lurium atom is slightly distorted.[2] In the present, not centrosymmetric structure, the distortion is found to be larger (Table 1). It is not clear if this can be interpreted as resulting from lattice-packing effects or from a stereochemical activity of the lone pair of electrons. However, the lone pair is not stereochemically active in the sense that it occupies a position in the coordination polyhedron.

The tellurium-chlorine bond lengths are ranging from 2.457(3) Å to 2.598(3) Å, while the tellurium-sulphur distances are found to be 2.649(3) Å and 2.725(3) Å. These values show a much greater variation than the corresponding ones in the orthorhombic structure; however, the average Te–Cl and Te–S bond lengths of 2.53 Å and 2.69 Å found here are in good agreement with the corresponding values of 2.53 Å and 2.70 Å found for the orthorhombic form. The average Te–Cl distance is further in good agreement with reported bond lengths in hexachlorotellurate species,[3,4] *i.e.*, equal to the sum of the octahedral radius of Te(IV) [5] and the covalent radius of Cl, while the average Te–S distance is significantly larger than the sum of the octahedral radius of Te(IV) and the covalent radius of S, 2.59 Å. Similar Te–S bond lengths or even greater ones are found in other tellurium(IV) complexes.[6,7] An explanation of why the Te–Cl bond lengths are normal while the Te–S ones are so large can at present not be given.

The S–C bond lengths of 1.745(9) Å and 1.740(9) Å are in good agreement with the corresponding values found for the orthorhombic structure and for tetramethylthiourea complexes of tellurium(II).

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Received February 21, 1973.

Synthesis of Tritium-labelled Tetrahydrocannabinol and Cannabidiol

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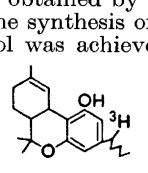
Several syntheses of ^{3}H-[1-5] and ^{14}C-labelled [1,6-8] tetrahydrocannabinols (THC's) have been reported. In distribution studies using autoradiography it is necessary to use compounds of high specific activity. For this purpose [9] tritium-labelled Δ^{1}-THC of specific activity 1.6 Ci/mmol has been prepared. This synthesis together with the preparation of tritium-labelled cannabidiol is here reported.

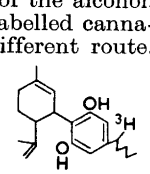
1-(3,5-Dimethoxyphenyl)-1-pentanone, synthesised according to the method of Baeckström and Sundström,[10] was reduced with sodium borohydride. The resulting alcohol,[4] dissolved in methanol, was tritiated with tritium gas over palladium supported on carbon. The reduction was then completed with hydrogen. The labelled dimethylolivetol was demethylated by heating with hydriodic acid.[11] The resulting olivetol was reacted with (+)-*trans*-*p*-menthadien-2,8-ol-1 to give (−)$\Delta^{1,6}$-THC,[12] which after purification on silica gel, was isomerised to (−)Δ^{1}-THC.[12]

1-(3,5-Dimethoxyphenyl)-1-pentanone could also be tritiated with tritium gas using the same condition as described above. The reduction was, however, slower than that of the corresponding alcohol and the exchange of tritium with the hydrogen atoms in the solvent occurred to such an extent that the resulting labelled dimethylolivetol had a specific activity only half of that obtained by tritiation of the alcohol.

The synthesis of tritium-labelled cannabidiol was achieved by a different route.



Δ¹-TETRAHYDROCANNABINOL



CANNABIDIOL

1-(3,5-Dimethoxyphenyl)-1-pentanone was reduced with lithium aluminium hydride-^{3}H. The alcohol obtained was further reduced with hydrogen to dimethylolivetol, which was demethylated as described above. The resulting labelled olivetol was reacted with (+)-*trans*-*p*-menthadien-2,8-ol-1 in the presence of *N*,*N*-dimethylformamide dineopentylacetal giving (−)-cannabidiol.[12]

The latter procedure using lithium aluminium hydride-^{3}H or sodium borohydride-^{3}H would, in general, be a suitable procedure for labelling cannabinoids of low or intermediate specific activity, since the pentanone derivative, an intermediate in the preparation of olivetol,[10] is readily available. Further, no special laboratory facilities are needed, and the hydride-^{3}H is efficiently utilized in the synthesis. In contrast to tritium-labelled cannabinoids prepared by exchange methods,[1,3,5] the stability of the label in the benzylic position in the side chain is satisfactory during both metabolic experiments [9] and isolation procedures.

Experimental. Δ^1-Tetrahydrocannabinol-^{3}H. A solution of 1-(3,5-dimethoxyphenyl)-1-pentanol (122 mg) in methanol (2 ml) was tritiated with tritium gas (19 Ci tritium) over palladium supported on carbon (10 %, 22 mg) at room temperature and atmospheric pressure. After 70 min the reduction was continued with hydrogen for 3 h, after which the catalyst was filtered off and the solvent evaporated. Any exchangeable tritium was removed by repeatedly dissolving the residue in methanol and evaporating the solvent. The crude dimethylolivetol and hydriodic acid (57 %, 4 ml) were stirred for 3 h at 110° under nitrogen. The mixture was poured into ice water and extracted with chloroform. The extract was subjected to preparative thin layer chromatography (silica gel, chloroform:methanol, 19:1) giving olivetol (70 mg) of specific activity 1.6 Ci/mmol. The labelled Δ^1-THC was then synthesised as previously described,[12] and purified by preparative thin layer chromatography (silica gel, ether:light petroleum, 1:9). The plates were developed three times.

Cannabidiol-^{3}H. To a solution of 1-(3,5-dimethoxyphenyl)-1-pentanone (424 mg) in ether (20 ml), lithium aluminium hydride-^{3}H (5 mg, 25 mCi) was added. The mixture was refluxed for 15 h, after which an excess of inactive lithium aluminium hydride was added. Refluxing was then continued for 5 h. The resulting alcohol (420 mg) was dissolved in methanol (5 ml) and hydrogenated over palladium (from 20 % palladium hydroxide[13] on carbon, 50 mg) at room temperature and atmospheric pressure. After 4 h one molar equivalent of hydrogen had been consumed. The catalyst was filtered off and the solvent evaporated. The crude dimethylolivetol was demethylated and purified as described above, giving olivetol (235 mg) with specific activity 11 mCi/mmol. Dilution with inactive olivetol (2.0 g) and condensation with (+)-*trans*-*p*-menthadien-2,8-ol-1 in the presence of *N*,*N*-dimethylformamide dineopentylacetal afforded (−)cannabidiol of specific activity 0.9 mCi/mmol.

Acknowledgements. We are indebted to the *Swedish Medical Research Council* and *Försvarsmedicinska Forskningsdelegationen* for support.

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Received March 2, 1973.