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### PREPARATION OF SELENA- AND TELLURA PHTHALIC ANHYDRIDE

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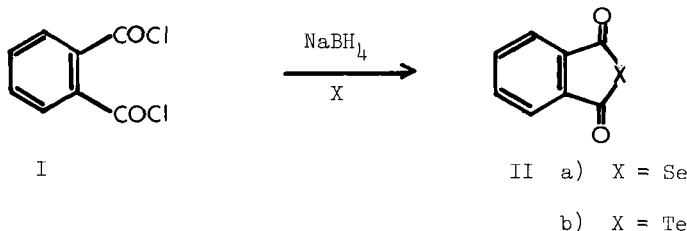
## OPPI BRIEFS

### PREPARATION OF SELENA- AND TELLURA PHTHALIC ANHYDRIDE

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

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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)].



### 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  $60^\circ$  after addition of tetrabutylammonium hydrogen sulfate (0.5 g). After 6 hrs, the phases were separated and

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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  $\text{CCl}_4$ /hexane, mp. 112-113°:

IR  $\nu_{\text{max}}^{\text{KBr}}$   $\text{cm}^{-1}$  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( $\text{M}^{+80}\text{Se}$ , 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 ( $\text{H}_2$ ) 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 phthalide. The product could be recrystallized from hexane, mp. 127°:

IR  $\nu_{\text{max}}^{\text{KBr}}$   $\text{cm}^{-1}$  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( $\text{M}^{+130}\text{Te}$ , 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).