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Document Title: A Systematic Evaluation of the Analysis of Drug Microcrystals Using Infrared Microspectroscopy

Author(s): Monica Joshi

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methanol. Tests were conducted by placing 1-5 μL methanolic solutions on a clean glass slide to achieve 5-25 μg of the test substance. The dried residue was dissolved in either 5 μL of H_2O , HCl or CH_3COOH . The acidic solvents were only used for the aqueous gold and platinum chloride reagents. To this drop 5 μL of the reagent was added. The drop was then mixed with a glass capillary to initiate crystallization. The glass slide was set aside and observed periodically for crystal growth. A Leica DM 750P polarized light microscope or an Olympus BX43F polarized light microscope were used to observe the resulting microcrystals. Characteristic microcrystals were documented as photomicrographs in the Olympus cellSense Entry imaging software at 100x magnification.

The following series of reagents were used for the project:

1. Gold chloride: Two formulations were used
 - a. 5% Aqueous HAuCl_4
 - b. 5% HAuCl_4 in 1:2 concentrated H_2SO_4 : H_2O
2. Gold bromide: 1 g HAuCl_4 + 0.76g NaBr in 5 mL glacial CH_3COOH + 15 mL 2:3 concentrated H_2SO_4 : H_2O
3. Platinum chloride: 5% Aqueous H_2PtCl_6
4. Platinum bromide: 1 g H_2PtCl_6 in 1.7 mL 40% HBr + 20 mL 2:3 concentrated H_2SO_4 : H_2O
5. Mercuric chloride: Two formulations were used depending on the length of time for crystal growth. The reagent crystallizes very quickly in the aqueous formulation and prevents growth of drug-reagent crystals.
 - a. 1 g HgCl_2 in 100 mL water
 - b. 1 g HgCl_2 2.5 mL 1:1 Glycerol: Water 14.2 mL water, 500 μL 3M HCl

