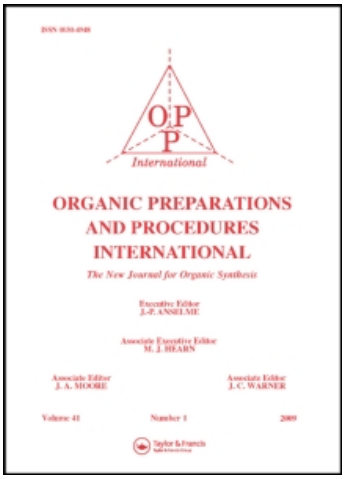

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PREPARATION OF SELENA- AND TELLURA PHTHALIC ANHYDRIDE

Jan Bergman[a]; L. Engman[a]
[a] Department of Organic Chemistry, Royal Institute of Technology, Stockholm, SWEDEN

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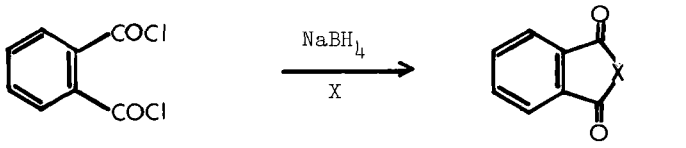
OPPI BRIEFS

PREPARATION OF SELENA- AND TELLURA PHTHALIC ANHYDRIDE

Submitted by Jan Bergman* and L. Engman
(6/1/78)

Department of Organic Chemistry, Royal Institute of Technology
100 44 Stockholm, SWEDEN

The hitherto unknown, relatively stable compounds selena phthalic anhydride (IIa) and tellura phthalic anhydride (IIb) from phthaloyl chloride (I) have been prepared by a phase transfer technique [D. L. Klayman and T. S. Griffin, J. Am. Chem. Soc., 95, 197 (1973)].



I II a) X = Se

b) X = Te

EXPERIMENTAL

All the reactions were run under a hydrogen atmosphere to prevent oxidation of the oxygen-sensitive selenide and telluride ions. All melting points are uncorrected. Infrared spectra were obtained using a Perkin Elmer 257 instrument. Mass spectra were obtained with an LKB 9000 mass spectrometer.

Selena phthalic anhydride (IIa).- Sodium borohydride (4.0 g, 106 mmoles) was added at room temperature to a well-stirred suspension of gray powdered selenium (4.0 g, 51 mmoles) in water (50 ml). Considerable foaming (H_2) occurred and the selenium was consumed in about 10 min. Phthaloyl chloride (10 g, 50 mmoles) in toluene (50 ml) was added through a dropping funnel. The temperature was then raised to 60° after addition of tetrabutylammonium hydrogen sulfate (0.5 g). After 6 hrs, the phases were separated and

the organic layer was washed with sodium carbonate (5%, aq.). Evaporation gave 6.5 g (61%) of a red product which could be recrystallized from CCl_4/hexane, mp. 112-113°:

IR ν_{max}^{KBr} cm^{-1} 1750(m), 1690(vs), 1605(w), 1585(m), 1450(m), 1325(m), 1270 (w), 1240 (m), 1215(s), 1210(s), 1155(w), 1025(w), 975(w), 910(s), 880(w), 840(s), 790(m), 780(s), 700(s), 660(s); M.S. m/e (rel. intensity) 214(13), 213(6), 212(M^{+80}Se, 68), 210(34), 209(12), 208(14), 156(6), 132(5), 104 (100), 77(7), 76(73), 75(13), 74(15), 66(7), 50(40).

Tellura phthalic anhydride (IIb).- Sodium borohydride (4.0 g, 106 mmoles) was added with magnetic stirring at room temperature to a suspension of tellurium (5.0 g, 51 mmoles) in water (50 ml). The mixture was heated to 40° whereupon a vigorous evolution of gas (H_2) started. All the tellurium was consumed in about 15 min. Phthaloyl chloride (10 g, 50 mmoles) in toluene (50 ml) was added through a dropping funnel. After addition of tetrabutylammonium hydrogen sulfate (0.5 g) the mixture was left overnight without additional heating. The organic layer was separated and washed with 5% aqueous sodium carbonate. Evaporation of the solvent gave 4.1 g (40%) of tellura phthalic anhydride together with a small amount of pthalide. The product could be recrystallized from hexane, mp. 127°:

IR ν_{max}^{KBr} cm^{-1} 1770(s), 1660(vs), 1575(s), 1465(w), 1440(m), 1340(w), 1310 (w), 1260(m), 1200(s), 1190(m), 1180(w), 1150(w), 1035(w), 1020(w), 880(s), 870(s), 860(s), 830(s), 780(s), 690(s), 640(s); M.S. m/e (rel. intensity) 263(8), 262(M^{+} ^{130}Te, 79), 261(7), 260(75), 259(5), 258(46), 257(20), 256 (12), 254(6), 234(5), 232(5), 206(12), 204(11), 202(7), 130(6), 128(10), 126(11), 105(9), 104(100), 77(7), 76(6), 75(13), 74(12), 50(31).