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REACTION OF 2-BENZOTELLUROPYRYLIUM SALTS WITH ORGANOCOPPER REAGENTS: INTRODUCTION OF A CARBON FUNCTIONAL GROUP AT THE C-1 POSITION OF THE TELLUROPYRYLIUM CATION RING [1]

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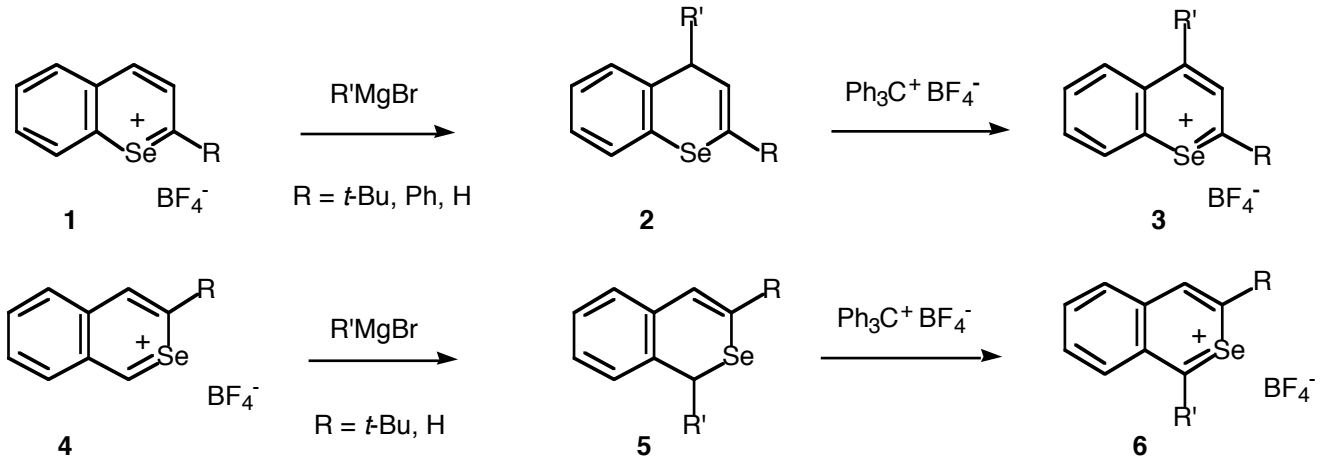
Abstract- 3-*tert*-Butyl-2-benzotelluropyrylium salt (**13A**) react with lithium dialkyl(phenyl)copper to give in good yield the corresponding isotellurochromenes (**15A**) having a carbon functional group at the C-1 position. Similarly, the 1-substituted isoselenochromenes (**15B**) and the 4-substituted tellurochromene (**19**) were also prepared from the corresponding pyrylium salts (**13B, 18**). The obtained isotellurochromenes (**15A**) were easily converted into the corresponding 2,4-disubstituted 2-benzotelluropyrylium salts (**20**) by the treatment with triphenylcarbenium tetrafluoroborate (Ph_3C^+ BF_4^-).

INTRODUCTION

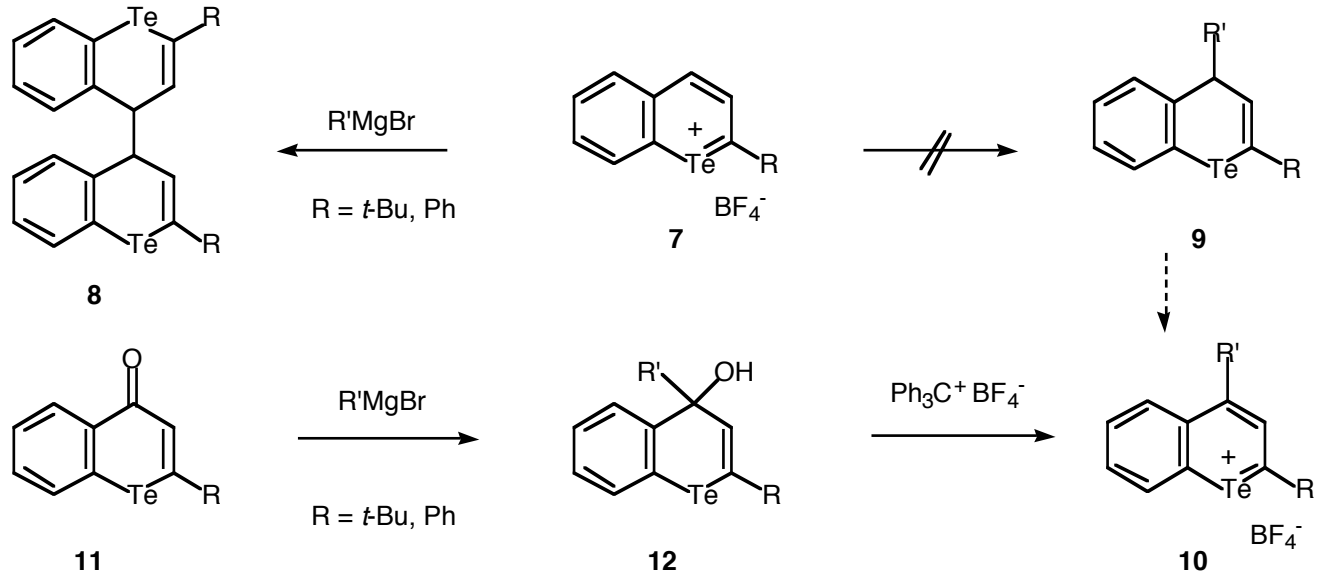
The chemistry of the telluropyrylium compounds, six-membered aromatic heterocycles containing a tellurium cation, has been covered in recent reviews.[2] Not a small number of monocyclic[3] telluropyrylium salts and 1-benzo derivatives[4] have been prepared, and their structures, physical properties and reactions are studied with considerable interest. Furthermore, the dibenzo[*b*]telluropyrylium salts[5] have also been synthesized. Among them, we have succeeded in the general and practical preparation of the novel 2-benzotelluropyrylium compounds[6] in very recent years, and reported the reactions with several nucleophiles including the alkoxide anion (OMe^-, O*i*-Pro^-, O*t*-Bu^-), primary (*n*-butylamine) and secondary

amine (diethylamine), active methyl compound (acetone) and also reducing agents ($LiAlH_4$, DIBALH, Zn dust, Pd-C).[7]

The reactions of the 1- and 2-benzochalcogenopyrylium salts with Grignard reagents have also been examined. The reaction of the 1-benzoselenopyrylium salts (**1**)[8] with Grignard reagents resulted in carbon-carbon bond formation at the C-2 or C-4 position to give the 2- or 2,4-(di)substituted 4*H*-selenochromenes (**2**)[9] in good yields. The 2-benzoselenopyrylium salts (**4**)[6b] also reacted with Grignard reagents to afford the 1-alkyl(phenyl)isoselenochromenes (**5**)[7, 10] as the sole products. The obtained selenochromenes (**2**) and isoselenochromenes (**5**) could be easily transformed in high yields into the corresponding pyrylium salts (**3**, **6**)[7,9] having two carbon functional groups on the hetero cation rings by treatment with triphenylcarbenium tetrafluoroborate ($Ph_3C^+ BF_4^-$). In this way, the introduction of an alkyl or phenyl group into the selenopyrylium nuclei could be achieved using a Grignard reagent as shown in Scheme 1.



Scheme 1



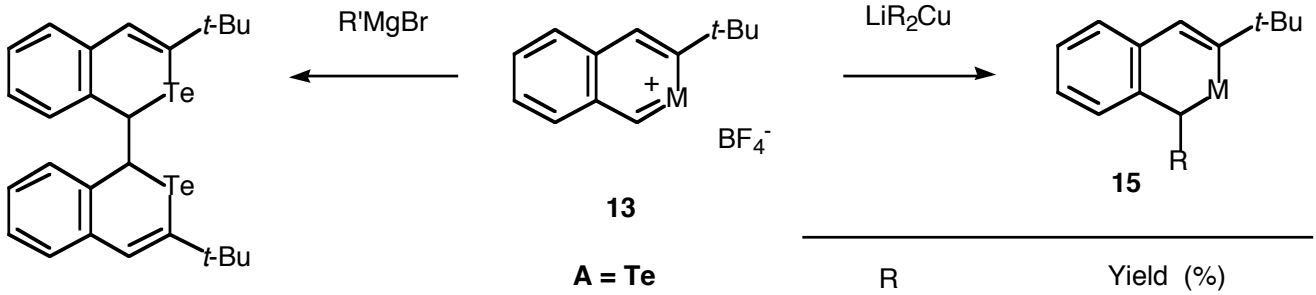
Scheme 2

In contrast, the 2-substituted 1-benzotelluropyrylium salts (**7**), the telluro analogue of **1**, reacted with Grignard reagents to give a complex mixture in a small quantity of the dimeric-type product (**8**).[11] No desire 4*H*-tellurochromenes (**9**), the precursors of the 2,4-disubstituted 1-benzotelluropyrylium salts (**10**), were obtained. However, the pyrylium salts (**10**)[12] were prepared from the 4-hydroxy-4*H*-tellurochromenes (**12**), which were produced by the reaction of the 2-substituted tellurochromen-4-ones (**11**)[13] with a Grignard reagent in moderate to good yields (Scheme 2).

No 2-benzotelluropyrylium salts having two carbon functional groups at the C-1 and C-3, a theoretically possible structural isomer of **10**, have been prepared until now, and their general successful preparation is still of interest. In this paper, we describe the reaction of the 2-benzotelluropyrylium and 2-benzoselenopyrylium salts with organocopper reagents, and the conversion of the obtained 1-substituted isotellurochromenes into the 2-benzotelluropyrylium salts having two carbon functional groups at the C-1 and C-3 positions on the heterocation ring.

RESULTS AND DISCUSSION

The treatment of the 2-benzotelluropyrylium salt (**13A**) with Grignard reagents such as methyl-, ethyl- and phenylmagnesium bromide (iodide), except for benzylmagnesium bromide, resulted in the decomposition of the starting material to afford a complex mixture including a small quantity of the dimeric-type product (**14**)[6a,7] without the formation of the normal coupling products. However, we found that the reaction of the pyrylium salts (**13**) with LiR_2Cu, which was reported by Corey and Posner,[14] formed the carbon-carbon bond at the C-1 position to give the desire 1-substituted isotellurochromenes (**15**). The 3-*tert*-butyl-2-benzotelluropyrylium salt (**13A**) reacted with 5 equivalents of $LiMe_2Cu$, which



14 **13** **15**

A = Te
B = Se

R	Yield (%)	
	Te	Se
a: Me	87	88
b: Et	78	73
c: *n*-Bu	56	58
d: *n*-Hex	67	56
e: Ph	64	66

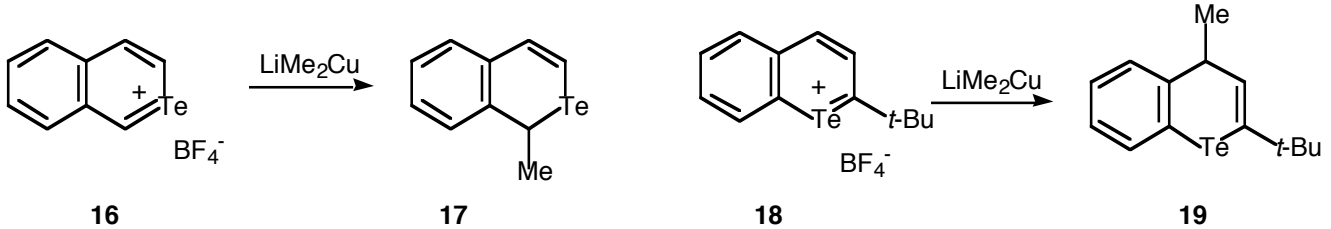
Scheme 3

was easily generated in situ from 2 mol of MeLi and 1 mol of CuI in Et_2O to give the 1-methylisotellurochromene (**15Aa**) at 0°C in 87 % yield as the sole product. Similarly, 1-ethyl (**15Ab**), 1-butyl (**15Ac**), 1-hexyl (**15Ad**) and 1-phenyl derivatives (**15Ae**) were also produced by the reaction of **13A** with the corresponding organocopper reagents at -20 to -50 °C in moderate to good yields. All the 1-alkyl(phenyl)isotellurochromenes (**15A**) are previously unknown compounds.

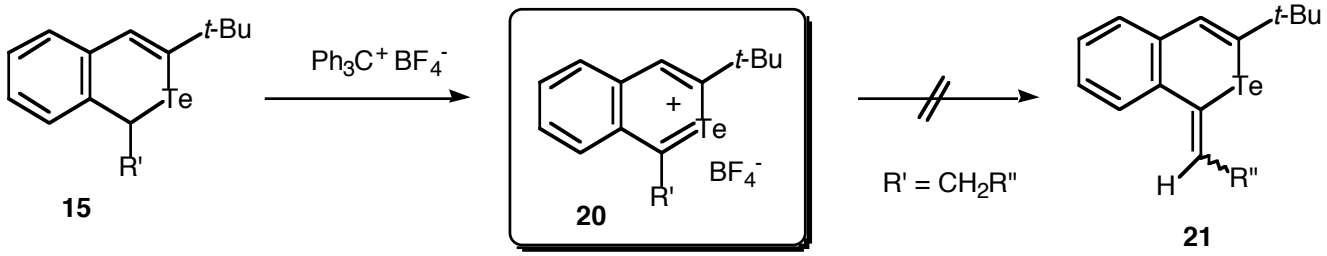
The difference in the reactivities of the telluropyrylium salt (**13A**) with lithium dialkyl(phenyl)copper and Grignard reagents could be explained by the following theories. The nucleophilicity of LiR_2Cu is higher than that of the Grignard reagents. LiR_2Cu is bulky and a soft alkylating agent. Thus, LiR_2Cu favors to react with the 2-benzotelluropyrylium salt (**13A**) to give the anticipated 1-alkyl(phenyl)-isotellurochromenes (**15A**); the tellurium cation is a soft acid. On the other hand, Grignard reagents react with the substrates through a radical mechanism involving a single electron transfer. This agrees very closely with the formation of the dimeric-type product (**14**).

Next, we examined the reaction of the 2-benzoselenopyrylium salt (**13B**) with the same copper reagents, although the 1-substituted isoselenochromenes (**5**)[7] were already obtained by the reaction of their salts (**4**) with Grignard reagents (Scheme 1). The salt (**13B**) were also treated with $LiMe_2Cu$, $LiEt_2Cu$, Li(*n*-Bu)$_2$Cu, Li(*n*-Hex)$_2$Cu and $LiPh_2Cu$ under the similar conditions to give 1-methyl- (**15Ba**), 1-ethyl- (**15Bb**), 1-butyl- (**15Bc**), 1-hexyl- (**15Bd**), and 1-phenyl-3-*tert*-butylisoselenochromenes (**15Be**) in moderate yields, respectively, as shown in Scheme 3.

Neither the 1-*sec*-butyl nor 1-*tert*-butyl substituted isochalcogenochromenes were obtained by the reaction of the salts (**13**) with the assumed lithiumcopper reagents, which were generated from the corresponding butyl lithium and CuI. In this case, the starting materials were decomposed, yielding a small amount of isochalcogenochromene having no substituent at the C-1 position. The reaction of the unsubstituted parent 2-benzoteluropyrylium salt (**16**) with $LiMe_2Cu$ under the conditions resulted in decomposition to give 1-methylisotellurochromene (**17**) in only 7 % yield due to the instabilities of the starting material and the product. Furthermore, the treatment of the 2-*tert*-butyl-1-benzotelluropyrylium salt (**18**), the regio isomer of the 2-benzotelluropyrylium salt (**13A**), gave the desire 4-methyl-4*H*-tellurochromene (**19**) in 20 % yield with a small amount of unknown compounds.



Scheme 4



Scheme 5

The transformation into the 1-substituted 3-*tert*-butyl-2-benzotelluropyrylium salts (**20**) was carried out using the corresponding obtained isotellurochromenes (**15A**). **15A** were treated with Ph_3C^+ BF_4^- in $MeNO_2$ at room temperature to give the desire stable 2-benzotelluropyrylium tetrafluoroborates (**20**) as yellow prisms in good yields. BF_4^-, the counter anion of the 1-benzotelluropyrylium salts having a primary alkyl group at the C-2 position, has been found to abstract the β–hydrogen of the methylene carbon of the alkyl group forming the unstable *exo*-methylene compounds.[11,12] Furthermore, we have also determined that the 1-benzyl-2-benzotelluropyrylium salts[6a,7] could be isolated while present in a solvent containing an equilibrium mixture of the salts and the 1-benzylideneisotellurochromenes, which are β–hydrogen eliminated compounds of the pyrylium salts. Thus, we suspect that the β–hydrogen elimination of the 1-alkyl-2-benzotelluropyrylium salts (**20a-d**) proceeds to form the 1-methylideneisotellurochromenes (**21**). However, the formation of the 1-*exo*-methylene compounds (**21**) could not be observed in the ^{1}H NMR spectrum. The salts (**20**) can be kept for a few weeks in refrigerator under argon, however unstable in solution and gradually decomposed to give any characterized products.

In conclusion, the present result provides a new practical method for introducing a carbon functional group to the C-1 position on the 2-benzopyrylium cation rings. Thus, general preparations for all four isomers of the benzopyrylium compounds (1-benzoseleno-, 1-benzotelluro-, 2-benzoseleno- and 2-benzotelluro-) having two carbon functional groups at the C-1 and C-3, or C-2 and C-4 position have been achieved.

EXPERIMENTAL

Melting points were measured on a Yanagimoto micro melting point hot stage apparatus and are uncorrected. IR spectra were recorded on a Horiba FT-720 spectrophotometer. MS and HRMS were recorded on a JEOL JMS-DX300 instrument. ^{1}H NMR spectra were recorded on a PMX-60SI (60 MHz) or JEOL EX-90A (90 MHz) spectrometer in $CDCl_3$ or CD_3CN using TMS as internal standard and *J* values are given in Hz. Microanalyses were performed in the Microanalytical Laboratory of this Faculty.

Reaction of 2-Benzotelluropyrylium Salt (13A) with Organocopper Reagents

3-*tert*-Butyl-1-methyl-1*H*-isotellurochromene (15Aa)

$LiMe_2Cu$ (5.0 mmol) in ether solution (20 mL), which was prepared from 2 equiv. of MeLi and 1 equiv. of CuI, was slowly added to a suspended mixture of the pyrylium salt (**13A**: 388 mg, 1 mmol) in ether (20 mL) at 0° C under an argon atmosphere. The mixture was stirred under the conditions for 30 min, and quenched by the addition of saturated aqueous NH_4Cl solution (10 mL), and then extracted with Et_2O (30 mL x 3). The organic layers were washed with brine (30 mL x 2) and dried ($MgSO_4$) and evaporated *in vacuo*. The resulting residue was chromatographed on silica gel eluted with *n*-hexane - CH_2Cl_2 (20:1) to give **15Aa** (87 % yield), orange oil; ^{1}H NMR (90 MHz, $CDCl_3$) 1.27 (9H, s, *t*-Bu), 1.86 (3H, d, *J* = 7 Hz, 1-Me), 4.03 (1H, q, *J* = 7 Hz, 1-H), 6.67 (1H, s, 4-H), 7.1-7.3, (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{14}H_{18}Te$: 316.0471. Found: 316.0478.

3-*tert*-Butyl-1-ethyl-1*H*-isotellurochromene (15Ab)

The pyrylium salt (**13A**) was treated with $LiEt_2Cu$, which was similarly prepared from EtLi and CuI, instead of $LiMe_2Cu$ and worked up as described for the preparation of **15Aa** to give **15Ab** (78 % yield), yellow oil; ^{1}H NMR (90 MHz, $CDCl_3$) 0.86 and 1.89 (3H, t, *J* = 7 Hz, 2H, dq, *J* = 7 and 8 Hz, Et), 1.27 (9H, s, *t*-Bu), 3.68 (1H, t, *J* = 8 Hz, 1-H), 6.65 (1H, s, 4-H), 7.1-7.3 (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{15}H_{20}Te$: 330.0628. Found: 330.0619.

1-Butyl-3-*tert*-butyl-1*H*-isotellurochromene (15Ac)

The pyrylium salt (**13A**) was treated with Li*n*-Bu_2Cu, which was similarly prepared from *n*-BuLi and CuI, instead of $LiMe_2Cu$ and worked up as described for the preparation of **15Aa** to give **15Ac** (56 % yield), orange oil; ^{1}H NMR (90 MHz, $CDCl_3$) 0.91, 1.0-1.5 and 1.7-2.0 (3H, t, *J* = 6 Hz, 4H, m, 2H, m, *n*-Bu), 1.27 (9H, s, *t*-Bu), 3.77 (1H, t, *J* = 7 Hz, 1-H), 6.65 (1H, s, 4-H), 7.1-7.3 (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{17}H_{24}Te$: 358.0941. Found: 358.0935.

3-*tert*-Butyl-1-hexyl-1*H*-isotellurochromene (15Ad)

The pyrylium salt (**13A**) was treated with Li*n*-Hex_2Cu, which was similarly prepared from *n*-HexLi and CuI, instead of $LiMe_2Cu$ and worked up as described for the preparation of **15Aa** to give **15Ad** (67 % yield), yellow oil; ^{1}H NMR (90 MHz, $CDCl_3$) 0.85, 1.0-1.5 and 1.8-2.0 (3H, t, *J* = 6 Hz, 8H, m, 2H, m, *n*-Hex), 1.27 (9H, s, *t*-Bu), 3.77 (1H, t, *J* = 8 Hz, 1-H), 6.65 (1H, s, 4-H), 7.1-7.3 (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{19}H_{28}Te$: 386.1254. Found: 386.1261.

3-*tert*-Butyl-1-phenyl-1*H*-isotellurochromene (15Ae)

The pyrylium salt (**13A**) was treated with $LiPh_2Cu$, which was similarly prepared from PhLi and CuI, instead of $LiMe_2Cu$ and worked up as described for the preparation of **15Aa** to give **15Ac** (64 % yield), yellow prisms, mp 92-94 °C (from acetone – *n*-hexane); ^{1}H NMR (90 MHz, $CDCl_3$) 1.17 (9H, s, *t*-Bu), 5.40 (1H, s, 1-H), 6.77 (1H, s, 4-H), 7.1-7.4 (9H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{19}H_{20}Te$: 378.0628.

Found: 378.0616.

Reaction of 2-Benzoselenopyrylium Salt (13B) with Organocopper Reagents

The pyrylium salt (**13B**: 338 mg, 1 mmol) was treated with $LiMe_2Cu$, $Li(n\text{-}Bu)_2Cu$, $Li(n\text{-}Hex)_2Cu$ or $LiPh_2Cu$, and worked up as described for the preparation of **15Aa** to give **15B**. Compounds (**15Ba-c**, **15Be**) were identical with the authentic samples prepared in our previous paper.[7]

3-*tert*-Butyl-1-hexyl-1*H*-isoselenochromene (15Bd)

The pyrylium salt (**13B**) was treated with $Li(n\text{-}Hex)_2Cu$, and worked up as described for the preparation of **15Aa** to give **15Bd** (56 % yield), pale yellow oil, ^{1}H NMR (90 MHz, $CDCl_3$) 0.85, 1.1-1.5 and 1.6-1.9 (3H, t, J = 6 Hz, 8H, m, 2H, m, *n*-Hex), 1.27 (9H, s, *t*-Bu), 3.76 (1H, t, J = 8Hz, 1-H), 6.70 (1H, s, 4-H), 7.1-7.3 (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{19}H_{28}Se$: 336.1356. Found: 336.1357.

1-Methyl-1*H*-isotellurochromene (17)

The pyrylium salt (**16**) was treated with $LiMe_2Cu$, and worked up as described for the preparation of **15Aa** to give **17** (7 % yield), pale yellow oil, ^{1}H NMR (90 MHz, $CDCl_3$) 1.91 (3H, d, J = 7 Hz, 1-Me), 4.00 (1H, q, J = 7 Hz, 1-H), 6.70 (1H, d, J = 10 Hz, 4-H), 7.1-7.4 (5H, m, 3-H and Ph-H). HRMS *m/z* M^+ Calcd for $C_{10}H_{10}Te$: 259.9845. Found: 259.9851.

2-*tert*-Butyl-4-methyl-4*H*-tellurochromene (19)

The pyrylium salt (**18**) was treated with $LiMe_2Cu$, and worked up as described for the preparation of **15Aa** to give **19** (20 % yield), pale yellow oil, ^{1}H NMR (90 MHz, $CDCl_3$) 1.17 (9H, s, *t*-Bu), 1.65 (3H, d, J = 7 Hz, 1-Me), 2.6-2.9 (1H, m, 4-H), 5.84 (1H, d, J = 5 Hz, 3-H), 6.9-7.4 (4H, m, Ph-H). HRMS *m/z* M^+ Calcd for $C_{14}H_{18}Te$: 316.0471. Found: 316.0485.

Preparation of 1-Substituted 2-Benzotelluropyrylium Tetrafluoroborate (20)

Ph_3C^+ BF_4^- (363 mg, 1.1 mmol) was added to a stirred solution of the isotellurochromene (**15A**, 1 mmol) in dry $MeNO_2$ (2.0 mL) and the mixture was stirred at rt for 1-2 h. To the reaction mixture was added dry ether (*ca*. 20 mL) to precipitate the telluropyrylium salt (**20**). The salt (**20**) was obtained in a nearly pure state, and recrystallized from $CHCl_3$.

3-*tert*-Butyl-1-methyl-2-benzotelluropyrylium Tetrafluoroborate (20a)

76 % yield, yellow prisms, mp 135-137 °C (decomp); IR (KBr)/cm^{-1} 1052 (BF_4^-); ^{1}H NMR (90 MHz, CD_3CN) 1.65 (9H, s, *t*-Bu), 3.40 (3H, s, 1-Me), 8.3-8.7 (4H, m, Ph-H), 8.99 (1H, s, 4-H). Anal. Calcd for $C_{14}H_{17}BF_4Te$: C, 42.07; H, 4.29. Found: C, 42.19; H, 4.35.

3-*tert*-Butyl-1-ethyl- 2-benzotelluropyrylium Tetrafluoroborate (20b)

59 % yield, yellow prisms, mp 144-146 °C (decomp); IR (KBr)/cm^{-1} 1084 (BF_4^-); ^1H NMR (90 MHz, CD_3CN) 1.66 (9H, s, *t*-Bu), 1.73 and 3.91 (3H, t, *J* = 7 Hz and 2H, q, *J* = 7 Hz, 1-Et), 8.3-8.8 (4H, m, Ph-H), 8.96 (1H, s, 4-H). Anal. Calcd for $C_{15}H_{19}BF_4Te$: C, 43.55; H, 4.63. Found: C, 43.50; H, 4.66.

1-Butyl-3-*tert*-butyl-2-benzotelluropyrylium Tetrafluoroborate (20c)

66 % yield, yellow prisms, mp 123-125 °C (decomp); IR (KBr)/cm^{-1} 1062 (BF_4^-); ^1H NMR (90 MHz, CD_3CN) 1.03, 1.9-2.2 and 3.95 (3H, t, *J* = 7 Hz, 4H, m and 2H, t, *J* = 8 Hz, *n*-Bu), 1.66 (9H, s, *t*-Bu), 8.3-8.6 (4H, m, Ph-H), 8.96 (1H, s, 4-H). Anal. Calcd for $C_{17}H_{23}BF_4Te$: C, 46.22; H, 5.25. Found: C, 45.99; H, 5.06.

3-*tert*-Butyl-1-hexyl-2-benzotelluropyrylium Tetrafluoroborate (20d)

57 % yield, yellow prisms, mp 174-177 °C (decomp); IR (KBr)/cm^{-1} 1055 (BF_4^-); ^1H NMR (90 MHz, CD_3CN) 0.68, 0.8-1.8 and 3.96 (3H, t, *J* = 8 Hz, 8H, m and 2H, br t, *J* = 8 Hz, *n*-Hex), 1.68 (9H, s, *t*-Bu), 8.2-8.7 (4H, m, Ph-H), 8.97 (1H, s, 4-H). Anal. Calcd for $C_{19}H_{27}BF_4Te$: C, 48.57; H, 5.79. Found: C, 48.79; H, 5.66.

3-*tert*-Butyl-1-phenyl-2-benzotelluropyrylium Tetrafluoroborate (20e)

66 % yield, yellow prisms, mp 114-116 °C (decomp); IR (KBr)/cm^{-1} 1062 (BF_4^-); ^1H NMR (90 MHz, CD_3CN) 1.68 (9H, s, *t*-Bu), 8.2-8.5 (9H, m, Ph-H), 9.14 (1H, s, 4-H). Anal. Calcd for $C_{19}H_{19}BF_4Te$: C, 49.42; H, 4.15. Found: C, 49.31; H, 4.36.

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