

Dichotomies in microwave-assisted propargyl-isomerization–Claisen domino sequences dependent on base strengths†‡

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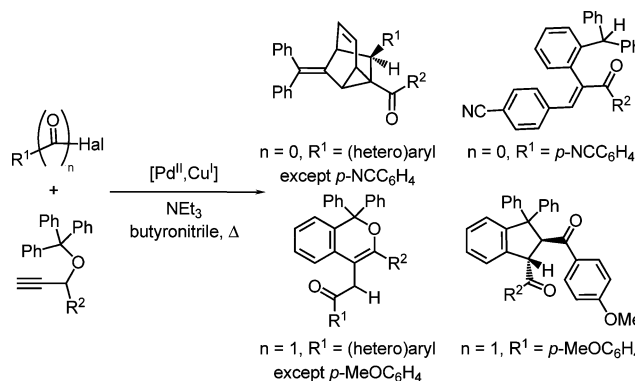
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Microwave-assisted unimolecular isomerization–Claisen domino reactions of 1,3-di(hetero)aryl propargyl trityl ethers lead, depending on the basicity of the amine, either to the formation of tricyclo[3.2.1.0^{2,7}]oct-3-enes (with triethylamine) or to indanes (with DBU). Based upon product analyses and computations, this base dependent dichotomy can be rationalized as a sequel of pericyclic reactions with intermediate protonation and deprotonation.

Introduction

In the past few years, the concept of domino reactions¹ has become almost synonymous for the rapid construction of complex structural frameworks in a one-pot fashion. With regard to synthetic efficiency and efficacy, reactive functionalities are generated and transformed in a programmed fashion without altering the reaction conditions nor adding further reagents or catalysts. Therefore, mastering unusual combinations and sequences of elementary organic reactions under identical conditions remains the major conceptual challenge in engineering novel types of domino sequences. Based upon the coupling–isomerization reaction² (CIR)—a Sonogashira coupling of electron deficient (hetero)aryl halides and (hetero)aryl propargyl alcohols followed by a base catalyzed transformation of the intermediate internal 3-aryl propargyl alcohol into a 1,3-aryl propenone—we have developed, in recent years, consecutive multi-component heterocycle syntheses initiated by transition metal catalysis³ giving rise to efficient and diverse syntheses of pharmaceutically relevant heterocycles in a one-pot fashion.⁴ Furthermore, a mechanistic expansion of CIR by etherification of the propargyl alcohol and intramolecular carbopalladation led to the formation of a new class of highly fluorescent spirocyclic benzofuranones and dihydroindolones.⁵ However, propargyl trityl ethers as alkynyl substrates set the stage for a whole multitude of domino sequels that, after CIR and Claisen rearrangement, culminated in dichotomizing final pericyclic steps with high selectivity as a consequence of minute electronic differences in the substitution pattern (Scheme 1).⁶

Since CIR can be considerably accelerated by microwave heating⁷ and encouraged by the amazing and highly selective bifurcation in the concluding steps of CI–Claisen domino reactions,⁶ the influence of dielectric heating and variation of the base strength



Scheme 1 Dichotomies in CI–Claisen domino sequences.

should render a deeper insight into the parameters of the new domino sequences. Here, we report on a series of unimolecular isomerization–Claisen domino sequences triggered by microwave heating and two bases with different base strengths.

Results and discussion

In all CIR based domino sequences, the cross-coupling event is always rapid in comparison to the isomerization step. Therefore, it is reasonable to treat both elementary steps separately and, thus, 1,3-di(hetero)aryl propargyl trityl ethers **1** are logical starting substrates for unimolecular sequences. Upon reaction of propargyl trityl ethers **1** in a 1 : 1 mixture of butyronitrile and triethylamine under microwave heating in a sealed vessel, tricyclo[3.2.1.0^{2,7}]oct-3-enes **2** were formed as sole products in excellent yields, whereas in a 1 : 1 mixture of DMSO and DBU, dielectric heating in a sealed vessel led to the selective formation of indanes **3** in good to excellent yields (Scheme 2, Table 1 and Table 2).

The structures of the tricyclo[3.2.1.0^{2,7}]oct-3-enes **2** and the indanes **3** were unambiguously supported by spectroscopic (¹H, ¹³C and DEPT, COSY, NOESY, HETCOR and HMBC NMR experiments, IR, UV–Vis, mass spectrometry) and combustion analyses. Additionally, the molecular structure of **2** was corroborated by X-ray structure analyses of compounds **2a** (Fig. 1) and **2c** (Fig. 2).§

§ Data were collected on a Bruker Smart CCD (**2c**) or a Bruker APEX diffractometer (**2a**, **11e**, **11f**). Mo K_α radiation (λ = 0.71073 Å) was used in

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† Electronic supplementary information (ESI) available: X-Ray structure data of **2a**, **2c**, **11e** and **11f**; computational studies on the intermediates and products. See DOI: 10.1039/b714351f

‡ CCDC reference numbers 660693, 660694, 641143 and 641144. For crystallographic data in CIF or other electronic format see DOI: 10.1039/b714351f

Table 1 Microwave-assisted isomerization–Claisen rearrangement–Diels–Alder domino sequence to tricyclo[3.2.1.0^{2,7}]oct-3-ene **2**^a

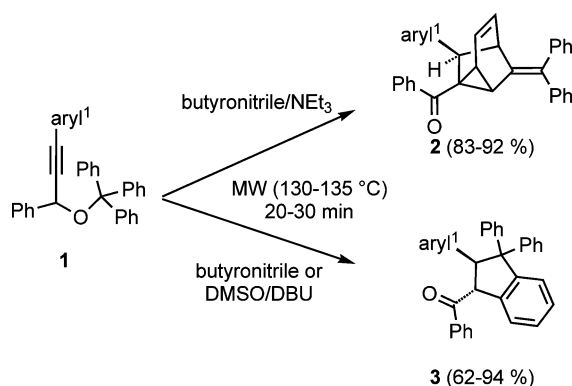
Entry	Tricyclo[3.2.1.0 ^{2,7}]oct-3-ene 2 (yield) ^b
1 ^c	2a (aryl ¹ = <i>p</i> -O ₂ NC ₆ H ₄ , 85%)
2 ^d	2b (aryl ¹ = <i>p</i> -MeO ₂ CC ₆ H ₄ , 83%)
3	2c (aryl ¹ = <i>p</i> -NCC ₆ H ₄ , 92%)
4	2d (aryl ¹ = <i>p</i> -MeC ₆ H ₄ , 0%)
5	2e (aryl ¹ = <i>p</i> -MeOC ₆ H ₄ , 0%)

^a Reaction conditions: **1** (0.2 M in butyronitrile–triethylamine 1 : 1), was dielectrically heated at 135 °C for 30 min in a sealed vial. ^b Yields refer to isolated yields of tricyclo[3.2.1.0^{2,7}]oct-3-ene **2** after flash chromatography on silica gel and were ≥95% pure as determined by NMR spectroscopy and elemental analysis. ^c After conductive heating in a coupling sequence and after isolation and recrystallization, a 91% yield of **2a** was obtained. ^d After conductive heating in a coupling sequence and after isolation and recrystallization, a 85% yield of **2b** was obtained.

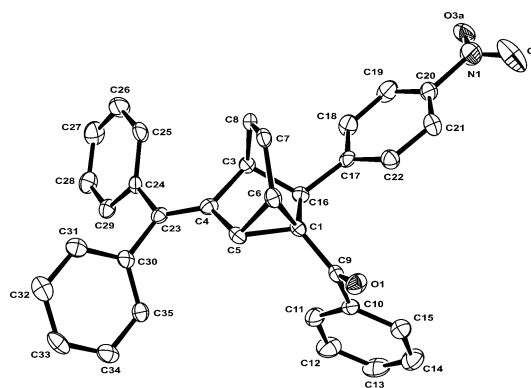
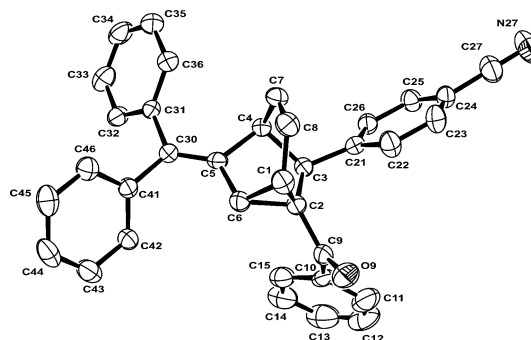
Table 2 Microwave-assisted isomerization–Claisen rearrangement–deprotonation–electrocyclization–protonation domino sequence to indanes **3**^a

Entry	Indane 3 (yield) ^b
1 ^c	3a (aryl ¹ = <i>p</i> -MeO ₂ CC ₆ H ₄ , 80%)
2	3b (aryl ¹ = Ph, 94%)
3 ^d	3c (aryl ¹ = <i>p</i> -NCC ₆ H ₄ , 62%)
4 ^e	3c (aryl ¹ = Ph, 92%)
5	3d (aryl ¹ = <i>p</i> -MeC ₆ H ₄ , 82%)
6	3e (aryl ¹ = <i>p</i> -MeOC ₆ H ₄ , 62%)

^a Reaction conditions: **1** (0.2 M in DMSO) and 1.6 eq DBU were dielectrically heated at 130 °C for 20 min in a sealed vial. ^b Yields refer to isolated yields of indanes **3** after flash chromatography on silica gel and were ≥95% pure as determined by NMR spectroscopy and elemental analysis. ^c The reaction was carried out in butyronitrile and 1.15 eq DBU at 100 °C for 18 min. ^d The reaction was carried out at 120 °C for 5 min. ^e The reaction was carried out in butyronitrile under otherwise identical conditions.

**Scheme 2** Base dependent dichotomy in microwave-assisted isomerization–Claisen domino sequences.

all cases and the intensities were corrected for absorption effects using SADABS¹⁴ based on the laue symmetry of the reciprocal space. The structures were solved by direct methods and refined against F^2 with a full matrix least square algorithm by using the SHELXTL¹⁴ software package. Hydrogen atoms were partially refined isotropically (at the core of **11e**), otherwise they were considered at calculated positions and refined using appropriate riding models. Relevant crystal and data collection parameters for the individual structures are given here. Crystal data: **2a**: C₁₇H₁₁NO₄, $M = 553.63$, monoclinic, space group $P2_1/c$, $a = 15.348(2)$, $b = 8.806(1)$,

**Fig. 1** Molecular structure of tricyclo[3.2.1.0^{2,7}]oct-3-ene **2a** (hydrogen atoms were omitted for clarity, ORTEP: 50% probability).**Fig. 2** Molecular structure of tricyclo[3.2.1.0^{2,7}]oct-3-ene **2c** (hydrogen atoms were omitted for clarity, ORTEP: 50% probability).

In consistency with the CI process carried out under conductive heating and in the presence of NEt₃ as the base,

$c = 22.612(4)$ Å, $\beta = 109.831(3)^\circ$, $V = 2874.9(7)$ Å³, $T = 100(2)$ K, $Z = 4$, $\rho = 1.28$ g cm⁻³, dimensions $0.43 \times 0.065 \times 0.04$ mm³, $\mu = 0.08$ mm⁻¹, 0.3° omega-scans, 16360 reflections measured, 3488 unique [$R(\text{int}) = 0.0536$], 2697 observed [$I > 2\sigma(I)$], 509 parameters refined, goodness of fit 1.04, $wR2 = 0.107$, $R1 = 0.048$ (observed reflections), residual electron density -0.59 to 0.54 eÅ⁻³, CCDC 660693. **2c**: C₃₅H₂₅NO, $M = 475.6$, triclinic, space group $P1$, $a = 12.1762(2)$, $b = 17.4387(2)$, $c = 20.1060(1)$ Å, $\alpha = 80.239(1)^\circ$, $\beta = 73.766(1)^\circ$, $\gamma = 73.137(1)^\circ$, $V = 3904.56(8)$ Å³, $T = 200(2)$ K, $Z = 6$, $\rho = 1.21$ g cm⁻³, crystal dimensions $0.40 \times 0.24 \times 0.08$ mm³, $\mu = 0.07$ mm⁻¹, 0.3° omega-scans, 26587 reflections measured, 10487 unique [$R(\text{int}) = 0.0491$], 7415 observed [$I > 2\sigma(I)$], 1000 parameters refined, goodness of fit 1.03, $wR2 = 0.107$, $R1 = 0.049$ (observed reflections), residual electron density -0.23 to 0.37 eÅ⁻³, CCDC 641143. **11e**: C₂₉H₂₄O, $M = 388.5$, triclinic, space group $P1$, $a = 8.5190(2)$, $b = 12.5850(3)$, $c = 10.2410(1)$ Å, $\alpha = 73.0570(1)^\circ$, $\beta = 86.7680(1)^\circ$, $\gamma = 98.8770(9)^\circ$, $V = 1031.73(4)$ Å³, $T = 200(2)$ K, $Z = 2$, $\rho = 1.25$ g cm⁻³, crystal dimensions $0.49 \times 0.17 \times 0.06$ mm³, $\mu = 0.07$ mm⁻¹, 0.3° omega-scans. The crystals turned out to be partially merohedrally twinned, so an artificially enlarged unit cell had to be used to be able to integrate all the reflections, 6151 reflections of solely component A, 6201 reflections of solely component B, and 2026 reflections belonging to both components result in 14378 observations measured, 9690 observed [$I > 2\sigma(I)$], 3505 after merging equivalents, 285 parameters refined, goodness of fit 1.22, $wR2 = 0.110$, $R1 = 0.048$ (observed reflections), residual electron density -0.17 to 0.17 eÅ⁻³, CCDC 641144. **11f**: C₂₉H₂₄O₂, $M = 404.48$, orthorhombic, space group $Pccn$, $a = 15.277(3)$, $b = 37.640(8)$, $c = 7.251(2)$ Å, $V = 4169.4(15)$ Å³, $T = 200(2)$ K, $Z = 8$, $\rho = 1.289$ g cm⁻³, dimensions $0.18 \times 0.12 \times 0.05$ mm³, $\mu = 0.08$ mm⁻¹, 0.3° omega-scans, 10547 reflections measured, 2173 unique [$R(\text{int}) = 0.0737$], 1621 observed [$I > 2\sigma(I)$], 281 parameters refined, goodness of fit 1.08, $wR2 = 0.133$, $R1 = 0.061$ (observed reflections), residual electron density -0.20 to 0.21 eÅ⁻³, CCDC 660694.

tricyclo[3.2.1.0^{2,7}]oct-3-enones **2** are the products of the intramolecular isomerization–Claisen sequence. However, increasing the base strength from NEt₃ ($pK_b = 3.35$) to DBU ($pK_b = 1.16$) favors the formation of indanes **3**. Furthermore, the stronger base DBU obviously also triggers the propyne–allene isomerization which does not occur with electron-donating aryl substituents in the 3-position when applying NEt₃ as the base (Table 1, entries 4 and 5 vs. Table 2, entries 3–6).

However, the concluding steps in this domino sequence seem to be relatively complex. For an insight into the energetic scenario, quantum chemical calculations on structures and energies of the propargyl trityl ether **1c**, the tentative intermediates **4c** and **5c**, and the possible reaction products **2c**, **3c**, and **6c** were carried out on high levels of theory (Fig. 3).⁸

Geometry optimizations were carried out using Becke Perdew (BP) 86 functionals [RBP86/6-31+G(d,p)]⁹ and based upon single point energy calculations using the Møller–Plesset correlation energy correction truncated at second-order [RMP2/6-311++G(2d,2p)//RBP86/6-31+G(d,p)].¹⁰ energetic estimations propose some profound mechanistic implications. Starting from the propargyl trityl ether **1c**, isomerization of the alkyne furnishes the allenyl trityl ether **4c** with a thermodynamic driving force of 5.75 kcal mol^{−1}. This allenyl intermediate is quite in agreement with other sequences that are based upon the coupling–isomerization reaction.^{2,5,6} Then, intermediate **4c** sets the stage for an allenyl–benzyl Claisen rearrangement giving rise to the formation of a *exo*-methylene cyclohexadiene enone **5c**, which is less than 1 kcal mol^{−1} more stable than **4c**. According to the product analysis, now, the bifurcation takes place. Computing

the energies of the possible products, which are indeed observed under special conditions, *i.e.* tricyclo[3.2.1.0^{2,7}]oct-3-ene **2c** (under microwave irradiation and with triethylamine as a base), 2-substituted triphenylmethyl enone **6c** (under conductive heating and with triethylamine as a base),⁶ and indane **3c** (under microwave irradiation and with DBU as a base), reveals interesting aspects of this sequence. Obviously, the thermodynamically most stable isomer is indane **3c** (−61.23 kcal mol^{−1}) and it is only formed at higher temperatures (microwave heating) and if the base is strong enough (DBU). Interestingly, the thermodynamically more favorable enone **6c** (−44.41 kcal mol^{−1}) is not formed if the energy is just high enough (microwave heating) to trigger an intramolecular [4 + 2]-cycloaddition as a kinetically controlled formation of the least favorable tricyclo[3.2.1.0^{2,7}]oct-3-ene **2c** (−20.46 kcal mol^{−1}). Only prolonged conductive heating furnishes the more stable isomer **6c** if the base is triethylamine. However, under dielectric heating in combination with sufficient base strength, kinetic control is apparently dominating by rapid energy transfer to the reactants in the vessel. Here again, as reported before,⁶ minute electronic differences in combination with the mode of heating and the basicity determine the bifurcation of isomerization–pericyclic sequences. Hence, triethylamine as a base is definitely strong enough to catalyze the isomerization from alkyne **1** to allene **4**, but it fails to catalyze the isomerization from Claisen rearrangement product **5** to indane **3** or enone **6c**. This can be accounted for by the intermediacy on an anion generated upon deprotonation with a sufficiently strong base. Otherwise, if no anion can be rapidly generated, the obvious reaction is the fast intramolecular cycloaddition that should proceed at higher rates than intermolecular processes.

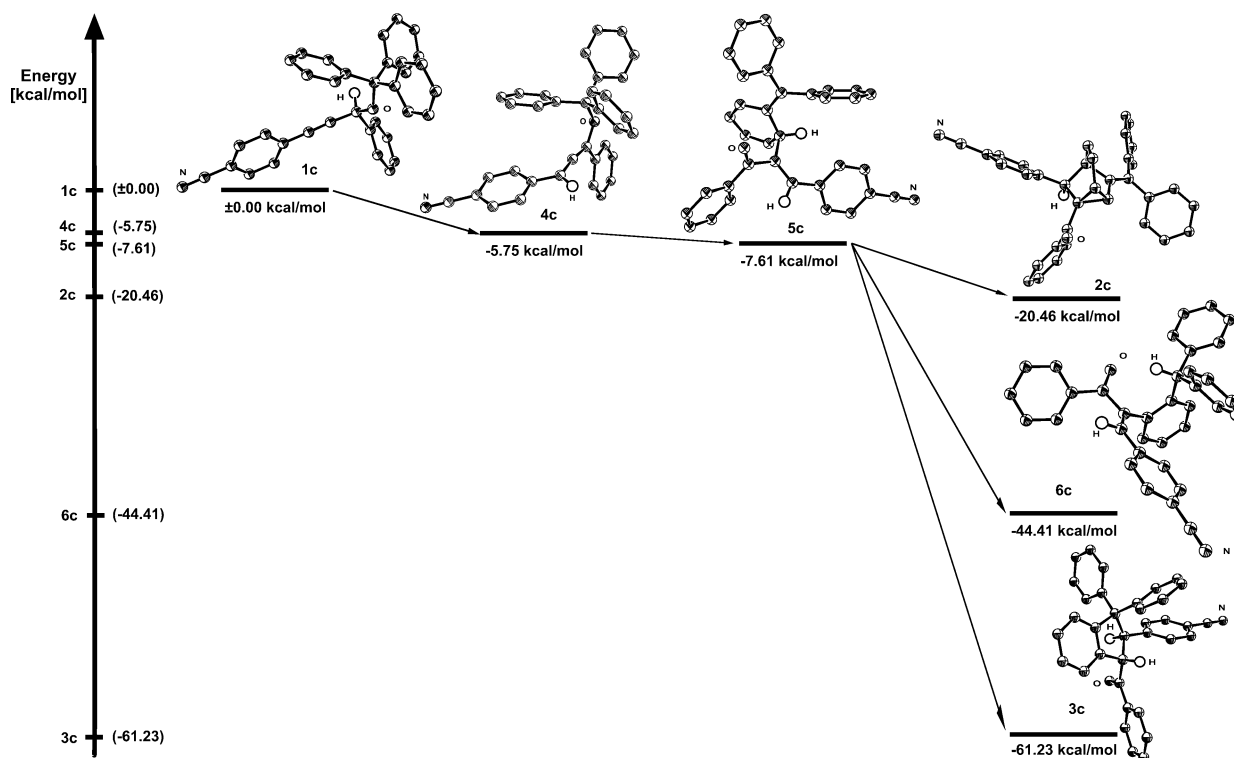
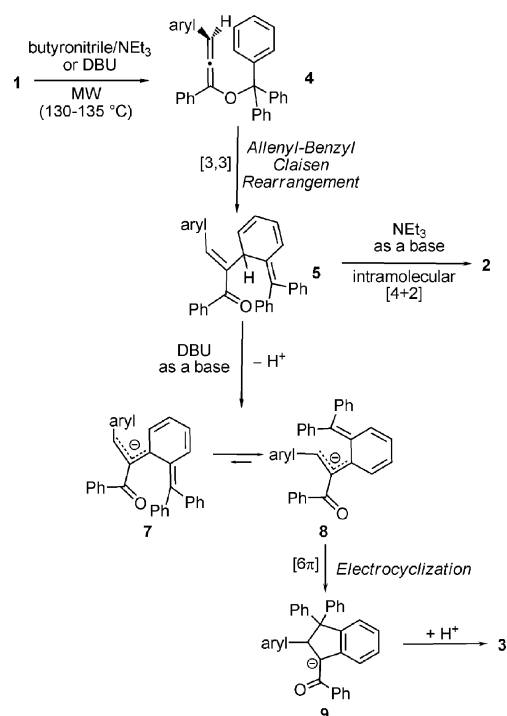


Fig. 3 Computed energies (RBP86/6-31+G(d,p) for geometries and RMP2/6-311++G(2d,2p)//RBP86/6-31+G(d,p) for energies) of the propargyl trityl ether **1c**, the tentative intermediates **4c** and **5c**, and possible reaction products **2c**, **3c**, and **6c**.

Based upon these energetic considerations, the tentative mechanism of these bifurcating domino reactions can be rationalized as follows (Scheme 3).



Scheme 3 Mechanistic rationale for the base dependent dichotomy in microwave-assisted isomerization-Claisen domino sequences.

After the propyne-allene isomerization-Claisen rearrangement, the cyclohexadienyl proton is susceptible to deprotonation with a sufficiently strong base. The presence of a weak base leads to an intramolecular [4 + 2] cycloaddition that immediately establishes the tricyclo[3.2.1.0^{2,7}]oct-3-ene framework of **2**, whereas deprotonation with DBU generates delocalized carbanions **7** and **8**, which are prone to *cis-trans* interconversion. However, the carbanion isomer **8** culminates in a 6 π -electrocyclization with concomitant rearomatization and generation of enolate **9**. Finally, protonation of **9** gives rise to the formation of the thermodynamically most stable *anti*-configured indane **3**. Hence, if equilibration is possible, the thermodynamic outcome of an isomerization-Claisen sequence with **1** as a substrate is always the indane motif **3**.

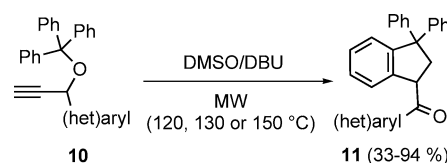
As a consequence, we also tested whether an aryl substituent in the 3-position of **1** is necessary at all to favor the generation of indane derivatives. Therefore, trityl ethers **10** with terminal alkyne functionality were submitted to the isomerization conditions in DMSO and with DBU as a base to selectively furnish 1-(hetero)aryl 3,3-diphenylindanes **11** in moderate to excellent yields (Scheme 4, Table 3).

The unambiguous structural assignments were based on extensive spectroscopic and combustion analyses. Additionally, the molecular structure of **11** was corroborated by X-ray structure analyses of compounds **11e** (Fig. 4) and **11f** (Fig. 5).§

Table 3 Isomerization-Claisen rearrangement-deprotonation-electrocyclization-protonation domino sequence to 1-(hetero)aryl 3,3-diphenylindanes **11**^a

Entry	1-(Hetero)aryl 3,3-diphenylindane 5 (yield) ^b
1 ^c	11a ((het)aryl = <i>p</i> -NCC ₆ H ₄ , 33%)
2	11b ((het)aryl = <i>p</i> -ClC ₆ H ₄ , 94%)
3	11c ((het)aryl = C ₆ H ₅ , 94%)
4 ^d	11d ((het)aryl = 2-thienyl, 84%)
5	11e ((het)aryl = <i>p</i> -MeC ₆ H ₄ , 90%)
6	11f ((het)aryl = <i>p</i> -MeOC ₆ H ₄ , 92%)
7 ^e	11g ((het)aryl = <i>p</i> - ⁿ hexyOC ₆ H ₄ , 89%)

^a Reaction conditions: **4** (0.2 M in DMSO) and 1.6 eq of DBU were dielectrically heated at 150 °C for 20 min in a sealed vial. ^b Yields refer to isolated yields of indanes **5** after flash chromatography on silica gel and were $\geq 95\%$ pure as determined by NMR spectroscopy and elemental analysis. ^c The reaction was carried out in butyronitrile using 1.6 eq DBU at 120 °C for 15 min. ^d The reaction was carried out at 130 °C for 10 min. ^e The reaction was carried out at 150 °C for 15 min.



Scheme 4 Microwave-assisted isomerization-Claisen-cyclization domino sequence to 1-(hetero)aryl 3,3-diphenylindanes **11**.

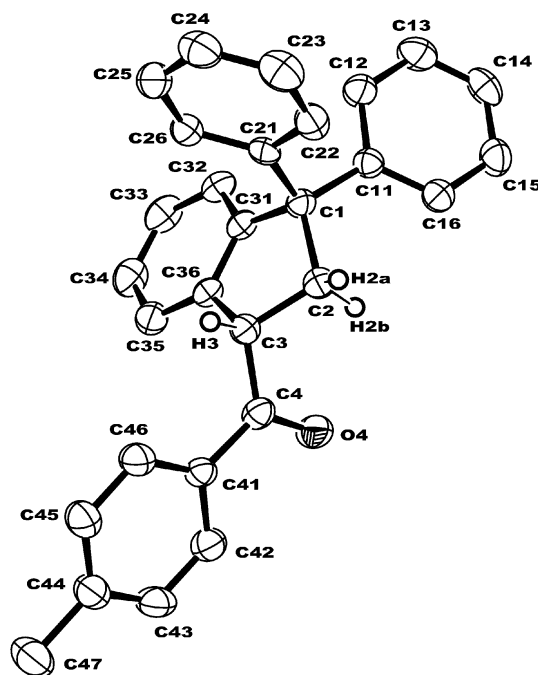


Fig. 4 Molecular structure of 1-(hetero)aryl 3,3-diphenylindane **11e** (most hydrogen atoms were omitted for clarity, ORTEP: 50% probability).

Conclusions

In conclusion, we have found that base strength exercises a considerable influence in microwave-assisted dichotomizing unimolecular isomerization-Claisen sequences. Particularly, computations and product analyses support the tentative mechanistic rationale where bifurcation occurs after the Claisen step as a consequence

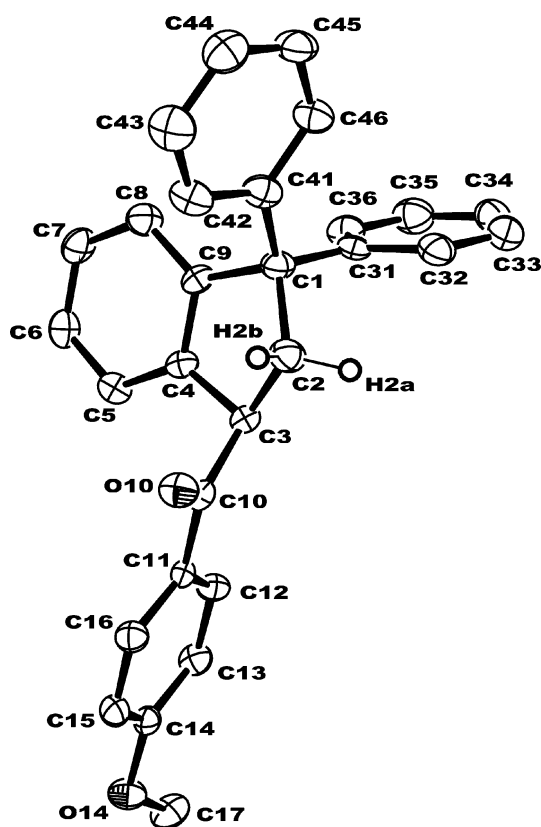


Fig. 5 Molecular structure of 1-(hetero)aryl 3,3-diphenylindane **11f** (most hydrogen atoms were omitted for clarity, ORTEP: 50% probability).

of the generation of anionic intermediates in the presence of sufficiently strong bases. Interestingly, excellent chemoselectivities underline the well balanced electronic interplay in concluding pericyclic steps. Both tricyclo[3.2.1.0^{2,7}]oct-3-enes **2** and indanes **3** or **11** are intriguing frameworks and are suitable for synthetic and methodological elaboration. In particular, highly substituted indanes display biological activity such as pharmacological¹¹ and olfactory properties.¹² Studies addressing the synthetic scope of these new domino reactions are currently under investigation.

Experimental

General considerations. All reactions involving water-sensitive compounds were carried out in oven-dried vials under a nitrogen atmosphere. The solvents were dried according to standard procedures¹³ and were distilled prior to use. Column chromatography: silica gel 60 M (mesh 230–400) Macherey-Nagel. Thin layer chromatography (TLC): silica gel layered aluminium foil (60 F₂₅₄ Merck, Darmstadt). Melting points (uncorrected): Reichert-Jung Thermovar and Büchi Melting Point B-540. 4-Iodobenzonitrile **4** was purchased from ACROS and used without further purification. Trityl propargyl ethers **2** were prepared from propargyl alcohols obtained by literature procedures.² ¹H and ¹³C NMR spectra: Bruker ARX250, Bruker DRX 300 with CDCl₃ as solvent. The assignments of quaternary C, CH, CH₂ and CH₃ were made on the basis of DEPT spectra. IR: Bruker Vector 22 FT-IR. UV-Vis: Hewlett Packard HP8452 A. MS: Jeol JMS-700 und Finnigan TSQ 700. Elemental analyses were carried out in the

microanalytical laboratory of the Organisch-Chemisches Institut der Universität Heidelberg.

General procedure for the synthesis of terminal propargyl trityl ethers **10** and internal propargyl trityl ethers **1**

Synthesis of compounds **10.** A detailed experimental procedure for the synthesis of compounds **10** has been previously published.⁶

Synthesis of compounds **1.** To a deaerated mixture of a magnetically stirred THF solution of compound **7** and 1.00 equivalent of the corresponding aryl halides in a Schlenk flask with a positive nitrogen atmosphere was gradually added 5 mol% of PdCl₂(PPh₃)₂, 5 mol% of CuI, and 1.05 equivalents of triethylamine. The reaction mixture was stirred at room temp until complete consumption of the aryl halide (monitored by TLC). For work up, the reaction mixture was diluted with ether, submitted to filtration for removal of the ammonia salts and the solvents were evaporated *in vacuo*. The residue was filtered on silica gel (hexane–ethyl acetate 10 : 1 up to 5 : 1) to give the internal propargyl trityl ethers **1** in quantitative yields as light yellow solids, which were submitted to the isomerization–Claisen sequences without further purification.

General procedure for the isomerization–Claisen sequences to the products **2**, **3**, or **11**

A magnetically stirred deaerated solution of compound **1** or **4** (0.3 mmol) in 3.0 mL of a mixture of butyronitrile and NEt₃ (1 : 1, v/v) was dielectrically heated in a sealed microwave vial at 135 °C for 30 min (see Table 4 for experimental details). After cooling to rt, the solvents were removed *in vacuo*, and the residue was chromatographed on silica gel to give rise to the desired products **2**, **3**, or **11**, respectively.

Table 4 Experimental details of CI–Claisen sequences^a

Entry	Propargyl trityl ether 1 or 7	Sequence products 2 , 3 , or 11 (yield) ^b
1 ^a	149 mg (0.3 mmol) of 1a	127 mg (85%) of 2a
2 ^b	153 mg (0.3 mmol) of 1b	127 mg (83%) of 2b
3	143 mg (0.3 mmol) of 1c	131 mg (92%) of 2c
4 ^c	153 mg (0.3 mmol) of 1b	122 mg (80%) of 3a
5 ^d	143 mg (0.3 mmol) of 1c	89 mg (62%) of 3b
6 ^e	135 mg (0.3 mmol) of 1d	127 mg (94%) of 3c
7 ^e	139 mg (0.3 mmol) of 1e	114 mg (82%) of 3d
8 ^e	144 mg (0.3 mmol) of 1f	89 mg (62%) of 3e
9 ^f	120 mg (0.3 mmol) of 4a	40 mg (33%) of 11a
10	123 mg (0.3 mmol) of 4b	116 mg (94%) of 11b
11	112 mg (0.3 mmol) of 4c	105 mg (94%) of 11c
12 ^g	114 mg (0.3 mmol) of 4d	96 mg (84%) of 11d
13	117 mg (0.3 mmol) of 4e	105 mg (90%) of 11e
14	121 mg (0.3 mmol) of 4f	111 mg (92%) of 11f
15 ^h	142 mg (0.3 mmol) of 4g	126 mg (89%) of 11g

^a Yields of a thermal conventionally heated bimolecular reaction after isolation and recrystallization (91%). ^b Yields of a thermal conventionally heated bimolecular reaction after isolation and recrystallization (85%).

^c The reaction was carried out in butyronitrile and 1.15 eq DBU at 100 °C for 18 min. ^d The reaction was carried out in DMSO and 1.6 eq DBU at 120 °C for 5 min. ^e The reaction was carried out in DMSO and 1.6 eq DBU at 130 °C for 20 min. ^f The reaction was carried out in butyronitrile using 1.6 eq DBU at 120 °C for 15 min. ^g The reaction was carried out at 130 °C for 10 min. ^h The reaction was carried out at 150 °C for 15 min.

6-Benzhydrylidene-8-(4-nitro-phenyl)-tricyclo[3.2.1.0^{2,7}]-oct-3-en-1-yl]-phenyl-methanone (2a)

Mp. 174–176 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.95–2.92 (m, 1 H), 3.25–3.21 (m, 1 H), 3.65–3.61 (m, 1 H), 4.14 (d, *J* = 6.0 Hz, 1 H), 5.61–5.55 (m, 1 H), 6.27–7.22 (m, 1 H), 7.17 (d, *J* = 7.8 Hz, 2 H), 7.51–7.24 (m, 13 H), 7.71–7.68 (m, 2 H), 7.97 (d, *J* = 9.0 Hz, 2 H). ¹³C NMR (125 MHz, CDCl₃): δ 27.5 (CH), 33.2 (CH), 43.3 (C_{quat}), 45.8 (CH), 46.8 (CH), 123.0 (CH), 125.2 (CH), 126.1 (CH), 127.2 (CH), 127.4 (CH), 127.5 (CH), 128.2 (CH), 128.3 (CH), 128.5 (CH), 129.1 (CH), 129.7 (CH), 129.8 (CH), 132.3 (CH), 136.3 (C_{quat}), 137.0 (C_{quat}), 138.2 (C_{quat}), 141.6 (C_{quat}), 146.2 (C_{quat}), 146.5 (C_{quat}), 199.9 (C_{quat}). EI MS (70 eV, *m/z* (%)): 495 (M⁺, 100), 165 (C₁₃H₉⁺, 51); HRMS (70 eV, EI) calcd. for C₃₄H₂₅NO₃: 495.1834; found, 495.1822. IR (KBr) 1666, 1598, 1517 cm^{−1}. Anal. calcd. for C₃₄H₂₅NO₃ (495.6): C, 82.40; H, 5.08; N, 2.83; found: C, 82.04; H, 5.52; N, 2.69%.

4-(8-Benzhydrylidene-7-benzoyl-tricyclo[3.2.1.0^{2,7}]-oct-3-en-6-yl)-benzoic acid methyl ester (2b)

Mp. 197–199 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.87 (dd, *J* = 7.9 Hz, *J* = 1.2 Hz, 1 H), 3.25–3.20 (m, 1 H), 3.63–3.59 (m, 1 H), 3.82 (s, 3 H), 4.10 (d, *J* = 4.5 Hz, 1 H), 5.59–5.53 (m, 1 H), 6.23–6.18 (m, 1 H), 7.07 (d, *J* = 8.1 Hz, 2 H), 7.44–7.22 (m, 13 H), 7.69–7.67 (m, 2 H), 7.77 (d, *J* = 8.7 Hz, 2 H). ¹³C NMR (125 MHz, CDCl₃): δ 27.4 (CH), 33.5 (CH), 43.3 (C_{quat}), 45.8 (CH), 46.9 (CH), 51.9 (CH₃), 125.0 (CH), 126.1 (CH), 126.3 (CH), 127.2 (CH), 127.5 (CH), 128.1 (CH), 128.2 (CH), 128.3 (CH), 128.4 (CH), 129.1 (CH), 129.8 (CH), 129.8 (CH), 132.1 (CH), 135.9 (C_{quat}), 137.7 (C_{quat}), 138.5 (C_{quat}), 141.7 (C_{quat}), 141.7 (C_{quat}), 143.7 (C_{quat}), 166.8 (C_{quat}), 200.4 (C_{quat}). EI MS (70 eV, *m/z* (%)): 508 (M⁺, 100), 165 (M⁺ − C₆H₅CO, 52); HRMS (70 eV, EI) calcd. for C₃₆H₂₈O₃: 508.2038; found, 508.2043. IR (KBr) 1724, 1669, 1611, 1598 cm^{−1}. Anal. calcd. for C₃₆H₂₈O₃ (508.6): C, 85.01; H, 5.55; found: C, 84.89; H, 5.59%.

4-(8-Benzhydrylidene-7-benzoyl-tricyclo[3.2.1.0^{2,7}]-oct-3-en-6-yl)-benzonitrile (2c)

Mp. 174–176 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.90 (d, *J* = 7.4 Hz, 1 H), 3.20 (m, 1 H), 3.60 (m, 1 H), 4.10 (d, *J* = 4.2 Hz, 1 H), 5.57 (m, 1 H), 6.24–6.21 (m, 1 H), 7.12 (d, *J* = 8.1 Hz, 2 H), 7.48–7.24 (m, 15 H), 7.69 (d, *J* = 7.2 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 27.4 (CH), 33.1 (CH), 43.1 (C_{quat}), 45.7 (CH), 46.9 (CH), 110.1 (C_{quat}), 118.8 (C_{quat}), 125.1 (CH), 126.1 (CH), 127.2 (CH), 127.3 (CH), 127.5 (CH), 128.1 (CH), 128.2 (CH), 128.5 (CH), 129.0 (CH), 129.7 (CH), 131.5 (CH), 132.2 (CH), 136.2 (C_{quat}), 137.1 (C_{quat}), 138.2 (C_{quat}), 141.6 (C_{quat}), 141.6 (C_{quat}), 143.9 (C_{quat}), 199.9 (C_{quat}). EI MS (70 eV, *m/z* (%)): 475 (M⁺, 100), 307 (M⁺ − C₆H₅CO, 53). HRMS (70 eV, EI) calcd. for C₃₅H₂₅NO: 475.1936; found: 475.1954. IR (KBr): ν_{max} 3045 (w), 2227 (s), 1668 (s), 1608 (s), 1598 (s) cm^{−1}. Anal. calcd. for C₃₅H₂₅NO (475.2): C, 88.39; H, 5.30; N, 2.95; found: C, 88.26; H, 5.34; N 2.95%.

4-(3-Benzoyl-1,1-diphenyl-indan-2-yl)-benzoic acid methyl ester (3a)

Mp. 204–205 °C. ¹H NMR (500 MHz, CDCl₃): δ 3.81 (s, 3 H), 5.35 (d, *J* = 11.0 Hz, 1 H), 5.62 (d, *J* = 10.5 Hz, 1 H), 6.33 (d,

J = 7.0 Hz, 2 H), 6.79–6.77 (m, 1 H), 6.85 (d, *J* = 8.0 Hz, 2 H), 7.04–6.99 (m, 3 H), 7.24–7.13 (m, 3 H), 7.30–7.27 (m, 1 H), 7.39–7.36 (m, 2 H), 7.50–7.47 (m, 2 H), 7.61–7.58 (m, 3 H), 7.65 (d, *J* = 8.0 Hz, 2 H), 8.01 (d, *J* = 7.0 Hz, 2 H). ¹³C NMR (125 MHz, CDCl₃): δ 51.9 (CH), 53.5 (CH₃), 58.2 (CH), 65.7 (C_{quat}), 123.6 (CH), 126.6 (CH), 126.7 (CH), 126.7 (CH), 127.1 (CH), 127.5 (CH), 128.2 (CH), 128.2 (CH), 128.6 (C_{quat}), 128.8 (CH), 128.9 (CH), 128.9 (CH), 129.2 (CH), 129.4 (CH), 130.5 (CH), 133.6 (CH), 137.8 (C_{quat}), 140.9 (C_{quat}), 141.4 (C_{quat}), 143.8 (C_{quat}), 144.7 (C_{quat}), 149.3 (C_{quat}), 166.9 (C_{quat}), 198.6 (C_{quat}). EI MS (70 eV, *m/z* (%)): 508 (M⁺, 52), 403 (M⁺ − C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₃₆H₂₈O₃: 508.2038; found, 508.2015. IR (KBr) ν_{max} 1722, 1682, 1600 cm^{−1}. Anal. calcd. for C₃₆H₂₈O₃ (508.6): C, 85.01; H, 5.55; found: C, 85.05; H, 5.59%.

Phenyl-(2,3,3-triphenyl-indan-1-yl)-methanone (3b)

Mp. 178–179 °C. ¹H NMR (300 MHz, CDCl₃): δ 5.35 (d, *J* = 10.5 Hz, 1 H), 5.59 (d, *J* = 9.9 Hz, 1 H), 6.35–6.32 (m, 2 H), 6.79–6.74 (m, 3 H), 7.04–6.94 (m, 6 H), 7.23–7.12 (m, 3 H), 7.29–7.27 (m, 1 H), 7.39–7.34 (m, 2 H), 7.50–7.45 (m, 2 H), 7.63–7.58 (m, 3 H), 8.03–8.00 (m, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 53.5 (CH), 58.3 (CH), 65.5 (C_{quat}), 123.6 (CH), 126.4 (CH), 126.5 (CH), 126.6 (CH), 126.8 (CH), 127.4 (CH), 127.6 (CH), 128.0 (CH), 128.0 (CH), 128.8 (CH), 128.9 (CH), 129.3 (CH), 130.6 (CH), 133.4 (CH), 138.0 (C_{quat}), 138.1 (C_{quat}), 141.2 (C_{quat}), 141.8 (C_{quat}), 145.1 (C_{quat}), 149.6 (C_{quat}), 199.0 (C_{quat}). EI MS (70 eV, *m/z* (%)): 450 (M⁺, 51), 345 (M⁺ − C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₃₄H₂₆O: 450.1984; found, 450.2014. IR (KBr) ν_{max} 1686, 1579 cm^{−1}. Anal. calcd. for C₃₄H₂₆O (450.6): C, 90.63; H, 5.82; found: C, 90.41; H, 5.91%.

4-(3-Benzoyl-1,1-diphenyl-indan-2-yl)-benzonitrile (3c)

Mp. 176–178 °C. ¹H NMR (300 MHz, CDCl₃): δ 5.32 (d, *J* = 10.5 Hz, 1 H), 5.64 (d, *J* = 10.8 Hz, 1 H), 6.32 (d, *J* = 8.5 Hz, 2 H), 6.79–6.77 (m, 1 H), 6.88 (d, *J* = 8.0 Hz, 2 H), 7.08–6.99 (m, 3 H), 7.41–7.15 (m, 8 H), 7.63–7.49 (m, 5 H), 8.02 (d, *J* = 8.0 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 53.4 (CH), 57.8 (CH), 65.7 (C_{quat}), 110.6 (C_{quat}), 118.8 (C_{quat}), 123.6 (CH), 126.6 (CH), 126.9 (CH), 126.9 (CH), 127.2 (CH), 127.6 (CH), 128.3 (CH), 128.4 (CH), 128.9 (CH), 129.0 (CH), 129.1 (CH), 130.0 (C_{quat}), 130.4 (CH), 131.3 (CH), 133.8 (CH), 137.6 (C_{quat}), 140.4 (C_{quat}), 141.2 (C_{quat}), 144.1 (C_{quat}), 144.3 (C_{quat}), 149.0 (C_{quat}), 198.1 (C_{quat}). EI MS (70 eV, *m/z* (%)): 475 (M⁺, 83), 370 (M⁺ − C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₃₅H₂₅NO: 475.1936; found, 475.1938. IR (KBr) ν_{max} 2233, 1682, 1618, 1608 cm^{−1}. Anal. calcd. for C₃₅H₂₅NO (475.2): C, 88.39; H, 5.30; N, 2.95; found: C, 88.36; H, 5.29; N, 2.93%.

(3,3-Diphenyl-2-*p*-tolyl-indan-1-yl)-phenyl-methanone (3d)

Mp. 184–186 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.17 (s, 3 H), 5.30 (d, *J* = 10.8 Hz, 1 H), 5.53 (d, *J* = 11.1 Hz, 1 H), 6.35 (d, *J* = 6.9 Hz, 2 H), 6.65 (d, *J* = 8.7 Hz, 2 H), 6.79–6.76 (m, 3 H), 7.06–6.97 (m, 3 H), 7.19–7.13 (m, 3 H), 7.29–7.24 (m, 1 H), 7.37–7.38 (m, 2 H), 7.50–7.45 (m, 2 H), 7.61–7.56 (m, 3 H), 8.00 (d, *J* = 7.5 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 21.0 (CH₃), 53.6 (CH), 58.2 (CH), 65.4 (C_{quat}), 123.6 (CH), 126.3 (CH), 126.5 (CH), 126.6 (CH), 126.8 (CH), 127.3 (CH), 128.0 (CH), 128.0 (CH), 128.2 (CH), 128.8 (CH), 128.9 (CH), 129.3 (C_{quat}), 129.4 (CH), 130.7

(CH), 133.4 (CH), 134.9 (C_{quat}), 136.3 (C_{quat}), 138.1 (C_{quat}), 141.4 (C_{quat}), 142.0 (C_{quat}), 145.2 (C_{quat}), 149.7 (C_{quat}), 199.1 (C_{quat}). EI MS (70 eV, *m/z* (%)): 464 (M⁺, 37), 359 (M⁺ – C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₃₅H₂₈O: 464.2140; found, 464.2128. IR (KBr) $\nu_{\max}$ 1683, 1596 cm⁻¹. Anal. calcd. for C₃₅H₂₈O (464.6): C, 90.48; H, 6.07; found: C, 90.41; H, 6.09%.

[2-(4-Methoxy-phenyl)-3,3-diphenyl-indan-1-yl]-phenyl-methanone (3e)

Mp. 205–206 °C. ¹H NMR (300 MHz, CDCl₃): δ 3.65 (s, 3 H), 5.26 (d, *J* = 11.3 Hz, 1 H), 5.52 (d, *J* = 11.3 Hz, 1 H), 6.35 (d, *J* = 8.6 Hz, 2 H), 6.51 (d, *J* = 8.5 Hz, 2 H), 6.69 (d, *J* = 8.6 Hz, 2 H), 6.80–6.77 (m, 1 H), 7.07–7.00 (m, 3 H), 7.19–7.14 (m, 3 H), 7.29–7.25 (m, 2 H), 7.38–7.34 (m, 2 H), 7.50–7.46 (m, 2 H), 7.61–7.56 (m, 3 H), 8.02–7.99 (m, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 53.8 (CH), 55.0 (CH₃), 57.8 (CH), 65.4 (C_{quat}), 112.9 (CH), 123.6 (CH), 126.1 (CH), 126.4 (CH), 126.5 (CH), 126.8 (CH), 127.3 (CH), 128.0 (CH), 128.3 (CH), 128.8 (CH), 128.9 (CH), 129.3 (CH), 130.1 (C_{quat}), 130.3 (CH), 130.7 (CH), 133.4 (CH), 138.0 (C_{quat}), 141.3 (C_{quat}), 142.0 (C_{quat}), 145.2 (C_{quat}), 149.6 (C_{quat}), 158.4 (C_{quat}), 199.2 (C_{quat}). EI MS (70 eV, *m/z* (%)): 480 (M⁺, 22), 375 (M⁺ – C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₃₅H₂₈O₂: 480.2089; found, 480.2114. IR (KBr) $\nu_{\max}$ 1681, 1613 cm⁻¹. Anal. calcd. for C₃₅H₂₈O₂ (480.6): C, 87.47; H, 5.87; found: C, 87.41; H, 5.94%.

4-(3,3-Diphenyl-indane-1-carbonyl)-benzonitrile (11a)

Mp. 157–158 °C. ¹H NMR (300 MHz, CDCl₃): δ 3.11 (dd, *J* = 6.3, 12.6 Hz, 1 H), 3.31 (dd, *J* = 10.2, 12.6 Hz, 1 H), 4.86 (dd, *J* = 6.3, 9.9 Hz, 1 H), 7.01 (d, *J* = 7.5 Hz, 1 H), 7.06 (d, *J* = 6.9 Hz, 1 H), 7.34–7.10 (m, 12 H), 7.79 (d, *J* = 8.4 Hz, 2 H), 8.07 (d, *J* = 8.4 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 47.1 (CH₂), 50.5 (CH), 60.9 (C_{quat}), 116.6 (C_{quat}), 117.8 (C_{quat}), 124.8 (CH), 126.4 (CH), 126.6 (CH), 126.8 (CH), 127.4 (CH), 127.7 (CH), 128.0 (CH), 128.2 (CH), 128.4 (CH), 128.5 (CH), 129.2 (CH), 132.7 (CH), 140.3 (C_{quat}), 140.7 (C_{quat}), 145.4 (C_{quat}), 147.0 (C_{quat}), 149.5 (C_{quat}), 198.6 (C_{quat}). EI MS (70 eV, *m/z* (%)): 399 (M⁺, 12), 269 (M⁺ – *p*-NCC₆H₄CO, 100); HRMS (70 eV, EI) calcd for C₂₉H₂₁NO: 399.1628; found, 408.1631. IR (KBr) $\nu_{\max}$ 2232, 1689, 1600 cm⁻¹. Anal. calcd. for C₂₉H₂₁NO (399.5): C, 87.19; H, 5.30; N, 3.51; found: C, 87.32; H, 5.38; N, 3.47%.

(4-Chloro-phenyl)-(3,3-diphenyl-indan-1-yl)-methanone (11b)

Mp. 180–181 °C. Anal. ¹H NMR (300 MHz, CDCl₃): δ 3.08 (dd, *J* = 6.4, 12.7 Hz, 1 H), 3.35 (dd, *J* = 10.2, 12.7 Hz, 1 H), 4.85 (dd, *J* = 6.4, 10.8 Hz, 1 H), 7.09–7.01 (m, 2 H), 7.33–7.16 (m, 12 H), 7.47 (d, *J* = 8.6 Hz, 2 H), 7.95 (d, *J* = 8.6 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 47.1 (CH₂), 50.1 (CH), 60.8 (C_{quat}), 124.7 (CH), 126.3 (CH), 126.5 (CH), 126.6 (CH), 127.3 (CH), 127.5 (CH), 128.2 (CH), 128.5 (CH), 128.6 (CH), 128.9 (CH), 129.1 (CH), 130.3 (CH), 135.7 (C_{quat}), 139.8 (C_{quat}), 141.3 (C_{quat}), 145.5 (C_{quat}), 147.3 (C_{quat}), 149.5 (C_{quat}), 198.5 (C_{quat}). EI MS (70 eV, *m/z* (%)): 410 (³⁷Cl-M⁺, 5), 408 (³⁵Cl-M⁺, 15), 269 (³⁵Cl-M⁺ – *p*-³⁵ClC₆H₄CO, 100); HRMS (70 eV, EI) calcd for C₂₈H₂₁³⁵ClO: 408.1281; found, 408.1275. IR (KBr) $\nu_{\max}$ 1682, 1588 cm⁻¹. Anal. calcd. for C₂₈H₂₁ClO (408.9): C, 82.24; H, 5.18; Cl, 8.67; found: C, 89.59; H, 5.23; Cl, 8.37%.

(3,3-Diphenyl-indan-1-yl)-phenyl-methanone (11c)

Mp. 168–169 °C. ¹H NMR (300 MHz, CDCl₃): δ 3.09 (dd, *J* = 6.2, 12.3 Hz, 1 H), 3.36 (dd, *J* = 10.3, 12.3 Hz, 1 H), 4.92 (dd, *J* = 6.2, 10.3 Hz, 1 H), 7.08–7.04 (m, 2 H), 7.33–7.15 (m, 12 H), 7.48 (t, *J* = 7.0 Hz, 2 H), 7.61–7.57 (m, 1 H), 8.02–8.00 (m, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 47.1 (CH₂), 50.0 (CH), 60.8 (C_{quat}), 124.9 (CH), 126.2 (CH), 126.4 (CH), 126.5 (CH), 127.2 (CH), 127.4 (CH), 128.0 (CH), 128.1 (CH), 128.5 (CH), 128.6 (CH), 128.8 (CH), 128.8 (CH), 133.3 (CH), 137.5 (C_{quat}), 141.7 (C_{quat}), 145.7 (C_{quat}), 147.4 (C_{quat}), 149.5 (C_{quat}), 199.8 (C_{quat}). EI MS (70 eV, *m/z* (%)): 374 (M⁺, 42), 269 (M⁺ – C₆H₅CO, 100); HRMS (70 eV, EI) calcd for C₂₈H₂₂O: 374.1671; found, 374.1678. IR (KBr) $\nu_{\max}$ 1680, 1596 cm⁻¹. Anal. calcd. for C₂₈H₂₂O (374.5): C, 89.81; H, 5.92; found: C, 89.57; H, 5.94%.

(3,3-Diphenyl-indan-1-yl)-thiophen-2-yl-methanone (11d)

Mp. 200–201 °C. ¹H NMR (300 MHz, CDCl₃): δ 3.12 (dd, *J* = 7.1, 12.8 Hz, 1 H), 3.38 (dd, *J* = 10.7, 12.8 Hz, 1 H), 4.75 (dd, *J* = 7.1, 10.7 Hz, 1 H), 7.33–7.06 (m, 15 H), 7.68 (d, *J* = 5.1 Hz, 1 H), 7.73 (d, *J* = 5.1 Hz, 1 H). ¹³C NMR (75 MHz, CDCl₃): δ 47.4 (CH₂), 51.6 (CH), 61.0 (C_{quat}), 124.8 (CH), 126.2 (CH), 126.4 (CH), 126.5 (CH), 127.3 (CH), 127.5 (CH), 128.0 (CH), 128.1 (CH), 128.3 (CH), 128.4 (CH), 128.6 (CH), 132.8 (CH), 134.4 (CH), 141.6 (C_{quat}), 144.8 (C_{quat}), 145.6 (C_{quat}), 147.4 (C_{quat}), 149.5 (C_{quat}), 192.7 (C_{quat}). EI MS (70 eV, *m/z* (%)): 382 (³⁴S-M⁺, 3), 380 (³²S-M⁺, 34), 269 (³²S-M⁺ – C₄H₃SCO, 100); HRMS (70 eV, EI) calcd for C₂₆H₂₀OS: 380.1235; found, 380.1261. IR (KBr) $\nu_{\max}$ 1659, 1653 cm⁻¹. Anal. calcd. for C₂₆H₂₀OS (380.5): C, 82.07; H, 5.43; S, 8.43; found: C, 81.84; H, 5.30; S, 8.41%.

(3,3-Diphenyl-indan-1-yl)-*p*-tolyl-methanone (11e)

Mp. 176–178 °C. ¹H NMR (300 MHz, CDCl₃): δ 2.43 (s, 3 H), 3.07 (dd, *J* = 6.4, 12.8 Hz, 1 H), 3.37 (dd, *J* = 10.9, 12.8 Hz, 1 H), 4.89 (dd, *J* = 6.4, 10.9 Hz, 1 H), 7.08–7.04 (m, 2 H), 7.31–7.17 (m, 14 H), 7.92 (d, *J* = 7.8 Hz, 1 H). ¹³C NMR (75 MHz, CDCl₃): δ 21.7 (CH₃), 47.1 (CH₂), 49.9 (CH), 60.8 (C_{quat}), 124.9 (CH), 126.2 (CH), 126.4 (CH), 126.5 (CH), 127.1 (CH), 127.3 (CH), 128.0 (CH), 128.1 (CH), 128.5 (CH), 128.6 (CH), 129.0 (C_{quat}), 129.4 (CH), 129.5 (CH), 135.0 (C_{quat}), 141.9 (C_{quat}), 145.8 (C_{quat}), 147.5 (C_{quat}), 149.5 (C_{quat}), 199.3 (C_{quat}). EI MS (70 eV, *m/z* (%)): 388 (M⁺, 20), 119 (*p*-H₃CC₆H₅CO⁺, 100); HRMS (70 eV, EI) calcd for C₂₉H₂₄O: 388.1827; found, 388.1813. IR (KBr) $\nu_{\max}$ 1674, 1604 cm⁻¹. Anal. calcd. for C₂₉H₂₄O (388.5): C, 89.66; H, 6.23; found: C, 89.49; H, 6.26%.

(3,3-Diphenyl-indan-1-yl)-(4-methoxy-phenyl)-methanone (11f)

Mp. 153–154 °C. ¹H NMR (300 MHz, CDCl₃): δ 3.06 (dd, *J* = 7.1, 12.6 Hz, 1 H), 3.39 (dd, *J* = 10.3, 12.6 Hz, 1 H), 3.88 (s, 3 H), 4.88 (dd, *J* = 7.1, 10.2 Hz, 1 H), 6.97 (d, *J* = 9.1 Hz, 2 H), 7.08–7.04 (m, 2 H), 7.34–7.16 (m, 12 H), 8.01 (d, *J* = 8.8 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃): δ 47.1 (CH₂), 49.6 (CH), 55.5 (CH₃), 60.8 (C_{quat}), 113.9 (CH), 124.8 (CH), 126.2 (CH), 126.3 (CH), 126.5 (CH), 127.1 (CH), 127.2 (CH), 127.9 (CH), 128.1 (CH), 128.5 (CH), 128.6 (CH), 130.5 (C_{quat}), 131.2 (CH), 142.0 (C_{quat}), 145.8 (C_{quat}), 147.6 (C_{quat}), 149.5 (C_{quat}), 163.7 (C_{quat}), 198.1 (C_{quat}). EI MS (70 eV, *m/z* (%)): 404 (M⁺, 22), 135 (*p*-H₃COC₆H₄CO⁺, 100);

HRMS (70 eV, EI) calcd for $C_{29}H_{24}O_2$: 404.1776; found, 404.1801. IR (KBr) 1673, 1600 cm^{-1} . Anal. calcd. for $C_{29}H_{24}O_2$ (404.5): C, 86.11; H, 5.98; found: C, 86.00; H, 6.01%.

(3,3-Diphenyl-indan-1-yl)-(4-hexyloxy-phenyl)-methanone (11g)

Mp. 118–119 °C. 1H NMR (300 MHz, $CDCl_3$): δ 0.93–0.89 (m, 3 H), 1.38–1.31 (m, 4 H), 1.50–1.45 (m, 2 H), 1.83–1.76 (m, 2 H), 3.05 (dd, $J = 6.4, 12.7$ Hz, 1 H), 3.39 (dd, $J = 10.6, 13.4$ Hz, 1 H), 4.03 (d, $J = 6.4$ Hz, 1 H), 4.87 (dd, $J = 6.4, 10.6$ Hz, 1 H), 6.95 (d, $J = 8.6$ Hz, 2 H), 7.08–7.03 (m, 2 H), 7.32–7.16 (m, 12 H), 8.00 (d, $J = 9.1$ Hz, 2 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.0 (CH_3), 22.6 (CH_2), 25.0 (CH_2), 29.0 (CH_2), 31.5 (CH_2), 47.1 (CH_2), 49.6 (CH), 60.8 (C_{quat}), 68.9 (CH_2), 114.4 (CH), 124.8 (CH), 126.1 (CH), 126.4 (CH), 126.5 (CH), 127.1 (CH), 127.2 (CH), 127.9 (CH), 128.1 (CH), 128.5 (CH), 128.6 (CH), 130.2 (C_{quat}), 131.2 (CH), 142.1 (C_{quat}), 145.8 (C_{quat}), 147.6 (C_{quat}), 149.5 (C_{quat}), 163.4 (C_{quat}), 198.1 (C_{quat}). EI MS (70 eV, m/z (%)): 474 (M^+ , 3), 205 (p -hexyloxy- $C_6H_4CO^+$, 100); HRMS (70 eV, EI) calcd for $C_{34}H_{34}O_2$: 474.2559; found, 474.2553. IR (KBr) ν_{max} 1797, 1673, 1600 cm^{-1} . Anal. calcd. for $C_{34}H_{34}O_2$ (474.7): C, 86.04; H, 7.22; found: C, 86.12; H, 7.19%.

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Notes and references

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