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TELLURAXANTHENE

I. D. Sadekov, A. A. Ladatko,
and V. I. Minkin

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Telluraxanthene was obtained by intramolecular electrophilic cyclization of 2-trichlorotelluriodiphenylmethane with subsequent reduction of the intermediately formed 10,10-dichlorotelluraxanthene. The chemical reactions that occur at the tellurium atom and the methylene group of telluraxanthene were studied. The lower limit of the activation energy of pyramidal inversion at the onium tellurium atom in telluroniaxanthenyldimedone ylid was determined by PMR spectroscopy.

In contrast to xanthene and its thia and selena analogs, telluraxanthene and its derivatives were unknown until recently. An attempt by Renson [1] to obtain telluraxanthene by cyclization of 2-chlorocarbonyldiphenyl telluride led to rearrangement of the starting compound and the formation of 2-chloroelluriabenzophenone. For the synthesis of telluraxanthene we recently found [2] a new (for the chemistry of xanthenes) reaction, viz., intramolecular electrophilic cyclization of 2-trichlorotelluriodiphenylmethane (I) with subsequent reduction of the resulting 10,10-dichlorotelluraxanthene (which is formed in high yield) to the desired compound. This paper is devoted to a description of the synthesis of telluraxanthene and a study of its properties.

The starting Ia for the preparation of telluraxanthene was obtained from bis(2-benzylphenyl) ditelluride (II), which was synthesized in 60-80% yields by the reaction of 2-lithiodiphenylmethane [from 2-iodo(bromo)diphenylmethane and lithium in ether] with powdered tellurium in an argon atmosphere with subsequent spontaneous oxidation by air oxygen of the corresponding tellurophenol formed by hydrolysis with dilute hydrochloric acid. The higher yields of ditelluride II when 2-bromodiphenylmethane is used are due to the fact that when 2-iododiphenylmethane is used to obtain 2-lithiodiphenylmethane, part of the resulting organolithium derivative undergoes side reactions involving condensation with the halo derivative. The chlorination and bromination of ditelluride II to the corresponding trihalides Ia, b give the products in quantitative yields. An alternative method for the preparation of Ia, viz., reaction between 2-trimethylsilyldiphenylmethane and tellurium tetrachloride in toluene (by the method in [3]) leads to lower yields (~50%) of Ia based on the starting 2-halodiphenylmethane.

The cyclization of 2-trichlorotelluriodiphenylmethane (Ia) to 10,10-dichlorotelluraxanthene (IIIa) does not proceed as readily as the cyclization of 2-chlorotelluriodiphenyl sulfide [4] and 2-trichlorotelluriodiphenyl oxide [5]. The latter reactions, although they do take place under rather severe conditions (heating for many hours at 200-250°C), do not require the presence of catalysts because of the sufficiently strong activation of the ortho positions of the aryl rings to electrophilic attack by the electron-donor oxygen and sulfur atoms. However, Ia undergoes cyclization when it is heated in o-dichlorobenzene or 1,2,4-trichlorobenzene in the presence of one equivalent of anhydrous aluminum chloride. Attempts to bring about the cyclization of 2-trichlorotelluriodiphenylmethane at elevated temperatures in the absence of anhydrous aluminum chloride led to the production of IIIa in a very low and difficult-to-reproduce yield. The role of aluminum chloride evidently consists in a significant increase in the manifestation of the electrophilic properties of the trichlorotellurio group in Ia due to the formation of a complex, to which, in analogy with the product of the reaction

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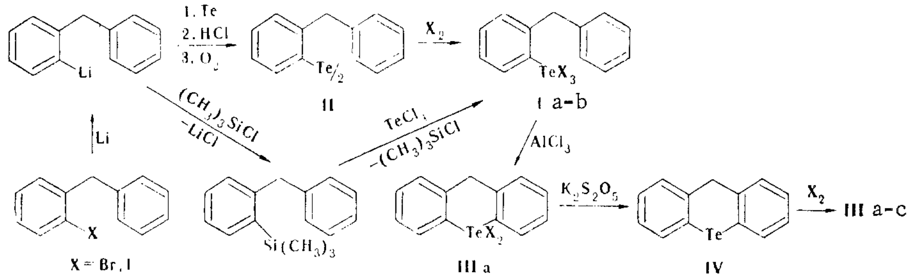
of tellurium tetrachloride and aluminum chloride, viz., $[TeCl_3]^+[AlCl_4]^-$ [6], the $[RTeCl_2]^+$-$[AlCl_4]^-$ structure, where R is a dephenylmethane residue, can be assigned. It is interesting to note that 2-tribromotelluriodiphenylmethane (Ib), which is obtained in virtually quantitative yield by oxidation of ditelluride II with bromine, does not give 10,10-dibromotelluraxanthene (IIIb) under similar conditions, probably because of the fact that the electrophilic properties of the trihalotellurio groups are diminished markedly on passing from chlorine to bromine [7]. Like noncyclic tellurides, the 10,10-dichlorotelluraxanthene (IV) obtained by cyclization of Ia readily undergoes reduction to telluraxanthene in almost quantitative yield.

The signals of the methylene protons in the PMR spectra of I-IV show up in the form of triplets at room temperature and are shifted to weak field on passing from divalent tellurium derivatives (II and IV) to tetracoordinated tellurium derivatives (Ia, b and IIIa; see the experimental section).



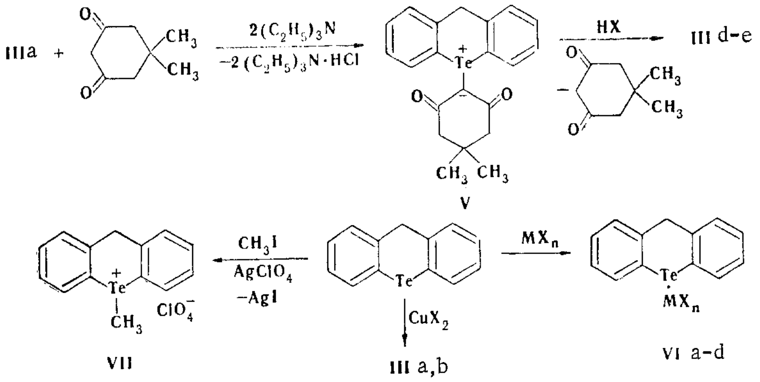
I, IIIa X = Cl; b X = Br; c X = I

For telluraxanthene one should have expected the manifestation of chemical properties that are, on the one hand, typical for xanthenes in general and are due to the presence of a methylene group in their composition; on the other hand, its chemical properties should have been to a certain extent similar to the chemical properties of its noncyclic analogs, viz., diaryltellurides. In fact, telluraxanthene is characterized by reactions that are usually observed for diaryl tellurides [8]: It readily adds halogens (chlorine, bromine, and iodine) to give the corresponding 10,10-dihalotelluraxanthenes (IIIa-c). On the basis of these telluraxanthene derivatives we were able to synthesize telluroniaxanthenyldimedone ylid (V) by reaction of 10,10-dichlorotelluraxanthene with dimedone in the presence of two equivalents of triethylamine. As a consequence of the fact that the direct synthesis of 10,10-difluorotellurananthene (IIId) is difficult, this compound, like 10,10-bis(trifluoroacetyl)telluraxanthene (IIIe), was obtained by an indirect method, viz., by the method that we previously developed for the preparation of various noncyclic σ-tellurans [9]. Treatment of telluroniaxanthenyldimedone ylid (V) with hydrofluoric and trifluoroacetic acids leads to the preparation of IIId, e.

Like its noncyclic analogs, telluraxanthene readily forms 1:1 molecular complexes with mercury (VIa, b) and silver (VIc, d) salts. However, whereas the complexes with mercury salts are so stable that they can even be recrystallized from toluene without decomposition, the complexes of telluraxanthene with silver salts (the perchlorate and nitrate) were not obtained in analytically pure form because of their rapid decomposition during storage. The behavior of the halides of divalent copper differs from that of the mercury and silver salts. The corresponding 10,10-dihalotelluraxanthenes (IIIa, b) are obtained when telluraxanthese is refluxed under relatively mild conditions (in solution in aqueous acetone).

In contrast to dialkyl and aryl alkyl tellurides, which have increased basicities [8] because of the presence of alkyl substituents attached to the divalent tellurium atom, telluraxanthene does not form telluronium salts when it is refluxed with methyl iodide. However, 9,9-dihydro-10-methyltelluraxanthenylium perchlorate (VII) is formed readily in high yield by the action of methyl iodide on telluraxanthene in the presence of an equimolar amount of silver perchlorate.

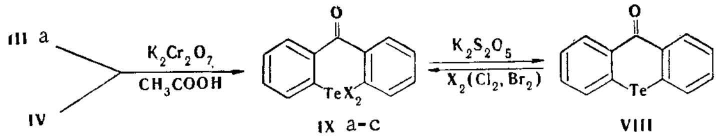
In conformity with the trigonal-pyramidal orientation of the bonds attached to the onium tellurium atom, the signals of the methylene protons of the telluraxanthenyl ring in the PMR spectrum of the telluronium ylid show up in the form of an AB quartet ($\Delta\nu = 44$ Hz, $J = 16$ Hz) up to 180°C (in benzonitrile). The sample begins to decompose above this temperature. Using the relationship proposed for the calculation of the exchange rate constants for the process $AB \rightarrow A_2$ [10], one can determine that $K_{180} \ll 130\ sec^{-1}$ and that the free energy of activation



III d X=F; e X=CF$_3$COO; VI a M=Hg, X=Cl; b M=Hg, X=Br; в M=Ag, X=ClO$_4$; d M=Ag, X=NO$_3$

of the inversion of the tricoordinated tellurium in the ylid $\Delta G^{\neq} > 23$ kcal/mole (96 kJ/mole). One should also note the increased thermal stability of this ylid as compared with diaryltelluroniadimedone ylids, which decompose even upon brief refluxing in o-xylene [11]. In the case of telluronium salt VII the signals of the methylene protons of the telluraxanthenyl fragment show up in the form of a very highly coupled AB quartet, the wing of which is very difficult to separate from the noise because of the low solubility of the salt in the available solvents. The anisochronicity of the methylene protons is retained unchanged at 180°C, and this constitutes evidence for the stability of the pyramid at the tellurium atom. This sort of behavior of the telluronium salt is in agreement with the fact that in the case of the similarly constructed sulfur compound, viz., 9,9-dimethyl-10-phenylthiaxanthenylium perchlorate, the free energy of activation of pyramidal inversion at the sulfur atom $\geqslant 25.4$ kcal/mole (coalescence of the gem-dimethyl groups is observed when the compound is heated in benzophenone at 200°C [12]), while in conformity with the quantum-chemical calculations that we made for various tricoordinated chalcogenonium derivatives (oxides, ylids, and imides [13]), the barriers to pyramidal inversion should increase in the order S < Se < Te.

We also studied some reactions of telluraxanthene that take place at the methylene group. The most important reaction of this type is oxidation, which leads to the production of telluraxanthone, the starting compound for the synthesis of various telluraxanthene derivatives. Telluraxanthone (VIII) was obtained by two independent methods from 10,10-dichlorotelluraxanthene (IIIa) and telluraxanthene (IV). Telluraxanthone (VIII) is obtained in high yield by oxidation of IIIa with potassium dichromate in glacial acetic acid with subsequent reduction without additional purification of the intermediately formed 10,10-dichlorotelluraxanthone (IXa) with potassium metabisulfite. However, oxidation of telluraxanthene itself under identical conditions leads to the production of 10,10-diacetyltelluraxanthone (IXb). The formation of this compound probably proceeds through the initial oxidation of telluraxanthene to the corresponding tellurium oxide, which, like diaryltellurium oxides, undergoes reaction with the excess glacial acetic acid present in the reaction mixture to give tellurium diacetate [14, 15], from which IXb, the reduction of which gives telluraxanthone in high yield, is obtained by subsequent oxidation of the methylene group.

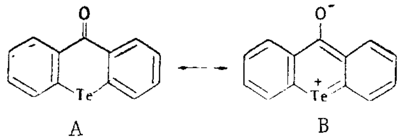


a X=Cl; b X=CH$_3$COO; c X=Br

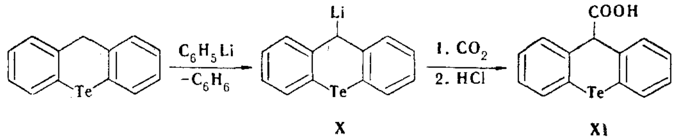
Like telluraxanthene, telluraxanthone readily adds halogens (chlorine and bromine) to give, respectively, 10,10-dichloro- and 10,10-dibromotelluraxanthones (IXa, c). It should be noted that whereas 10,10-dibromotelluraxanthone is completely stable suring storage, its chloro analog is unstable and rapidly changes color under the influence of light.

A comparison of the frequencies of the vibrations of the carbonyl group in xanthone (1660 cm^{-1}) [16] and its analogs, viz., thiaxanthone (1645 cm^{-1}) [16], selenaxanthone (1620 cm^{-1}) [17], and telluraxanthone (1590 cm^{-1}), indicates a successive decrease in the frequency of the carbonyl vibration; this to some extent may constitute evidence for an increase in the contribution of the polar resonance forms of the B type as compared with A to the ground electronic state of the xanthone molecules on passing from oxygen to tellurium.

Like xanthene [18], telluraxanthene is readily metallated by phenyllithium.



9-Lithiotelluraxanthene (X) was synthesized in greater than 60% yield, as evidenced by the results of its treatment with carbon dioxide [telluraxanthene-9-carboxylic acid (XI) was obtained in 65% yield].



EXPERIMENTAL

The IR spectra of mineral oil suspensions of the compounds were recorded with a UR-20 spectrometer. The PMR spectra of solutions in d_6-acetone were recorded with a Tesla BS-587C spectrometer (80 MHz) with hexamethyldisiloxane as the internal standard.

2-Bromo- and 2-iododiphenylmethanes were obtained by reduction of the corresponding benzophenones with lithium aluminum hydride in the presence of anhydrous aluminum chloride by the method presented in [19].

Bis(2-benzylphenyl) Ditelluride (II). A solution of 45 g (0.18 mole) of o-bromodiphenylmethane in 50 ml of ether was added gradually with vigorous stirring in an argon atmosphere to a refluxing mixture of 2.5 g (0.36 mole) of finely cut lithium in 450 ml of absolute ether, during which the solution turned red. After all of the halo derivative had been added, the mixture was refluxed with stirring for another hour. It was then cooled, and 30 g (0.24 mole) of powdered tellurium was added to it in small portions with cooling and vigorous stirring. At the end of the exothermic reaction, the mixture was refluxed with stirring for 30 min, cooled, and treated carefully under a hood (H_2Te was evolved) initially with 300 ml of cold water and then with 100 ml of dilute hydrochloric acid (1:3). The mixture was allowed to stand overnight to complete the oxidation of H_2Te, after which it was filtered to remove the unchanged tellurium, and the tellurium was washed thoroughly on the filter with hot ether until the latter was no longer red-tinted. The combined ether extracts were washed twice with cold water and dried over anhydrous sodium sulfate, and the ether was evaporated to give 41.4 g (78%) of large red crystals of II with mp 101°C (from petroleum ether). PMR spectrum: 4.17 (s, 2H, CH_2),and 6.95-7.35 ppm (m, 9H, aromatic protons). Found: C 52.9; H 3.2%. $C_{26}H_{22}Te_2$. Calculated: C 53.0; H 3.7%.

Bis(2-benzylphenyl) ditelluride was similarly obtained in 61% yield from 2-iododiphenylmethane.

2-Trichlorotelluriodiphenylmethane (Ia). A) From Bis(2-benzylphenyl) Ditelluride. A stream of dry chlorine was passed through a solution of 17 g (0.029 mole) of II in 160 ml of dry methylene chloride cooled with ice water until the color of the solution changed from dark-red to light-yellow. The precipitated trichloride IA was removed by filtration, washed on the filter with petroleum ether, and dried to give 16 g of Ia in the form of fine light-yellow crystals. Evaporation of the filtrate gave an additional 5.5 g of Ia for an overall yield of Ia of 21.5 g (93%). The light-yellow crystals had mp 199-200°C (from glacial acetic acid). PMR spectrum: 4.53 (s, 2H, CH_2) and 7.27-7.67 ppm (m, 9H, aromatic protons). Found: C 38.4; H 2.8%. $C_{13}H_{11}Cl_3Te$. Calculated: C 38.9; H 2.7%.

B) From 2-Trimethylsilyldiphenylmethane. A solution of 32 g (0.14 mole) of 2-trimethylsilyldiphenylmethane [bp 127°C (2 mm)], obtained in 75% yield from 2-lithiodiphenylmethane and trimethylchlorosilane, was added to a refluxing solution of 33.7 g (0.13 mole) of tellurium tetrachloride in 400 ml of absolute toluene, and the resulting mixture was refluxed for 1 h. Approximately four-fifths of the solvent was removed by vacuum distillation, and the resulting solution was cooled to give 35 g (67%) of tellurium trichloride Ia.

2-Tribromotelluriodiphenylmethane (Ib). A solution of 1.82 g (11.4 mmole) of bromine in 5 ml of methylene chloride was added with stirring and cooling with ice water to a solution of 2 g (3.8 mmole) of II in 100 ml of methylene chloride, and the resulting yellow precipitate was removed by filtration, washed on the filter with petroleum ether, and dried to give 3.74 g (98%) of large yellow crystals with mp 197°C (from toluene). PMR spectrum: 4.85 (s, 2H, CH_2) and 7.25-7.78 ppm (m, 9H, aromatic protons). Found: C 29.0; H 2.0%. $C_{13}H_{11}Br_3Te$. Calculated: C 29.2; H 2.1%.

10,10-Dichlorotelluraxanthene (IIIa). A) From 2-Trichlorotelluriodiphenylmethane. A 4-g (0.03 mole) sample of anhydrous aluminum chloride was added in portions with stirring to a suspension of 12 g (0.03 mole) of Ia in 80 ml of freshly distilled o-dichlorobenzene (or 1,2,4-trichlorobenzene), and the resulting suspension was stirred at 65-70°C for 5 h. It was then cooled, and the resulting dark-green solution was poured into 300 ml of chloroform. A total of 300 ml of dilute hydrochloric acid (1:3) was added, and the mixture was stirred for 5 min and filtered. The organic layer was separated and dried over anhydrous Na_2SO_4, and the solvent was removed by distillation until the first crystal precipitated out of solution. The concentrated solution was cooled to room temperature, and 8.76 g (80%) of slightly yellowish crystals were removed by filtration. Recrystallization from chlorobenzene gave large colorless plates with mp 250-270°C. PMR spectrum: 4.83 (s, 2H, CH_2) and 7.13-7.93 ppm (m, 8H, aromatic protons). Found: C 42.4; H 2.6%. $C_{13}H_{10}Cl_2Te$. Calculated: C 42.8; H 2.7%.

B) By Oxidation of Telluraxanthene with Chlorine. A stream of dry chlorine was passed through a solution of 1.47 g (5 mmole) of telluraxanthene in 20 ml of benzene for 20 min, after which most of the solvent was removed by distillation, and the precipitate was removed by filtration, washed with petroleum ether, and recrystallized from chlorobenzene to give 1.76 g (96%) of product.

C) By Oxidation of Telluraxanthene with Cupric Chloride. An aqueous acetone solution of 1.40 g (11 mmole) of cupric chloride was added dropwise with stirring to a solution of 1.47 g (5 mmole) of telluraxanthene in 50 ml of acetone, and the mixture was refluxed for 0.5 h, and the resulting solution was filtered to remove the cuprous chloride. The filtrate was poured into water, and the precipitate was removed by filtration. Recrystallization from chlorobenzene gave 1.20 g (76%) of 10,10-dichlorotelluraxanthene.

Telluraxanthene (IV). A 10-g sample of finely ground potassium metabisulfite was added in small portions at 40-50°C in the course of 30 min to a suspension of 4 g (11 mmole) of IIIa in 30 ml of water, and the mixture was cooled. The precipitate was removed by filtration, washed on the filter with a small amount of cold water, and dried to give 3 g (93%) of colorless needles with mp 141°C (from petroleum ether). PMR spectrum: 3.85 (s, 2H, CH_2) and 6.90-7.93 ppm (m, 8H, aromatic protons). Found: C 52.9; H 3.2%. $C_{13}H_{10}Te$. Calculated: C 53.2; H 3.4%.

10,10-Dibromotelluraxanthene (IIIb). A) By Oxidation of Telluraxanthene with Bromine. A solution of 0.28 g (1.7 mmole) of bromine in 5 ml of benzene was added with stirring and cooling to 0-5°C to a solution of 0.5 g (1.7 mmole) of IV in 20 ml of benzene, and the resulting yellow precipitate was removed by filtration, washed with petroleum ether, and dried to give 0.75 g (98%) of yellow crystals with mp 260°C (decomp., from chlorobenzene). Found: C 34.7; H 2.0%. $C_{13}H_{10}Br_2Te$. Calculated: C 34.3; H 2.2%.

B) By Oxidation of Telluraxanthene with Cupric Bromide. An aqueous acetone solution of 2.24 g (0.01 mole) of cupric bromide was added dropwise with stirring to a solution of 1.47 g (5 mmole) of telluraxanthene in 50 ml of acetone, and the mixture was refluxed for 0.5 h. The cuprous bromide was removed by filtration, and the filtrate was poured into water. The resulting precipitate was removed by filtration, dried, and recrystallized from chlorobenzene to give 1.79 g (79%) of 10,10-dibromotelluraxanthene.

10,10-Diiodotelluraxanthene (IIIc). A solution of 1.27 g (5 mmole) of iodine in 10 ml of chloroform was added with stirring and cooling at 0-5°C to a solution of 1.4 g (4.8 mmole) of IV in 20 ml of chloroform, and the brown precipitate was removed by filtration, washed with petroleum ether, and dried to give 2.6 g (99%) of dark-red crystals with mp >230°C (decomp., from chlorobenzene). Found: C 28.2; H 1.6%. $C_{13}H_{10}I_2Te$. Calculated: C 28.5; H 1.8%.

Telluroniaxanthenyldimedone Ylid (V). A 7-ml sample of triethylamine was added with stirring and heating at 60-70°C to a solution of 7.28 g (0.02 mole) of 10,10-dichlorotelluraxanthene (IIIa) and 2.8 g (0.02 mole) of dimedone in 60 ml of benzene. After all of the triethylamine had been added, the mixture was stirred with heating for another 20 min. The hot solution was filtered to remove the precipitated triethylamine hydrochloride, and the solvent was removed from the filtrate to give 8.6 g (~100%) of colorless crystals of V with mp 155-156°C (benzene—petroleum ether). Found: C 58.0; H 4.2%. $C_{21}H_{20}O_2Te$. Calculated: C 58.4; H 4.6%.

10,10-Difluorotelluraxanthene (IIId). A 1-ml sample of concentrated HF was added to a solution of 2 g (4.6 mmole) of ylid V in 10 ml of hot ethanol, after which the solvent was evaporated, and the precipitate was washed with benzene and dried to give 1.51 g (98%) of colorless cyrstals of IIId with mp >260°C (decomp., from chlorobenzene). Found: C 46.7; H 3.2%. $C_{13}H_{10}F_2Te$. Calculated: C 47.0; H 3.0%.

10,10-Bis(trifluoroacetyl)telluraxanthene (IIIe). A 1-ml sample of trifluoroacetic acid was added to a solution of 2 g (4.6 mmole) of V in 10 ml of hot ethanol, and the colorless precipitate that formed instantaneously was removed by filtration and dried to give 2.08 g (87%) of colorless crystals with mp 165°C (from ethanol). Found: C 39.5; H 1.8%. $C_{17}H_{10}F_6O_4Te$. Calculated: C 39.3; H 1.9%.

Complex (VIa) of Telluraxanthene with Mercuric Chloride. A solution of 1.36 g (5 mmole) of mercuric chloride in 10 ml of alcohol was added to a solution of 1.47 g (5 mmole) of telluraxanthene (IV) in 50 ml of benzene, and the resulting light-yellow precipitate was refluxed with stirring for 30 min. The solution was cooled, and the precipitate was removed by filtration to give 2.38 g (84%) of VIa with mp 205°C (from toluene). Found: C 27.4; H 1.9%. $C_{13}H_{10}Cl_2HgTe$. Calculated: C 27.6; H 1.8%.

Complex (VIb) of Telluraxanthene with Mercuric Bromide. This compound was obtained by a method similar to that presented above. The reaction of 1.47 g (5 mmole) of telluraxanthene and 1.81 g (5 mmole) of mercuric bromide gave 3.15 g (96%) of a complex in the form of a light-yellow powder with mp 206°C (from toluene). Found: C 23.6; H 1.4%. $C_{13}H_{10}Br_2HgTe$. Calculated: C 23.8; H 1.5%.

Complex (VIc) of Telluraxanthene with Silver Perchlorate and Complex (VId) of Telluraxanthene with Silver Nitrate. These compounds were obtained by mixing a benzene solution of telluraxanthene with acetonitrile solutions of equimolar amounts of silver perchlorate and silver nitrate. Both complexes darkened rapidly during storage, evidently as a consequence of decomposition with the liberation of metallic silver; acceptable analyses were therefore not obtained for them.

9,10-Dihydro-10-methyltelluraxanthenylium Perchlorate (VII). A solution of 2.08 g (0.01 mole) of $AgClO_4$ in 50 ml of acetonitrile was added with stirring to a solution of 2.94 g (0.01 mole) of telluraxanthene and 5 ml of methyl iodide in 250 ml of acetonitrile, and the mixture was refluxed with stirring for 30 min. The resulting solution was cooled, and the precipitated silver iodide was removed by filtration (2.32 g). Evaporation of the filtrate gave VII in the form of large colorless regular hexahedra with mp 183°C (explosively, from acetonitrile—toluene). Found: C 41.0; H 3.4%. $C_{14}H_{13}ClO_4Te$. Calculated: C 41.1; H 3.2%.

Telluraxanthone (VIII). A solution of 3.65 g (0.01 mole) of dichlorotelluraxanthene and 2.94 g (0.01 mole) of potassium dichromate in 30 ml of glacial acetic acid was refluxed for 8 h, and the resulting solution was cooled and poured into 300 ml of cold water. The precipitated 10,10-dichlorotelluraxanthone (IXa) was removed by filtration, washed with water, dried, and suspended in 100 ml of water. A 13.6-g sample of potassium metabisulfite was added with vigorous stirring, after which the mixture was stirred for another 30 min, at the end of which period the temperature was raised to 50-60°C. The mixture was cooled, and the precipitated telluraxanthone was removed by filtration, washed with ice water, and dried to give 2.32 g (75%) of telluroxanthone. Recrystallization from heavy petroleum ether gave large yellow crystals with mp 115°C. IR spectrum: 1590, 1575, 1420, 1285, 1150, 1110, 1020, 920, 875, 815, 770, 730 cm^{-1}. Found: C 50.6; H 2.9%. $C_{13}H_8OTe$. Calculated: C 50.7; H 2.6%.

10,10-Diacetyltelluraxanthone (IXb). A solution of 2.94 g (0.01 mole) of IV and 5.88 g (0.02 mole) of potassium dichromate in 30 ml of glacial acetic acid was refluxed for 8 h, after which it was cooled and poured into 200 ml of cold water. The resulting precipitate was removed by filtration, washed with water, and dried. Recrystallization from chlorobenzene gave 2.68 g (63%) of IXb in the form of large light-yellow plates with mp 233°C (decomp.). Found: C 47.7; H 3.1%. $C_{17}H_{14}O_5Te$. Calculated: C 47.9; H 3.3%. Reduction of IXb with an aqueous solution of potassium metabisulfite gave telluraxanthone in 87% yield.

10,10-Dichlorotelluraxanthone (IXa). A stream of dry chlorine was passed with stirring through a solution of 1.54 g (5 mmole) of telluraxanthone in 50 ml of chloroform for 10 min, after which the solvent was evaporated, and the residue was dried to give 1.85 g (98%) of Xa, which was recrystallized from chlorobenzene. The product rapidly turned bright-red during storage.

10,10-Dibromotelluraxanthone (IXc). A solution of 0.88 g (5.5 mmole) of bromine in 5 ml of chloroform was added with stirring and cooling with ice water to a solution of 1.54 g (5 mmole) of telluraxanthone in 50 ml of chloroform, after which the solvent was evaporated, and the residue was dried to give 2.3 g (98%) of yellow crystals with mp >260°C (from chlorobenzene). Found: C 33.0; H 1.9%. $C_{13}H_8Br_2OTe$. Calculated: C 33.4; H 1.7%.

Lithiation of Telluraxanthene. Telluraxanthene-9-carboxylic Acid (XI). A solution of phenyllithium, prepared by the usual method from 3.5 g of bromobenzene and 0.3 g of lithium in 50 ml of absolute ether, was added with stirring to a solution of 5.88 g (0.02 mole) of telluraxanthene in 50 ml of absolute benzene, and the solution was stirred at 20°C for 1 h and refluxed for 1 h. The resulting dark-red solution of 9-lithiotelluraxanthene (X) was poured over dry ice, after which the carbon dioxide was allowed to evaporate. The organic layer was treated with an aqueous solution of sodium hydroxide, the aqueous and organic layers were separated, and the aqueous layer was extracted several times with benzene. The aqueous layer was then acidified with excess concentrated HCl, and the resulting precipitate was removed by filtration, washed on the filter with ice water, and dried to give 4.42 g (65%) of XI. Recrystallization from ethanol gave light-yellow crystals with mp 208-209°C (decomp.). Found: C 49.5; H 2.8%. $C_{14}H_{10}O_2Te$. Calculated: C 49.8; H 3.0%. IR spectrum: 2625, 1700, 1590, 1575, 1405, 1305, 1230, 1170, 1030, 935, 770 cm^{-1}.

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