A Stereoselective and Regioselective Synthesis of trans,trans-Configured 1,2,3-Trisubstituted Indanes: Cycloaddition of Alkenes with Iodonium Ylides of β-Disulfones

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Dedicated to Prof. Dr. Dieter Seebach (ETH Zürich) on the occasion of his 65th birthday, an appreciated friend and excellent colleague.

Abstract: The reaction of phenyliodonium-bis(sulfonyl) methylylides with alkenes affords the multiply trisubstituted indanes in moderate to good yields, through an unusual cycloaddition. The present stereoselective and regioselective cycloaddition provides a convenient preparative route to trans,trans-configured 1,2,3-trisubstituted indanes, in which the benzene ring derives from the arene-sulfonyl functionality of the bis(sulfonyl)iodonium ylide. The mechanistically puzzling structural feature is the fact (X-ray structure) that the para-methyl substituent of the original p-toluenesulfonfonyl group in the iodonium ylide is located in the C-6 position of the resulting indane benzene ring, that is, a meta relationship with respect to the original methylide carbon atom.

Key words: cycloaddition, 1,2,3-trisubstituted indanes, iodonium ylides, diastereoselective synthesis

The reaction of phenyliodonium-bis(sulfonyl) methylylides, a rare class of reactive ylides, expectedly affords the corresponding cyclopropanes, especially under photochemical and copper(II)-catalyzed thermal activation (Scheme 1). Presumably, analogous to the related bis(sulfonyl) diazomethanes, carbene or carbenoid reactivity manifests itself in the form of [2+1] cycloaddition. An exception is the thermal reaction of the ylide with trans-stilbene (1a, R2 = Ph), which leads to a trisubstituted indane, evidently through an unusual cycloaddition process, whose mechanism is still little understood. Nevertheless, an elaborate 1,2,3-trisubstituted indane ring system is produced in a single step from simple and readily available starting materials. Provided the stereochemical course of this unusual cycloaddition process may be defined and its scope generalized, a potentially useful synthetic method would become accessible for the preparation of multiply substituted indanes. Numerous synthetic sequences for such indanes have been reported over the years, but these are hardly as convenient and effective as the direct cycloaddition of iodonium methylylides to olefins (Scheme 1). Moreover, it should be appreciated that the sulfonyl substituent may be reductively cleaved to afford 2,3-disubstituted indanes of determined configuration. Also, the sulfonyl-bearing position may be appropriately modified through established carbanion methodology and thereby the synthetic utility of such indane chemistry increased.

Scheme 1

The incentive of the present study was to examine the scope of the indane-forming process and rigorously determine whether the trans,trans configuration of the 1,2,3-trisubstituted indanes is formed from the aryl substituted olefins. Of the iodonium ylides chosen for this work, the last two derivatives should establish whether the aryl group of the ylide is the source of the benzene ring in the resulting indane. The regiochemistry with regard to the R substituent was also of concern, i.e., is the R group located in the C-5 or the C-6 position; the latter regioisomer is shown in Scheme 2.

Scheme 2
Indeed, the results presented herein demonstrate that the reaction between iodonium ylides and olefins constitutes a convenient and efficient cycloaddition for the stereoselective and regioselective preparation of trans,trans-configured 1,2,3-trisubstituted indanes, with the R substituent in the C-6 position of the benzene ring.

The disulfoniliodonium ylides α–γ were readily prepared[^2] from the corresponding disulfones 3 by treatment with iodobenzene diacetate and KOH as base at –10 °C (Scheme 2). These labile ylides may be stored at –10 °C (Scheme 2). These labile ylides may be stored at –30 °C for several weeks without decomposition. They are practically insoluble in common organic solvents (except DMSO), so that the reaction of the ylides with the olefins is very sluggish. Since the reaction of the ylide with the olefin under these conditions, the extent of olefin cleavage was minimal, e.g., for the trans-stilbene, trans-1a/ylide α pair only about 3% benzaldehyde was observed.

All reactions of the ylides with the alkenes were carried out in the presence of a catalytic amount of Rh₂(OAc)₄; in the absence of the catalyst, much longer reaction times were necessary, but the same product composition was obtained. The indanes 2 (Table 1) were isolated by flash chromatography on silica gel in moderate yields (up to 56%, relative to the ylide), the remainder of the ylides were converted to the corresponding disulfones 3. It should be emphasized that when referred to the amount of olefin consumed, high yields (up to 90%) of indanes 2 are registered (Table 1, last column). This indicates that an efficient and clean process operates, except that substantial amounts (up to 62%) of the ylide are diverted to the disulfones through decomposition.

As for the specific cases, the reaction of ylide α with either trans- or cis-stilbene (1a) afforded in both cases exclu-

### Table 1  Cycloaddition of Iodonium Ylides α–γ with Olefins 1a–c[^4]

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<td>27</td>
<td>63</td>
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[^2]: All reactions were carried out by stirring a suspension of ylide (1 equiv) and olefin 1 (2.78 equiv) in the presence of a catalytic amount of Rh₂(OAc)₄.
[^3]: In MeCN, except entries 1, 2, 5, 6, 8, and 9, which were run in a 5:1 mixture of MeCN–CH₂Cl₂.
[^4]: Time required for complete consumption of the ylide.
[^5]: Yield of isolated product (relative to 100% conversion of the ylide) after silica gel chromatography.
[^6]: Normalized to 100% conversion of the olefin; the conversion of the olefin was 16–24%.
sively the indane *trans*-2aβ in 86 and 80% yields (Table 1, entries 1 and 2). The replacement of one of the phenyl groups of the stilbene by a methyl or a benzoyl group had no effect on the reactivity of these substrates towards the ylide α. Thus, the reaction of the ylide α with *trans*-1-phenylprop-1-ene (1b) and the chalcone 1c gave the indanes *trans*-2bα and *trans*-2cα in 55 and 90% yields (Table 1, entries 3 and 4).

The cycloaddition was extended to the ylide β, whose *p*-methyl substituent of the phenylsulfonyl moiety allowed to assess which phenyl group (ylide or the olefin) is the source of the benzene ring in the indane 2 product. Furthermore, it permitted to determine the regioselectivity of the cycloaddition process with regard to the location of the R substituent in the benzene ring. The reaction of ylide β with *trans*- or *cis*-stilbene (1a) was much faster and afforded only one diastereomer, namely the *trans*-2aβ indane in 80 and 77% yields (Table 1, entries 5 and 6). Analogously, the ylide β reacted with *trans*-1-phenylprop-1-ene (1b) to give the indane *trans*-2bβ in 59% yield (Table, entry 7).

The unsymmetric ylide γ was selected to determine whether the aliphatic sulfonyl group still possesses sufficient reactivity for cycloaddition. Indeed, under the same conditions, the ylide γ and *trans*- or *cis*-stilbene (1a) gave in both cases the indane *trans*-2ary 79 and 71% yields (Table 1, entries 8 and 9). Likewise, the reaction of the ylide γ with *trans*-1-phenylprop-1-ene (1b) led to 63% of the indane *trans*-2ary (Table 1, entry 10). The structural assignment of the indanes is exemplified for the *trans*-2aβ derivative. The 1H NMR spectrum of the indane *trans*-2aβ displays a triplet signal at δ = 3.57 with J = 7.4 Hz for the proton at C-2, a doublet at δ = 4.25 with J = 7.4 Hz for the proton at C-3, and a doublet at δ = 5.04 with J = 7.4 Hz for the proton at C-1. The lack of a ROESY signal between the protons at C-1 and C-2 positions and the protons at C-2 and C-3 positions indicates the *trans*,*trans* configuration of the three substituents in the five-membered ring. An X-ray analysis of this cycloadduct (Figure 1) confirmed this stereochemical assignment. Furthermore, the X-ray structure reveals that the methyl group of the benzene ring and the sulfonyl-bearing C-1 position of the five-membered ring are located *meta* to one another in the product, although originally this methyl substituent occupied the *para* position in the *para*-toluenesulfonyl group of the iodonium ylide.

The direct cycloaddition of the bis(sulfonyl)iodonium methyldides α–γ with the aryl substituted olefins 1 presented herein is to date undoubtedly the most effective and convenient method for the diastereoselective synthesis of trisubstituted indanes 2. Since the required iodonium ylides α–γ are readily accessible by condensation of bis(sulfonyl)benzenes 3 with the commercially available (diacetoxyiodo)benzene, combined with the reductive removal of the sulfonyl functionality, this novel process constitutes an attractive diastereoselective route for the preparation of *trans*-disubstituted indanes. Unfortunately, the *cis*-stilbene is isomerized under the reaction conditions to the more stable *trans*-stilbene; nonetheless, the advantage of this shortcoming is that irrespective of the initial olefin geometry, the same *trans*,*trans*-trisubstituted indane is obtained diastereoselectively, such that even isomeric mixtures of the starting olefin may be used.

Although the synthetic utility of this unusual cycloaddition is quite evident, the mechanism of this complex reaction is still poorly understood, and we may only speculate. A carbene or metal carbenoid mechanism is ruled out for the following reasons: The mild thermal reaction conditions make it unlikely that ylides α–γ decompose into phenyl iodide and the bis(sulfonyl)carbene, as is the case in the thermal Cu(acac)2-catalyzed decomposition or in the photolysis. Moreover, in the absence of the olefin substrate, the iodonium-bis(arylsulfonyl) methylides thermally decompose to the bis(sulfonyl)methanes, phenyl benzenethiosulfonate (PhSO2SPh) and carbon dioxide. In addition, even if the carbene had been generated, it takes place under the metal-catalyzed or photochemical conditions, the resulting cyclopropanes should have been sufficiently persistent for isolation. Instead of rearranging to the observed 6-methyl-2,3-diphenyl-1-sulfonyledane (2αβ), obtained from the cycloaddition between stilbene 1a and ylide β, the carbene cycloadduct (Scheme 3) would be expected to undergo dipolar ring opening, reclosure and aromatization to afford the 1,1-bis(sulfonyl)-2-phenylindane, in which the benzene ring stems from the olefin and not, as observed (see X-ray structure in Figure 1), from the ylide. It is difficult to conceive rational pathways to transform the carbene-derived indane (Scheme 3) to the observed 2αβ (Scheme 2).

With the carbene route discounted, a reasonable mechanism must account for the following experimental facts: a) The olefin substrate is isomerized (*cis*- to *trans*-stilbene); b) the benzene ring is derived from the ylide phenyl group (Figure 1); c) the initial *para*-methyl group in the aryl group of the ylides β and γ acquires a *meta* regiochemistry.
The generated directly by electrophilic attack of the iodonium group would afford the observed 1,2,3-trisubstituted indane, and the latter leads on coupling to the dipolar intermediate to afford the radical ion experimental facts a–d. Either electron transfer takes deliberately chosen because it illustrates appropriately the given for the (well understood) to account these experimental facts is A speculative but plausible mechanism (the role of the rhodium catalyst has not been considered since it is not known, as well as trivalent iodine compounds, but no known, 5 as well as trivalent iodine compounds, 8 and bis(sulfonyl)iodonium methylides α-γ through an unusual direct cycloaddition. This direct process manifests advantages over established synthetic methods for such multiply substituted indanes, since the trans,trans-configuration of the three substituents in the five-membered ring is well controlled, as is the regiochemistry of these substituents and that of the benzene ring.

A speculative but plausible mechanism (the role of the rhodium catalyst has not been considered since it is not well understood) to account these experimental facts is given for the cis-stilbene (1a)/ylide γ pair in Scheme 4, deliberately chosen because it illustrates appropriately the experimental facts a–d. Either electron transfer takes place between the two reactants to afford the radical ion pair and the latter leads on coupling to the dipolar intermediate A, or alternatively, the dipolar species A may be generated directly by electrophilic attack of the iodonium ylide. The cis- to trans-isomerization of stilbene cis-1a (fact a) may occur at the stage of the olefin radical cation 1a+ or in the dipolar intermediate A due to steric repulsion between the olefinic phenyl groups to form eventually the thermodynamically preferred trans,trans-configured (fact d) 1,2,3-trisubstituted indane. Nucleophilic attack by the bis(sulfonyl)-centered carbanion on the phenyl ring of the ylide γ and closure of the resulting dipole leads to the tricyclic trivalent iodine species B (this accounts for the facts b and c). Thietane S,S-dioxides are known, as well as trivalent iodine compounds, but no cases appear to have so far been documented for this strained combination. Reductive elimination of iodobenzene from the hypervalent iodine compound would afford intermediate C, with the remaining sulfonyl group trans-configured (fact d). On sulfur dioxide elimination (fact e) from the highly strained thietane S,S-dioxide and subsequent aromatization by hydrogen shift would afford the observed 1,2,3-trisubstituted indane 2αγ (Scheme 4). Consequently, the two phenyl groups in the 2,3 position and the sulfonyl functionality are both trans-configured (fact d), while the original para-methyl group of the p-toluenesulfonyl substituent in the ylide γ is regioselectively placed at the C-6 position of the benzene ring (facts b and c). Finally, it should be noted that for unsymmetrical olefin partners like β-methylstyrene (1b) and the chalcone 1c, the dipolar intermediate A would prefer to localize the positive charge at the phenyl terminal during its formation (fact f).

In conclusion, irrespective of the mechanistic complexities, we have developed a convenient one-step synthesis of multiply substituted indanes 2 from readily accessible olefins 1 and bis(sulfonyl)iodonium methylides α-γ through an unusual direct cycloaddition. This direct process manifests advantages over established synthetic methods for such multiply substituted indanes, since the trans,trans-configuration of the three substituents in the five-membered ring is well controlled, as is the regiochemistry of these substituents and that of the benzene ring.

The solvent was removed (20 °C/10 Torr) and the residue was chromatographed on silica gel with mixtures of CH2 Cl2 and petroleum ether as eluent. Iodobenzene was eluted first, subsequently followed by the indanes 2a, 2α, 2bβ, and 2bγ (Table 1).

Scheme 3

in the final indane product (Figure 1); d) the trans,trans-configured 1,2,3-trisubstituted indane is exclusively formed (Figure 1); e) sulfur dioxide is eliminated (detected by control experiment); and f) for the unsymmetrical olefins 1b and 1c, the phenyl group is regioselectively placed at the C-3 position of the benzene ring. Consequently, the two phenyl groups in the 2,3 position and the sulfonyl functionality are both trans-configured (fact d), while the original para-methyl group of the p-toluenesulfonyl substituent in the ylide γ is regioselectively placed at the C-6 position of the benzene ring (facts b and c). Finally, it should be noted that for unsymmetrical olefin partners like β-methylstyrene (1b) and the chalcone 1c, the dipolar intermediate A would prefer to localize the positive charge at the phenyl terminal during its formation (fact f).

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Reaction of Ylide $^{a}$ with trans-Stilbene or cis-Stilbene (1a)

A suspension of ylide $^{a}$ (500 mg, 1.0 mmol) and trans- or cis-stilbene (1a: 500 mg, 2.78 mmol) in a mixture of MeCN–CH$_2$Cl$_2$ (5:1, 10 mL) was stirred for 100 h (trans-stilbene) or 8 h (cis-stilbene) according to the above general procedure. The solvent was evaporated (20 °C/10 Torr) and the residue was chromatographed on silica gel [eluent: CH$_2$Cl$_2$–petroleum ether, 4:1] to yield 230 mg (56%) from trans-1a or 200 mg (48%) from cis-1a of the 2,3-diphenyl-1-(phenylsulfonyl)indane (trans-2a) at an olefin conversion of 24% for trans-1a and 22% for cis-1a; colorless plates; mp 142–143 °C (Lit. 2a mp 139–141 °C).

IR (KBr): 3050, 3020, 2890, 1595, 1580, 1490, 1475, 1445, 1310, 1300, 1290, 1280, 1210, 1195, 1150, 1130, 1100, 1080, 1025, 865, 830 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $^a$= 3.63 (t, 1 H, $J$ = 7.4 Hz), 4.33 (d, 1 H, $J$ = 7.4 Hz), 5.13 (d, 1 H, $J$ = 7.4 Hz), 6.74 (d, 2 H, $J$ = 6.6 Hz), 6.86–6.88 (m, 2 H), 6.94 (d, 1 H, $J$ = 7.6 Hz), 7.14–7.20 (m, 6 H), 7.31–7.52 (m, 5 H), 7.69 (d, 2 H, $J$ = 7.7 Hz), 7.96 (d, 1 H, $J$ = 7.6 Hz).

$^{13}$C NMR (100 MHz, CDCl$_3$): $^a$= 56.9 (d), 60.7 (d), 76.2 (d), 125.6 (d), 126.4 (d), 126.8 (d), 126.9 (d), 127.3 (d), 128.2 (t), 128.5 (t), 128.6 (t), 128.8 (t), 129.4 (t), 129.7 (d), 133.5 (d), 134.2 (s), 137.1 (s), 141.9 (s), 142.9 (s), 146.8 (s).

Reaction of Ylide $^a$ with trans-1-Phenylprop-1-ene (1b)

A suspension of the ylide $^a$ (500 mg, 1.0 mmol) and trans-1-phenylprop-1-ene (328 mg, 2.78 mmol) in MeCN (10 mL) was stirred for 70 h according to the above general procedure. The solvent was evaporated (20 °C/10 Torr) and the residue was chromatographed on silica gel [eluent: CH$_2$Cl$_2$–petroleum ether, 1:1) to yield 90.0 mg (26%) of the 2-methyl-3-phenyl-1-(phenylsulfonyl)indane (trans-2b) at an olefin conversion of 24%; colorless cubes; mp 141–142 °C.

IR (KBr): 2970, 2890, 1500, 1480, 1455, 1450, 1315, 1305, 1285, 1245, 1210, 1135, 1090, 1030, 1005, 815 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $^a$= 1.24 (d, 3 H, $J$ = 6.7 Hz), 2.42–2.60 (m, 1 H), 3.78 (d, 1 H, $J$ = 7.8 Hz), 4.53 (d, 1 H, $J$ = 7.6 Hz), 6.63–6.68 (m, 2 H), 6.78 (d, 1 H, $J$ = 7.5 Hz), 7.15–7.48 (m, 7 H), 7.58–7.67 (m, 1 H), 7.72–7.76 (m, 2 H), 7.93 (d, 1 H, $J$ = 7.6 Hz).

$^{13}$C NMR (50 MHz, CDCl$_3$): $^a$= 18.6 (q), 45.9 (d), 58.5 (d), 75.9 (d), 125.7 (d), 126.5 (d), 127.1 (d), 127.9 (t), 128.5 (t), 129.2 (d), 129.8 (d), 130.2 (t), 134.0 (d), 135.0 (s), 136.8 (s), 144.0 (s), 147.8 (s).

HRMS (CI): $m/z$ Calcd for C$_{22}$H$_{20}$O$_2$S$^+$ (M + NH$_4^+$): 366.1528. Found: 366.1523.

Reaction of Ylide $^a$ with Chalcone 1c

A suspension of ylide $^a$ (500 mg, 1.0 mmol) and trans-benzylideneacetophenone (580 mg, 2.78 mmol) in MeCN (10 mL) was stirred for 62 h according to the above general procedure. The solvent was evaporated (20 °C/10 Torr) and the residue was chromatographed on silica gel [eluent: CH$_2$Cl$_2$–petroleum ether, 1:1) to yield 220 mg (52%) of the [3-phenyl-(1-phenylsulfonyl)indan-2-yl]phenylmethanone (trans-2c) at an olefin conversion of 24%; colorless needles; mp 150–151 °C.

IR (KBr): 3070, 3030, 2940, 2920, 1675, 1665, 1595, 1575, 1495, 1480, 1445, 1360, 1340, 1305, 1245, 1240, 1205, 1165, 1145, 1085, 1040, 1000, 980, 965, 885, 870 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $^a$= 4.22 (d, 1 H, $J$ = 7.8 Hz), 4.56 (t, 1 H, $J$ = 7.5 Hz), 5.76 (d, 1 H, $J$ = 7.1 Hz), 6.83 (d, 1 H, $J$ = 7.6 Hz), 6.85–6.88 (m, 2 H), 7.17–7.25 (m, 5 H), 7.28–7.38 (m, 6 H), 7.42–7.48 (m, 2 H), 7.77–7.80 (m, 2 H), 7.82 (d, 1 H, $J$ = 7.7 Hz).

$^{13}$C NMR (100 MHz, CDCl$_3$): $^a$= 56.4 (d), 57.1 (d), 71.5 (d), 125.5 (d), 126.1 (d), 127.5 (d), 128.1 (d), 128.2 (t), 128.6 (t), 128.7 (t), 128.8 (t), 129.0 (t), 129.1 (t), 129.8 (d), 133.4 (d), 133.8 (d), 133.9 (s), 135.5 (s), 137.3 (s), 142.4 (s), 146.1 (s), 198.4 (s).

Anal. Calcd for C$_{28}$H$_{22}$O$_2$S$^+$ (M + NH$_4^+$): 438.15. Found: C, 76.69; H, 5.06; S, 7.31.


Scheme 4
Reaction of Ylide $\gamma$ with trans-1-Phenyl-1-propene (1b)

A suspension of the ylide (450 mg, 1.0 mmol) and trans-1-phenyl-1-propene (328 mg, 2.78 mmol) in MeCN (10 mL) was stirred for 24 h according to the above general procedure. The solvent was evaporated (20 °C/10 Torr) and the residue was chromatographed on silica gel (eluent: CH$_2$Cl$_2$–petroleum ether, 1:1) or yield 56 mg (27%) of the 2,5-dimethyl-3-(methylsulfonyl)-1-phenylindane (trans-2a). Synthesis 2002, No. 14, 2084–2090 ISSN 0039-7881 © Thieme Stuttgart · New York

Crystallographic Data for trans-2a

Crystals of trans-2a suitable for X-ray analysis were obtained by recrystallization from CH$_2$Cl$_2$.

C$_{18}$H$_{20}$O$_2$S (438), colorless; triclinic; space group P-1; \(a = 931.01(6)\), \(b = 980.04(7)\), \(c = 1368.83(9)\) pm; \(\alpha = 92.5360(10)^{\circ}\), \(\beta = 107.8960(10)^{\circ}\), \(\gamma = 105.8540(10)^{\circ}\); \(V = 1.13205(13)\) nm$^3$, \(Z = 2\); \(D_x = 1.287\) g cm$^{-3}$. The data were collected from shock-cooled crystals on a BRUKER SMART-APEX diffractometer with D8 goniometer (graphite-monochromated Mo-K$_\alpha$ radiation, \(\lambda = 0.7073\) Å), equipped with a low-temperature device in omega mode at 193(2) K.$^1$ A total of 15943 reflections were collected, of which 5082 were unique [\(R(int) = 0.0224\)]. Data were collected to a maximum 2$\theta$ of 56.38$\circ$.

The data was integrated with SAINT$^2$ and an empirical absorption correction was applied.$^3$ The structures were solved by direct methods (SHEXLXL-97)$^4$ and refined by full-matrix, least-squares methods against $\sum F^2$ (SHEXLXL-97).$^5$ All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed at idealized positions except those of water. The final difference Fourier had a height of 0.534 e Å$^{-3}$ and the minimum negative peak had a height of −0.363 e Å$^{-3}$. Crystallographic drawings were done using XSEED in SHEXLXL-97. Crystallographic data for the structure reported in this
paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 186374. Copies of the data may be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44(1223)336033; e-mail: deposit@ccdc.cam.ac.uk].

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