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Fast microwave assisted synthesis of *p*-methoxyphenyltellurium trichloride

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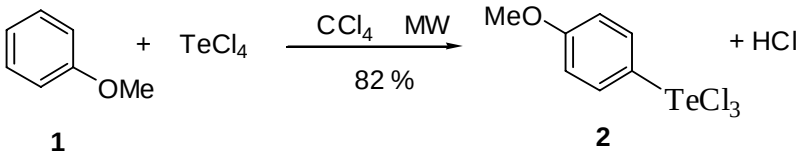
Abstract: Microwave assisted formation of C-Te bonds is applied to the preparation of p-methoxyphenyltellurium trichloride in a fast, direct and simple way.

Aryl chalcogenides structural motifs are commonly found in a variety of molecules with biological interest [1] and new materials[2]. Furthermore, organotellurium compounds have been used as precursors in the synthesis of molecules with different functionalities, such as dienes and enediynes,[3] present in the structure of important classes of natural products.[4]

This communication deals with the synthesis of aryltellurium trihalides $ArTeX_3$. These can be used in the arylation of olefins[5] and in the preparation of several organotellurium derivatives[6] and have been identified as biocides.[7]

Up to date, few studies have been devoted to the use of microwave irradiation to enhance tellurium chemistry either with[8] or without[9] formation of C-Te bonds. Among the several methods[6] available for the synthesis of $ArTeX_3$ we chose the condensation of tellurium tetrachloride with aromatic compounds, in order to study the possibility of enhancing the reaction by microwave irradiation.

The first attempt, based on Comasseto's synthesis by conventional heating,[10] to get *p*-methoxyphenyltellurium trichloride from anisole (**1**) and tellurium tetrachloride was done by irradiating a mixture of both reagents at 120ºC under solvent-free conditions. However, only decomposition of the reaction mixture was achieved. Thus, the reaction was checked in the presence of solvent.[11] The irradiation for 15 minutes of a suspension of anisole and $TeCl_4$ in refluxing carbon tetrachloride did not lead to an appreciable amount of **2**.



1 **2**

Scheme 1

With the purpose of using higher temperatures the irradiation was carried out in a sealed vessel. Hence, a 1:1 ratio of anisole and $TeCl_4$ was irradiated for 10 minutes with the maximum temperature control set at 120ºC and irradiation power 80W, yielding a 73% of *p*-methoxyphenyltellurium trichloride (**2**). Raising the irradiation power to 100W in the same reaction time the yield reached 82%, higher irradiation powers (200W, 250W) did not increase the yield.

In summary, here is reported the first microwave-assisted formation of aryltellurides. This proves that microwaves are a valuable tool for the formation of Ar-C bonds, opening a new facet in the field of organochalcogenide chemistry.

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Experimental procedure

Anisole (540 mg, 5 mmol), $TeCl_4$ (135 mg, 0,5 mmol) and CCl_4 are mixed in a sealed vessel. Reaction mixture was irradiated in a CEM Discover monomode oven for 10 minutes at 120ºC (100W). On cooling the reaction mixture, a yellow crystalline solid crystallizes out, this was filtered and vacuum dried. The solid (139 mg, 82%) was identified as *p*-methoxyphenyltellurium trichloride (**2**). IR(*Golden-Gate*): 3013, 1566, 1485, 1256, 1180, 1046, 817 cm^{-1}. ^1H NMR ($CDCl_3$) δ 3.81 (s, 3H, OCH_3), 6.99 (d, 2H, *J*=9Hz.), 8.31 (d, 2H, *J*= 9Hz.). ^{13}C NMR ($CDCl_3$) δ 56.16, 114.06, 114.64, 134.59, 135.93. MS *m/z (%)* 344 (M^++4, 39), 342 (M^++2, 36), 340 (M^+ [$3xCl_{35}$, Te_{128}], 23), 307 (19), 305 (15), 303 (8), 272 (85), 270 (73), 268 (40), 237 (65), 235 (60), 233 (37), 214 (100), 199 (87), 142 (90).

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