

# Novel microwave-assisted synthesis of the organotellurium compound ammonium trichloro (dioxoethylene-O,O') tellurate (AS101)

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## *Abstract:*

*Ammonium trichloro[1,2-ethanediolato-O,O']-tellurate (AS101) is a potent immunomodulator with a variety of potential therapeutic applications.. The effect of microwave power, synthesis temperature and time on AS101 synthesis was examined in this work. An easy, efficient and fast procedure for syntheses of AS101 by MAOS is presented*

Keywords: tellurium, AS101, immunomodulator, microwave

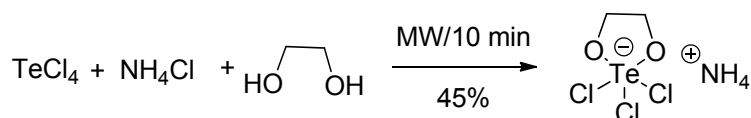
Over recent years, heating and driving chemical reactions by microwave (MW) energy has been a significant interest in the scientific community, in particular applied to microwave-assisted organic synthesis (MAOS) and drug discovery [1]. Ammonium trichloro[1,2-ethanediolato-O,O']-tellurate (AS101) is the most important synthetic Tellurium compound from the standpoint of its biological activity. It is a potent immunomodulator with a variety of potential therapeutic applications [2-4]. Continuing with our studies on MAOS [5-10] we draw our attention to this compound. The effect of microwave power, synthesis temperature and time on AS101 synthesis was examined in this work. Ç

The first attempt, based on Albeck's synthesis by conventional heating [11], to get AS101 from TeCl<sub>4</sub> and NH<sub>4</sub>Cl dissolved in ethylene glycol, was done by irradiating a mixture of both reagents at 120°C under solvent-free conditions. However, only decomposition of the reaction mixture was achieved. Thus, the reaction was checked in the presence of solvent, but using ethylene glycol both as reagent and solvent. The reaction was carried out in a sealed tube, the reaction time found was of 10 minutes with a yield of 45%; and by conventional heating to get AS101 4 hours are required (51% of yield). The procedure based on microwave irradiation has the potential to evolve to incorporate new

processes for obtaining AS101 derivatives, since the optimization of newly discovered lead compounds, relies upon the advancement of synthetic technologies.

### General Experimental procedure

TeCl<sub>4</sub> (1.35 g) and NH<sub>4</sub>Cl (0.387 g) were dissolved in ethylene glycol (5 mL). The mixture was MW irradiated in a CEM Discover monomode oven for 10 minutes at 80°C (80W).



Scheme1

On cooling the reaction mixture, a white crystalline solid is obtained, washed with acetonitrile, filtered and vacuum dried. The solid (706 mg, 45%) was identified as:

C <sub>2</sub> H <sub>8</sub> Cl <sub>3</sub> NO <sub>2</sub> Te	Calc. C 7,70	H 2,58	N 4,49
	Enc. C 7,85	H 2,37	N 4,42

IR (Golden-Gate):  $\nu = 3183$  (NH):  $1390$  (NH<sub>4</sub><sup>+</sup>):  $1019$ :  $894$  cm<sup>-1</sup>.

RMN-<sup>1</sup>H (DMSO-*d*):  $\delta = 4.36$  (s. 4H. CH<sub>2</sub>):  $7.16$  (t. 4H.  $J = 50$ Hz. NH<sub>4</sub><sup>+</sup>)

RMN-<sup>13</sup>C (DMSO-*d*):  $68,13$ .

EM:  $m/z$  =  $292$  (M<sup>+</sup>-NH<sub>4</sub>,1),  $290$  (M<sup>+</sup> - NH<sub>4</sub>,1),  $260$  (M<sup>+</sup>-NH<sub>4</sub><sup>+</sup> - Cl [Te130, 2Cl35],7),  $258$  (M<sup>+</sup>-NH<sub>4</sub><sup>+</sup> - Cl [Te128, 2Cl35],13),  $256$  (M<sup>+</sup>-NH<sub>4</sub><sup>+</sup> - Cl [Te126, 2Cl35],13),  $254$  (9),  $224$  (42),  $223$  (35),  $221$  (19),  $200$  (36),  $198$  (27),  $196$  (14),  $190$  (69),  $188$  (63),  $186$  (37),  $165$  (21),  $163$  (18),  $161$  (11),  $146$  (5),  $130$  (25),  $128$  (23),  $126$  (13).

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