOGISTS' OPINION ON THE MANAGEMENT OF ASYMPTOMATIC HYPERURICAEMIA IN PATIENTS WITH CHRONIC KIDNEY DISEASE.....	208
	<i>¹Kurtaran, M., ¹Tecen-Yucel, K., ¹Bayraktar-Ekincioglu, A., ²Erdem, Y.</i>	
P096:	EVALUATION OF DRUG BURDEN IN GERIATRIC PATIENTS: A POINT PREVALENCE STUDY.....	208
	<i>¹Kurtaran, M., ¹Gokcay, H., ¹Demirkan, K., ²Halil, M.</i>	
P097:	COLISTIN INDUCED NEPHROTOXICITY: EXPERIENCE FROM A UNIVERSITY HOSPITAL	209
	<i>¹Bakir Ekinci, P., ¹Kurtaran, M., ¹Kara, E., ²Avoi, H., ¹Demirkan, K., ³Metan, G.</i>	
P098:	PSYCHOMETRIC PROPERTIES OF TURKISH VERSION OF IDENTIFICATION OF MEDICATION ADHERENCE BARRIERS QUESTIONNAIRE IN PATIENTS WITH CHRONIC DISEASES: PRELIMINARY FINDINGS.....	210
	<i>¹Yağmur, M., ²Sancar, M., ³Ay, P., ⁴Okuyan, B.</i>	
P099:	EVALUATION OF COMMUNITY PHARMACISTS' PRACTICES REGARDING SUPPLYING AND STORAGE OF THE VACCINES.....	210
	<i>Ozdemir, N., Kara, E., Tecen-Yucel, K., Aras Atik, E., Celiker, A., Bayraktar-Ekincioglu, A., Demirkan, K.</i>	
P100:	EVALUATION OF RENAL DRUG DOSING IN HOSPITALIZED PATIENTS WITH RENAL IMPAIRMENT.....	211
	<i>¹Memis, H., ¹Cakir, A., ¹Guzel, S., ²Ozdemir, N., ¹Gun, ZU.</i>	
P101:	INFLUENZA VACCINATION COVERAGE AMONG PHYSICIANS AND NURSES IN ONCOLOGY SETTINGS	211
	<i>¹Ozdemir, N., ¹Aras Atik, E., ¹Tecen-Yucel, K., ¹Bayraktar-Ekincioglu, A., ²Kilickap, S.</i>	
P102:	EFFECTS OF ACRYLAMIDE, HYDROXYMETHYL FURFURAL AND CAFFEIC ACID ON DNA DAMAGE IN V79 CELLS.....	212
	<i>Babacanoğlu, C., Çal, T., Aydin Dilsiz, S., Ündeğer Bucurgat, Ü.</i>	
P103:	INVESTIGATION OF THE CYTOTOXICITY OF BISPHENOL A AND ITS ANALOGS (BPS, BPF, BPAF, BPZ) IN MCF-7 AND HSeC CELL LINES	212
	<i>¹Erdogmus, E., ²Ipek, S., ³Iyigundogdu, I., ²Ustundag, A., ²Duydu, Y.</i>	
P104:	PROTECTIVE ROLE OF SELENOCOMPOUNDS AGAINST DNA DAMAGE AND OXIDATIVE STRESS CAUSED BY BISPHENOL A IN HUMAN PAPILLARY THYROID CANCER CELL LINE	213
	<i>¹Tan, E., ²Ozkemahli, G., ³Bacanlı, M., ⁴Balci, A., ⁵Baysal, E., ⁶Zeybek, ND., ⁴Erkekoglu, P., ⁴Başaran, N., ⁶Koçer-Gümüşel, B.</i>	
P105:	POTENTIAL HAZARD IN THE METFORMIN PRODUCTS, NITROSAMINES	214
	<i>¹Tan, E., ²Baysal, E., ³Coşkun, M., ³Yetkin, İ.</i>	
P106:	DETERMINATION OF CYP1B1'3 (LEU432VAL) POLYMORPHISM IN A TURKISH POPULATION	214
	<i>¹Kargin Solmaz, FÖ., ²Ada, AO.</i>	

P107:	SOME GENE POLYMORPHISMS AFFECTING DIABETES MELLITUS TYPE 2 DEVELOPMENT	215
	<i>Ates, I., Gumus Kus, CA.</i>	
P108:	POSSIBLE RELATIONSHIPS BETWEEN SOME GENE POLYMORPHISMS AND DIABETES MELLITUS TYPE 2 IN A TURKISH POPULATION	215
	<i>Ates, I., Arazi Erdem, S.</i>	
P109:	IDENTIFYING POTENTIAL GENETIC BIOMARKERS OF THE CARDIOTOXICITY INDUCED BY ANTHRACYCLINES.....	216
	<i>Demirbugen Oz, M.</i>	
P110:	COMPUTATIONAL MODEL FOR INVESTIGATING THE TOXICITY OF CHEMICALS USED MAINLY IN COSMETIC PRODUCTS	216
	<i>¹Ulker, OC., ²Banerjee, P.</i>	
P111:	CYTOTOXIC AND GENOTOXIC EFFECTS OF ALUMINUM COMPOUNDS IN ANTIPERSPIRANTS <i>IN VITRO</i>	217
	<i>¹Ipek, S., ²Cebi, G., ¹Ustundag, A., ¹Duydu, Y.</i>	
P112:	ASSESSMENT OF CYTOTOXICITY AND ANTIOXIDANT PROPERTY OF METHANOL AND AQUEOUS EXTRACTS OF <i>SMILAX EXCELSA</i>	217
	<i>¹Yilmaz Sarialtin, S., ²Cicek Polat, D., ³Yalgin, C.O.</i>	
P113:	SIMULTANEOUS DETERMINATION OF SOME ANTIFUNGAL PESTICIDES FROM HUMAN BIOLOGICAL SAMPLES BY HPLC.....	218
	<i>Barut, BB., Erkmen, C., Uslu, B.</i>	
P114:	A GQDS@PEDOT NPS-BASED ELECTROCHEMICAL TYROSINASE ENZYME BIOSENSOR FOR ADRENALINE DETECTION	219
	<i>Erkmen, C., Demir, Y., Kurbanoglu, S., Uslu, B.</i>	
P115:	VOLTAMMETRIC STUDIES ON THE ANTIBIOTIC DRUG CEFPROZIL USING A GLASSY CARBON ELECTRODE	220
	<i>Ozturk, G., Kul, D., Kiraz, B., Yartaşı, B., Agin, F.</i>	
P116:	EFFECTIVENESS OF <i>ACHILLEA GONOCEPHALA</i> LOADED NANOPARTICLE ENCAPSULATION ON ANTIOXIDANT AND CYTOTOXIC PROPERTIES	220
	<i>¹Taskin, D., ²Doğan, M., ³Ermanoglu, M., ⁴Arabaci, T.</i>	
P117:	SIMULTANEOUS QUANTITATION OF SULFUR METABOLITES IN CELL EXTRACT BY LC-MS/MS	221
	<i>^{1,2}Gök Topak, ED., ¹Eylem, CC., ³Baysal I., ³Yabanoğlu-Çiftçi S., ¹KIR. S., ¹Nemutlu, E.</i>	
P118:	DEVELOPMENT AN ANALAYTICAL METHODODGY FOR ANALYSIS OF NAPROXEN SODIUM AT TRACE LEVELS in BIOLOGICAL SAMPLES BY HPLC-DAD	221
	<i>Şahin, E., Alamdar, NB., Morgül, U. Ulusoy, HI.</i>	
P119:	ELECTROANALYTICAL ANALYSIS OF GUAIFENESIN ON POLY(ACRIDINE ORANGE) MODIFIED GLASSY CARBON ELECTRODE AND ITS DETERMINATION IN PHARMACEUTICALS AND SERUM SAMPLES.....	222
	<i>Isik, H., Agin, F., Ozturk, G., Kul, D.</i>	
P120:	DEVELOPMENT AND VALIDATION OF HPLC METHOD FOR THE DETERMINATION OF IMIDUREA IN CREAM FORMULATION	223
	<i>¹Ergin Kizilçay, G., ¹Ertürk Toker, S., ²Matur D.</i>	
P121:	DEVELOPMENT OF CE-MS METHOD FOR ANALYSIS OF TRIPTORELIN	223
	<i>¹Čížmárová, I., ¹Matušková, M., ¹Čaňová, P., ^{1,2}Mikuš, P., ³Galba, J., ^{1,2}Piešťanský, J.</i>	

P122:	APPLICATION OF MAGNETIC SOLID PHASE EXTRACTION FOR PARABEN RESIDUES IN COSMETIC SAMPLES	224
	¹ Çakir, K., ² Gürbüz, A., ¹ Morgül, U., ¹ Ulusoy, Hİ.	
P123:	DEVELOPMENT OF A NOVEL HPLC-DAD-FLD-MS METHOD FOR THE SIMULTANEOUS DETERMINATION OF FIVE ANTICANCER DRUGS.....	224
	¹ Turković, L., ² Silovski, T., ³ Kostešić, M., ³ Radić, I., ¹ Nigović, B., ¹ Sertić, M.	
P124:	DEVELOPMENT OF FABRIC PHASE SORPTIVE EXTRACTION METHOD FOR DETERMINATION OF AZINPHOS-METHYL AND CHLORFENVINFOS PESTICIDES BEFORE HPLC-DAD ANALYSIS	225
	^{1,2} Sattari Dabbagh, M., ¹ Ulusoy, Hİ., ¹ Morgül, U., ³ Tartaglia, A., ⁴ Kabir, A., ³ Locatelli, M.	
P125:	DETERMINATION OF ORNIDAZOLE IN PHARMACEUTICAL DOSAGE FORMS USING BSA COATED FLUORESCENT COPPER NANOCLUSTER	226
	Bilkay, M., Satana Kara, HE.	
P126:	2D-ITP-CZE-MS/MS METHOD FOR ANALYSIS OF SEROTONIN IN URINE	226
	¹ Matušková, M., ¹ Čižmárová, I., ¹ Chałová, P., ^{1,2} Mikuš, P., ³ Kováč, A., ³ Majerová, P., ⁴ Galba J., ^{1,2} Piešťanský, J.	
P127:	DETERMINATION AND POSSIBLE MECHANISMS OF FORMATION LUMACAFTOR DEGRADATION PRODUCTS WITH USING LCMS-IT-TOF	227
	^{1,2} Özcan, S., ¹ Erdogán, Ü., ^{2,3} Levent, S., ^{1,2} Can, NÖ.	
P128:	THE NOVEL APPROACH TOWARDS GRADIENT ELUTION HPLC METHOD DEVELOPMENT	227
	Milenković, M., Djajić, N., Krmar, J., Rašević M., Malenović, A., Otašević, B., Protić, A.	
P129:	CHEMOMETRICALLY SUPPORTED OPTIMIZATION OF RP/WCX-HPLC METHOD.....	228
	Svrkota, B., Krmar, J., Djajić, N., Protić, A., Otašević, B.	
P130:	SIMULTANEOUS DETERMINATION OF SULFACETAMIDE, BETAMETHASONE, METHYL PARABEN AND PROPYL PARABEN IN PHARMACEUTICAL EYE DROP USING RP- HPLC	229
	¹ Demir, O., ¹ Kanbeş Dindar, Ç., ² Erkmen, C., ² Uslu, B., ¹ Günden Göger, N.	
P131:	SIMULTANEOUS DETERMINATION OF A BINARY MIXTURE IN A DOSAGE FORM BY CHEMOMETRIC METHODS.....	230
	Üstündağ, Ö., Dinç, E.	
P132:	APPLICATION OF CHEMOMETRIC TECHNIQUES TO THE CHROMATOGRAPHIC DATA FOR DETERMINATION OF ACTIVE COMPOUNDS IN TABLETS.....	230
	Üstündağ, Ö., Dinç, E.	
P133:	STUDY OF SPONTANEOUS REGRESSION OF CANCER AND SUBSEQUENT USE OF ADVANCED ANALYTICAL METHODS.....	230
	^{1,2} Chałová, P., ¹ Matušková, M., ¹ Čižmárová, I., ¹ Mikuš, P., ² Minichová, L., ² Škultéty, L., ² Lakota, J., ¹ Piešťanský, J., ² Galba, J.	
P134:	SPRAY DRYER OPTIMIZATION OF TEA (<i>Camellia sinensis</i> L.) EXTRACT FROM DUST CHAMBER RESIDUES AND OVEN FIBERS COUPLED WITH ARTIFICIAL INTELLIGENCE	231
	¹ İşik, S. ¹ Usman, AG. ² Aslan Erdem, S.	
P135:	DETERMINATION OF THYMOQUINONE FROM BLACK CUMIN USING HPLC TECHNIQUE: A CHEMOMETRICS BASED APPROACH.....	232
	İşik, S., Usman, AG.	

P136:	SENSITIVE DETERMINATION OF KETOPROFEN AND IBUPROFEN IN URINE SAMPLES	232
	¹ Temiz, S., ² Durgun, E. ¹ Morgül, U., ³ Ullusoy, S., ¹ Ulusoy, Hİ.	
P137:	STABILITY-INDICATING RP-HPLC METHOD FOR ROBUST DETERMINATION OF LUMACAFTOR IN THE PRESENCE OF IVACAFTOR AND ANALYSIS OF ITS PHARMACEUTICAL FORMULATION.....	233
	^{1,2} Özcan, S., ¹ Erdogan, Ü., ^{2,3} Levent, S., ^{1,2} Can, NÖ.	
P138:	ANATOMICAL EXAMINATION OF <i>FERULAGO PAUCIRADIATA</i> BOISS. & HELDR.	233
	<i>Cumhur Türker, B., Kılıç, CS.</i>	
P139:	ESSENTIAL OIL COMPOSITION OF ROOTS AND AERIAL PARTS OF <i>FERULAGO GLAREOSA</i> KANDEMİR & HEDGE	234
	¹ Kılıç, CS., ² Demirci, B., ^{2,3} Kirci, D., ⁴ Duman, H., ⁵ Gurbuz, I.	
P140:	EVALUATION OF <i>AEGOPODIUM PODAGRARIA</i> EXTRACTS IN TERMS OF CYTOTOXICITY AND ANTIOXIDANT PROPERTIES.....	234
	¹ Çiçek Polat, D., ² Yılmaz Sarıaltın, S., ³ Yalçın, CÖ.	
P141:	INVESTIGATION OF ANATOMICAL STRUCTURE OF <i>PRIMULA VERIS</i> L.....	235
	¹ Yuca, H., ² Aydin, B., ³ Karakaya, S., ¹ Guvenalp, Z.	
P142:	CHEMICAL COMPOSITIONS OF ESSENTIAL OILS OF <i>OPOPANAX HISPIDUS</i> AND <i>OPOPANAX PERSICUS</i>	236
	¹ Gümüşok, S., ² Kirci, D., ³ Demirci, B., ¹ Kılıç, CS.	
P143:	STEM AND LEAF ANATOMY OF FIVE <i>ARTEMISIA</i> L. SPECIES THAT GROW IN TURKEY.....	236
	¹ Osmannioglu Dag, SR., ² Kursat, M., ³ Gencler Ozkan, AM.	
P144:	THE EFFECT OF CONTROLLED ATMOSPHERE COMPOSITION ON CHANGES OF TRITERPENIC COMPOUNDS OF APPLE PEEL SAMPLES DURING STORAGE.....	237
	¹ Butkevičiūtė, A., ^{1,2} Liaudanskas, M., ² Viškelis, J., ² Viškelis, P., ² Bobinas, Č., ¹ Janulis, V.	
P145:	ANTIOXIDANT CAPACITY AND PHENOLIC COMPOSITION OF WHEAT GENOTYPE	237
	¹ Aydin, B., ² Ozbek, H., ² Kasil, HG., ³ Ozturk, A., ³ Kodaz, S., ⁴ Aydin, M., ³ Akkus Ekinici, S., ² Guvenalp, Z.	
P146:	ANATOMICAL CHARACTERIZATION OF <i>CERINTHE MINOR</i> L. (BORAGINACEAE)	238
	¹ Aydin, B., ² Yuca, H., ³ Karakaya, S., ² Guvenalp, Z.	
P147:	ESSENTIAL OIL ANALYSIS OF <i>HELICHRYSUM ITALICUM</i> (ROTH) G.DON WHICH IS CULTIVATED IN TURKEY	238
	<i>Yardimci Buran, B., Aslan, M.</i>	
P148:	BIOLOGICAL ACTIVITIES OF <i>PHLOMIS NISSOLII</i>	239
	¹ Eruygur, N., ¹ Kirci, D., ¹ Bosdancı, G., ¹ Doğru, T., ¹ Ayaz, F., ² Bağcı, Y.	
P149:	MICROBIAL TRANSFORMATION OF HESPERIDIN VIA HUMAN PROBIOTICS	239
	^{1,2} Kirci, D., ³ Demirci, B.	
P150:	EVALUATION OF ANTI-INFLAMMATORY ACTIVITY OF FOUR <i>HERACLEUM</i> TAXA.....	240
	¹ Kurtul, E., ² Karpuz, B., ¹ Yaylacı, B., ² Küpeli Akkol, E., ¹ Bahadır Acıkara, Ö.	
P151:	IN VITRO CARBONIC ANHYDRASE ACTIVITY OF <i>PAEONIA MASCULA</i> (L.) MILLER SUBSP. <i>ARIETINA</i> (ANDERS.) CULLEN ET HEYWOOD EXTRACTS	241

	¹ Aydin, FG., ² Türkoğlu, EA., ³ Taşkin, T.
P152:	INHIBITORY EFFECT OF SOME MEDICINAL PLANT EXTRACTS ON THIOREDOXIN REDUCTASE 241 ¹ Aydin, FG., ² Türkoğlu, EA., ³ Kuzu, M., ⁴ Taşkin, T.
P153:	CHEMICAL COMPOSITION OF <i>HYPERICUM SCABRUM</i> L. ESSENTIAL OIL 242 ¹ Yıldız, G., ² Kurkcuglu, M., ³ Kose, YB., ⁴ Baser, KHC.
P154:	ANTIOXIDANT CAPACITY AND PHENOLIC COMPOSITION OF HUSKED BARLEY GENOTYPE 242 ¹ Ozbek, H., ¹ Kasıl, HG., ² Aydin, B., ³ Ozturk, A., ³ Kodaz, S., ⁴ Aydin, M., ³ Akkus Ekinci, S., ¹ Guvenalp, Z.
P155:	ESSENTIAL OIL COMPOSITION OF DIFFERENT PARTS OF <i>ASPHODELUS AESTIVUS</i> BROT. FROM TURKEY 243 ¹ Servi, H.,
P156:	ANALYSIS OF VOLATILE COMPOUNDS OF HAWTHORN TEA 243 ¹ Servi, H., ² Yıldırım Servi, E.
P157:	ESSENTIAL OIL COMPOSITION OF <i>ONOPORDUM TAURICUM</i> WILLD. FROM TURKEY 244 ¹ Servi, H., ² Yıldırım Servi, E., ³ Doğan, A.
P158:	VOLATILE AND PHENOLIC COMPONENTS OF <i>Anthemis tinctoria</i> ssp. <i>tinctoria</i> GROWING IN TURKIYE 244 ¹ Erik, İ., ¹ Kılıç G., ¹ Şener, SÖ., ² Terzioğlu S., ¹ Yaylı, N.
P159:	BIOLOGICAL ACTIVITY GUIDED INVESTIGATION OF ANTIOXIDANT EFFECTS OF <i>TANACETUM ARMENUM</i> (DC) SCH. BIP. EXTRACTS 245 ¹ Ayçiçek, K., ² Yur, S., ¹ Göger, F., ^{2,3} Yaylaci, ÖK. ¹ Özek, G.
P160:	ANATOMICAL CHARACTERIZATION AND ESSENTIAL OIL COMPOSITION OF <i>HYPERICUM SCABRUM</i> 245 ¹ Nalkiran Ergin, K., ² Karakaya, S., ³ Demirci, B.
P161:	ESSENTIAL OIL AND FATTY ACIDS INVESTIGATION OF <i>SCABIOSA PSEUDOGRAMINIFOLIA</i> HUB.- MOR 246 ¹ Ogut K., ¹ Ozek G., ² Tekin M., ^{1,3} Ozek T.
P162:	QUALITATIVE AND QUANTITATIVE COMPOSITION OF ANTHOCYANINS IN THE FRUIT OF AMERICAN CRANBERRY (<i>VACCINIUM MACROCARPON</i> AITON) 246 ¹ Urbštaite, R., ^{1,2} Liaudanskas, M., ³ Stackevičienė, E.
P163:	BIOLOGICAL ACTIVITIES OF THE EXTRACTS AND ESSENTIAL OIL FROM <i>ANTHEMIS KOTSCHYANA</i> VAR. <i>GYPSICOLA</i> (ASTERACEAE) 247 ¹ Özek, G., ¹ Tüysüz, T., ¹ Göger, F., ^{1,2} Yaylaci, ÖK., ³ Yur, S., ^{1,3} Özek, T.
P164:	BIOLOGICAL ACTIVITIES OF <i>MALABAILA NYDEGGERİ</i> (YILD. & DINÇ) MENEMEN 248 ¹ Ayaz, F., ² Bağcı, Y., ¹ Eruygur, N., ¹ Bosdancı, G., ¹ Kirci, D., ¹ Doğru, T.
P165:	BIOLOGICAL ACTIVITIES METHANOL EXTRACTS OF <i>SMYRNium CONNATUM</i> BOISS. AND <i>KOTSCHY</i> 248 ¹ Eruygur, N., ¹ Ayaz, F., ² Bağcı, Y., ¹ Dogru, T., ¹ Kirci, D.
P166:	ANNUAL OUTLINING OF NEUROBIOLOGICAL EFFECT OF THE LEAF AND BERRY EXTRACTS AND ESSENTIAL OIL OF <i>MYRTUS COMMUNIS</i> L. 249 ¹ Erkan, N., ² Alim, E., ¹ Erdogan Orhan, I.

P167:	ANTIOXIDANT AND PROOXIDANT PROPERTIES OF <i>Citrus bergamia</i> Rissó et Poiteau (BERGAMOT) USED FOR THE MANAGEMENT OF HYPERLIPIDEMIA	249
	¹ Akyıldız, Zl., ² Kose, FA., ¹ Unver-Somer, N..	
P168:	ANTIOXIDANT AND PROOXIDANT PROPERTIES OF SELECTED HERBS USED FOR THE MANAGEMENT OF HYPERLIPIDEMIA	250
	¹ Akyıldız, Zl., ² Kose, FA., ¹ Unver-Somer, N.	
P169:	AN EVALUATION ON THE KNOWLEDGE LEVEL OF PATIENTS AND FAMILY PHYSICIANS ABOUT HERBAL PRODUCTS	251
	Erten, T., Aslan, M.	
P170:	ANTIBACTERIAL ACTIVITY OF <i>TANACETUM PARTHENIUM</i> (L.) SCH. BIP. ESSENTIAL OIL.....	251
	¹ Yıldırım Servi, E., ² Servi, H., ³ Doğan, A.	
P171:	CHEMICAL COMPOSITION AND ANTIBACTERIAL ACTIVITY OF ESSENTIAL OIL OF <i>CENTAURIUM ERYTHRAEA</i> RAFN.	252
	¹ Yıldırım Servi, E., ² Servi, H.	
P172:	THE <i>IN VITRO</i> ANTIBACTERIAL EVALUATION OF COMMERCIAL ESSENTIAL OIL OF <i>HELICHRYSUM ITALICUM</i> FROM SERBIA.....	252
	¹ Yıldırım Servi, E., ² Servi, H.	
P173:	THE SUSCEPTIBILITY OF ESBL POSITIVE <i>KLEBSIELLA</i> spp. STRAINS TO A NEWLY ISOLATED VB_K1 BACTERIOPHAGE	253
	Erol, HB., Kaskatepe, B.	
P174:	EXAMINATION OF IMMATURE GRANULOCYTE (IG) VALUES COMPLETE BLOOD COUNT IN PATIENTS WITH ACTIVE PULMONARY TUBERCULOSIS.....	253
	¹ Yalıçır, A., ¹ Yalın, S., ² Tamer, L., ³ Aslan, G.	
P175:	PRODUCTION OF THE HEMAGGLUTININ SURFACE ANTIGENIC PROTEIN OF INFLUENZA A VIRUS AS A SOLUBLE FORM IN MICROORGANISMS.....	254
	¹ Gül, AA., ² Turan, K.	
P176:	EVALUATION OF SERUM DEATH RECEPTOR 4 AND CCL5 LEVELS IN BREAST CANCER	254
	¹ Demirdögen, KK., ¹ Taskan, T., ² Noori, F., ³ Karaman, N., ² Kurukahvecioğlu, O., ¹ Gonenc, A.	
P177:	LACK OF ASSOCIATION BETWEEN VARIATIONS ON TOLL-LIKE RECEPTOR GENES AND BREAST CANCER IN MERSİN, SOUTHERN TURKEY	255
	¹ Topal, K., ¹ Akkapulu, M., ² Erçolak, V., ² Sezer, E., ¹ Yalın, AE.	
P178:	CHEMICAL, ANTIOXIDANT AND ANTIMICROBIAL PROPERTIES OF <i>Alburnus tarichi</i> ROE PROTEIN HYDROLYSATE	256
	¹ Berköz, M., ² Yunusoğlu, O., ³ Ozkan-Yılmaz, F., ³ Ozluer-Hunt, A., ⁴ Yıldırım, M., ⁵ Yalın, S., ¹ Türkmen, O.	
P179:	PROTECTIVE EFFECTS OF CURCUMIN AND NARINGENIN ON LIVER DAMAGE CAUSED BY COPPER NANOPARTICLES	256
	¹ Lalou, H., ² Yıldırım, M., ¹ Akkapulu, M., ¹ Yalın, S., ¹ Yalın, AE.	
P180:	AN INVESTIGATION ON THE ASSOCIATION BETWEEN ATP DEPENDENT POTASSIUM CHANNELS AND CORONARY ARTERY DISEASE.....	257
	¹ Seçer, D., ¹ Akkapulu, M., ² Yıldırım, M., ³ Çelik, A., ⁴ Vezir, Ö., ⁵ Sucu, N., ¹ Yalın, AE.	
P181:	NVOLVEMENT OF GENETIC VARIANTS ASSOCIATED WITH PRIMARY OPEN-ANGLE GLAUCOMA PATHOGENESIS	258
	¹ Çifçi, İF., ¹ Akkapulu, M., ² Demirci, Y., ³ Argın, MA., ⁴ Hatungil, ZE., ¹ Yalın, AE.	

P182:	INVESTIGATION OF BIOACTIVE PHYTOCHEMICALS OF <i>MATRICARIA CHAMOMILLA</i> L. AND <i>MATRICARIA DECIPIENS</i> K. KOCH AND THEIR <i>IN VITRO</i> BIOLOGICAL ACTIVITIES.....	258
	¹ Zorlu, N., ² Cakmar-Hatipoglu, SD., ¹ Ogan, A.	
P183:	IN VITRO ACETYLCHOLINESTERASE INHIBITORY ACTIVITY OF COUMARIN-SELENOPHENE HYBRID COMPOUNDS.....	259
	¹ Yildirim, M., ² Ersatir, M., ³ Akkapulu, M., ² Sultan-Giray, E., ³ Yalin, S.	
P184:	INVESTIGATION OF BIOCHEMICAL ACTION MECHANISMS OF SOME 2-HYDRAZINOTHIAZOLE DERIVATIVES.....	259
	¹ Çiyancı, ZŞ., ² Evren, AE., ² Yurttaş, L., ¹ Akalın Çiftçi, G.	
P185:	THE EFFECT OF PHARMACIST IN RATIONAL ANTIBIOTIC USE: A META ANALYSIS STUDY	260
	¹ Aydin Guldur, E., Ozcelikay, G.	
P186:	CANNABIS IN PORTUGAL: THE REBIRTH OF THE ONE THAT WAS ALREADY THE MOST IMPORTANT CULTURE IN THE COUNTRY.....	260
	¹ Paiva, C., ² Pereira, AL., ³ Pita, JR.	
P187:	PROFESSIONAL EXPERIENCES OF ENTREPRENEUR COMMUNITY PHARMACISTS	261
	¹ Yalım, İD., ² Sözen-Şahne, B.	
P188:	THE ROLE OF PHARMACIST JOAQUIM DOS SANTOS E SILVA (1842-1906) IN CINCHONA BARK AND QUININE RESEARCH IN PORTUGAL	262
	^{1,2} Semedo, M., ^{1,2} Pita, J., ^{1,3} Pereira, A.	
P189:	DETERMINATION OF PHARMACY STUDENTS' READINESS FOR INTER-PROFESSIONAL LEARNING.....	262
	¹ Baykan, RB., ² Sözen-Şahne, B.	

POSTER PRESENTATIONS

Materials and Methods: A modular capillary electrophoresis analyzer EA-102 (Villa Labeco, Spišská Nová Ves, Slovakia), assembled in the column-coupling configuration of the separation unit, was used in this work for performing the ITP-CZE runs. This CE analyzer was coupled to the triple quadrupole mass spectrometry detector (Agilent Technologies, Santa Clara, CA) via an elution block developed by Foret et al. (2).

Results: The optimized composition of electrolyte systems was: a) ITP stage – leading electrolyte (LE) = 10 mM NH₄Ac + 20 mM HAc, terminating electrolyte (TE) = 10 mM HAc; b) CZE stage – background electrolyte (BGE) = 20 mM HAc. The limit of detection was predicted at pg/ml concentration level.

Conclusions: Two dimensional capillary electrophoresis based on on-line combination of capillary isotachophoresis and capillary zone electrophoresis hyphenated with mass spectrometry is an effective and sensitive tool for serotonin determination at very low concentration levels in real biological samples.

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P127: DETERMINATION AND POSSIBLE MECHANISMS OF FORMATION LUMACAFTOR DEGRADATION PRODUCTS WITH USING LCMS-IT-TOF

^{1,2}Özcan, S., ¹Erdoğan, Ü., ^{2,3}Levent, S., ^{1,2}Can, NÖ.

¹Department of Analytical Chemistry, Faculty of Pharmacy, Anadolu University, Eskisehir, Turkey, nafizoc@anadolu.edu.tr

²Doping and Narcotic Compounds Analysis Laboratory, Faculty of Pharmacy, Anadolu University, Eskisehir, Turkey.

³Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Anadolu University, Eskisehir, Turkey.

Introduction: Lumacaftor (LUMA) is used as a combination therapy in cystic fibrosis, especially for homozygous for the F508del mutation patients (aged ≥12 years) (1). In the current study forced degradation products of LUMA were obtained, determined and possible formation mechanisms were proposed using high-resolution mass spectrometric data.

Methods: LCMS-IT-TOF analyses were performed using a hybrid IT-TOF mass spectrometer with ESI interface (Shimadzu, Japan). Analysis conditions were as follows: Nebulizing gas flow: 1.5 L/min; high voltage probe: -3.5 kV, drying gas pressure: 200 KPa, heat block temperature and CDL temperature: 200 °C. CID parameters are chosen 50% for collision gas parameter, 50% for CID energy and argon gas for CID. In addition, the detector voltage of TOF was set to 1.6 kV. The degradation conditions to which the active ingredient is exposed are made according to the ICH Q2 (R1) guideline (2).

Results: The results of forced degradation experiments revealed two new compounds in alkali and acid conditions, and three compounds in oxidation conditions. On the other hand, LUMA had no degradation in forced heat, moisture and UV-light conditions. Three of obtained degradation products had ionization in mass detector and their structure and possible formation mechanisms were proposed. The others had no ionization in mass detector, however detected using DAD detector.

Conclusion: LCMS-IT-TOF method was developed and examined for forced degradation products of LUMA. In addition to the above, new degradation products were added in to the literature.

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P128: THE NOVEL APPROACH TOWARDS GRADIENT ELUTION HPLC METHOD DEVELOPMENT

Milenković, M., Djajić, N., Krmar, J., Rašević M., Malenović, A., Otašević, B., Protić, A.

University of Belgrade – Faculty of Pharmacy, Department of Drug Analysis, Belgrade, Serbia mmilann 92@yahoo.com; nevena.maljuric@pharmacy.bg.ac.rs; jojanak@pharmacy.bg.ac.rs; marija.rasevic@pharmacy.bg.ac.rs; andja@pharmacy.bg.ac.rs; biljana.otasevic@pharmacy.bg.ac.rs; anna@pharmacy.bg.ac.rs

Introduction: Gradient elution HPLC finds its purpose in simultaneous analyses of solutes covering wide range of polarities. However, the instrument related factors, especially dwell volume,

are frequently responsible for fizzy transfer and short life cycle of the gradient elution method. Therefore, it is advisable to incorporate dwell volume into the optimization stage and avoid transfer related failures. The chemometric approach would enable selection of optimal chromatographic conditions for different HPLC instruments. The aim of this study was to propose and test this approach in gradient elution method's development.

Materials and Methods: The experiments were carried out on three chromatographic systems (UPLC, UHPLC and HPLC), while the separation was achieved on Kinetex C18 Core-shell column (100 mm × 2.1 mm, 2.6 µm particle size). Design of experiments was constructed in Design-Expert 11.0. Indirect modeling, grid point search and graphical presentations were done in Matlab 7.10.0.

Results: Dabigatran etexilate mesylate and nine structurally related compounds were selected as suitable model mixture due to its complexity and polarity. Method development was supported with experimental design methodology, Placket – Burman for screening and D-optimal design for optimization purposes. Dwell volumes were included in the optimization phase and in this way the same optimal chromatographic conditions for all three instruments were selected.

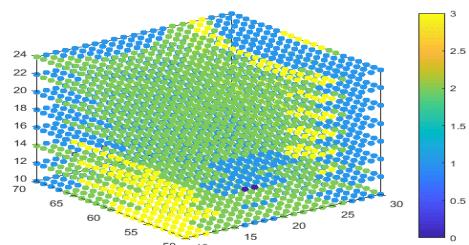


Fig 1: Experimental space overlap of instruments: yellow – 3 instruments, green – 2 instruments, light to dark blue 0 – 1 instrument

They included 10 mM ammonium acetate buffer with pH set to 4.9 using acetic acid, and acetonitrile. The components of the mobile phase were pumped into chromatographic system with flow rate of 400 µL min⁻¹ in a linear gradient mode: at 0 minutes 24% (v/v) acetonitrile and 76% (v/v) of buffer solution, at 15 minutes 54% (v/v) acetonitrile and 46% (v/v) buffer solution. At 16 minutes the acetonitrile content was back to 24% (v/v) and 76% (v/v) of buffer solution. The re-equilibration time was set to 5 minutes. The examined chromatographic region is graphically presented and optimal conditions are noticed as the cross sections (yellow dots). The method was validated and confirmed its utility on all instruments.

Conclusions: The proposed methodology demonstrated its ability to predict joint optimal chromatographic conditions for instruments with

different values of dwell volume. The potential was confirmed on complex model mixture and instruments significantly differing in dwell volume values. In this way the gap between developing and routine needs could be overwhelmed, followed by facilitated transfer of methods.

Acknowledgements

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P129: CHEMOMETRICALLY SUPPORTED OPTIMIZATION OF RP/WCX-HPLC METHOD

Svrkota, B., Krmar, J., Djajić, N., Protić, A., Otašević, B.

*University of Belgrade – Faculty of Pharmacy, Department of Drug Analysis, Belgrade, Serbia,
svrkotabojana@gmail.com,
jovana.krmar@pharmacy.bg.ac.rs,
nevena.maljuric@pharmacy.bg.ac.rs,
ana.protic@pharmacy.bg.ac.rs,
biljana.otasevic@pharmacy.bg.ac.rs*

Introduction: Active pharmaceutical ingredients (APIs) are often used in salt form, which is why the inclusion of weak cation exchange (WCX), in addition to reverse-phased (RP) hydrophobic interactions, could improve APIs' separation (1). Due to the limited knowledge about the RP/WCX bimodal system, the aim was to elucidate the experimental factors' influence on the retention of diverse ionized APIs, and provide efficient method optimization.

Materials and Methods: Acidic (ibuprofen (IB), aceclofenac (AC)) and basic (escitalopram (ES), aripiprazole (AR), atomoxetine (AT)) analytes were tested. Chromatography experiments were performed on Thermo Acclaim Mixed Mode WCX-1 (5 µm, 3x10 mm) column. Mobile phase consisted of ACN (30-50% (v/v)) and acetic buffer (pH 3.8 - 5.6; ionic strength (I) 20-40 mM). Temperature (T) was varied in range 30–38 °C. Variations of these factors were conducted according to Full Factorial Design 2⁴. Optimization phase was executed by using face-centered Central Composite Design (Design-Expert 7.0.0).

Results: Screening results showed that %ACN had the greatest impact on analytes' retention factors (k), so increasing in %ACN caused a decrease in k. T had the same effect, but much less pronounced. Changes in mobile phase pH affected k, with the opposite effect on anionic and cationic