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Library of Microcrystalline Tests for Novel Psychoactive Substances

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Introduction

A microcrystalline test is a precipitation reaction between a drug and a reagent, forming an insoluble drug-reagent complex that is unique to that specific test. These tests are quick, requiring minimal sample preparation and can be non-destructive. Therefore, they can be used as preliminary and confirmatory tests with expertise. Microcrystalline tests are one of the oldest analytical chemistry practices and their use for classic drugs such as cocaine, heroin and amphetamines is well-documented. However, there is very limited research on microcrystalline tests for the novel compounds encountered by law enforcement today. This research is an effort to increase understanding and promote use of microcrystalline tests for novel psychoactive substances.

Infrared spectroscopy is a well-established technique for the identification of drugs and adulterants and is categorized as a SWGDRUG category A technique, providing selectivity through structural information. Microcrystalline tests are a category B technique that provides selectivity by leveraging differences in physical and chemical characteristics of substances. An analytical scheme that combines these two techniques yields both visual data and structural information.

This library compiles the images of microcrystals and an infrared spectrum for the most characteristic microcrystal of that substance. The 30 substances included in the library represent the ever-evolving structural classes of cathinones, phenethylamines, piperazines, aminoindanes and opioids. The section for each substance includes a brief introduction of the substance, descriptions and photomicrographs of microcrystals observed with different test reagents, and infrared spectra of one or more microcrystal tests. The infrared spectra demonstrate that the microcrystal observed with a specific reagent is indeed representative of the structure of the substance being studied. The microcrystal characteristics combined with the infrared spectrum give both a SWGDRUG category A and category B test for that substance.

The sensitivity of the tests varies widely both between classes of substances and within a class. Characteristic crystals were observed with test amounts ranging between 1-30 μg of the substance. The microcrystalline tests that performed reliably with a practical crystal formation time are reported in the library.

The purpose of the library is to serve as a reference to analysts performing microcrystalline tests. We invite analysts working with typical forensic samples to provide feedback on the performance of the tests and any deviations from crystals observed with reference chemicals.

Procedures and Instrumentation

Chemical reference standards for all substances were purchased from Cayman Chemical (Ann Arbor, MI) as 1 mg/mL methanolic solutions or as 1 mg solids. The solids were dissolved in 1 mL methanol for the study. Reagents used in this study were made as per the recipes listed below. A drop of test solution was placed on a glass slide to achieve a dried residue amount between 1- 30 µg depending on the analyte. The residue was dissolved in 5-10 µL of water, 10% hydrochloric acid or 10% acetic acid solutions. To this drop, 10 µL of reagent was added and the drop mixed to stimulate crystal growth. Microcrystal formation time, shape, habit, and features under crossed polars were observed and documented.

A Leica DM 750P polarized light microscope and 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 100-200x magnification. A Thermo Scientific™ Nicolet™iN™10 Infrared Microscope with a liquid nitrogen cooled mercury, Cadmium, Tellurium (MCT) detector was used for analysis. All data was processed and analyzed in the Thermo Scientific™ OMNIC™ Picta™ software. The spectra were collected in transmission mode on AMTIR windows. Crystals grown on glass slides were carefully moved to the AMTIR windows with Microtools (MiTeGen LLC, Ithaca, NY). Excess reagent was wicked away with fine paper wicks and the crystals were washed with chloroform. This procedure removed interferences in the spectra and improved the quality of the infrared spectrum collected for a microcrystal.

Reagents Evaluated

1. Gold chloride: Two formulations were used
    - 5% Aqueous $HAuCl_4$
    - 5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20
2. Gold bromide ($HAuBr_4$): 1 g $HAuCl_4$ + 0.76g NaBr in 5 mL glacial CH_3COOH + 15 mL 2:3 concentrated H_2SO_4: H_20
3. Platinic chloride (H_2PtCl_6): 5% Aqueous H_2PtCl_6
4. Platinic bromide (H_2PtBr_6): 1 g H_2PtCl_6 in 1.7 mL 40% HBr + 20 mL 2:3 concentrated H_2SO_4: H_20
5. Mercuric chloride ($HgCl_2$): 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.
    - 1 g $HgCl_2$ in 100 mL water
    - 1 g $HgCl_2$ 2.5 mL 1:1 Glycerol: Water 14.2 mL water, 500 µL 3M HCl
6. Mercuric iodide (HgI_2): 1 g HgI_2 in 20ml 27:73 HCl: H_20

Novel Psychoactive Substances Studied

I. Cathinones

For more information about this class of substances:
https://www.emcdda.europa.eu/publications/drug-profiles/synthetic-cathinones_en
https://www.drugabuse.gov/publications/drugfacts/synthetic-cathinones-bath-salts

1. 3,4-Methylenedioxypyrovalerone (3,4-MDPV)
2. Methcathinones
3. Ethcathinone
4. Pentedrone
5. Mephedrone
6. 2-Ethylmethcathinone (2-EMC)
7. 3-Ethylmethcathinone (3-EMC)
8. 3-Methylbuphedrone
9. 4-Methylbuphedrone
10. Butylone
11. Methylone
12. NRG-3
13. 4-Fluoromethcathinone
14. α-Pyrrolidinopentiophenone
15. 4'-methyl-α-Pyrrolidinohexanophenone (MPHP)
16. 4'-methyl-α-Pyrrolidinopropiophenone (4-MePPP)

II. Phenethylamines
For more information about this class of substances:
https://www.unodc.org/LSS/SubstanceGroup/Details/275dd468-75a3-4609-9e96-cc5a2f0da467
https://www.nflis.deadiversion.usdoj.gov/DesktopModules/ReportDownloads/Reports/NFLIS_SR_Emerging_II.pdf
https://www.nflis.deadiversion.usdoj.gov/DesktopModules/ReportDownloads/Reports/NFLIS-SR-2CPEA-PiperazineTrypt.pdf

1. 25B-NBOMe
2. 2C-B
3. 2C-B-BZP
4. 2C-B-FLY
5. Bromo-DragonFLY

III. Piperazines
https://www.unodc.org/LSS/SubstanceGroup/Details/8242b801-355c-4454-9fdc-ba4b7e7689d5
https://www.nflis.deadiversion.usdoj.gov/DesktopModules/ReportDownloads/Reports/NFLIS_SR_Emerging_II.pdf
https://www.nflis.deadiversion.usdoj.gov/DesktopModules/ReportDownloads/Reports/NFLIS-SR-2CPEA-PiperazineTrypt.pdf

1. BZP
2. 1-(4-Methoxyphenyl) piperazine (4-MeOPP)

3. 4-FluoroBZP

IV. Aminoindanes
https://www.unodc.org/LSS/SubstanceGroup/Details/8fd64573-c567-4734-a258-76d1d95dca25
1. 5-IAI
2. MDAI

V. Opioids
https://www.dea.gov/taxonomy/term/331
https://www.dea.gov/sites/default/files/2018-07/DIR-040-17_2017-NDTA.pdf
https://www.unodc.org/LSS/announcement/Details/ff4b4da3-1220-49e3-8010-aadf7206572e
1. Fentanyl
2. FIBF
3. Furanyl Fentanyl
4. W-18

Overall Observations and Conclusions

Microcrystalline tests are quick tests to determine the presence of target substances in a given liquid or solid. Some substances give easily recognizable crystals and others give crystals that are amenable for infrared microspectroscopy. In some cases, the crystals are unique to that substance when studied as a pure substance but in mixtures or in unknown samples, the crystal form may be similar to other crystals of other substances with the same reagent. Therefore, the crystals need to be studied carefully for characteristic features and must be combined with instrumental techniques to improve selectivity.

Overall the gold-based reagents gave multiple reactions and formed distinct crystals with several of the substances studied.

A study of positional isomers such as 2- EMC and 3-EMC, 3-Methylbuphedrone and 4-Methylbuphedrone demonstrates the strength of the microcrystalline tests. Crystals of both isomers are observed in a mix of the isomers. Each isomer gives distinct crystals with the appropriate test reagent. When combined with IR microspectroscopy, the isomers can be structurally identified.

Additional Resources

1. The challenge of novel psychoactive substances, Global SMART Programme, United Nations Office on Drugs and Crime (UNODC), 2013
https://www.unodc.org/documents/scientific/NPS_Report.pdf

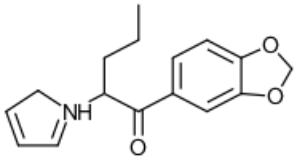
2. McCrone Research Institute. A Modern Compendium of Microcrystal Tests for Illicit-Drugs and Diverted Pharmaceuticals, 2015.

3. Leonie Elie, Mark Baron, Ruth Croxton, Mathieu Elie, *Microcrystalline identification of selected designer drugs*, Forensic Science International, Volume 214, Issue 1, 2012, Pages 182-188

4. C.C. Fulton, *Modern Microcrystal Tests for Drugs*, first ed., John Wiley & Sons, Inc., New York, 1969

Cathinone Class

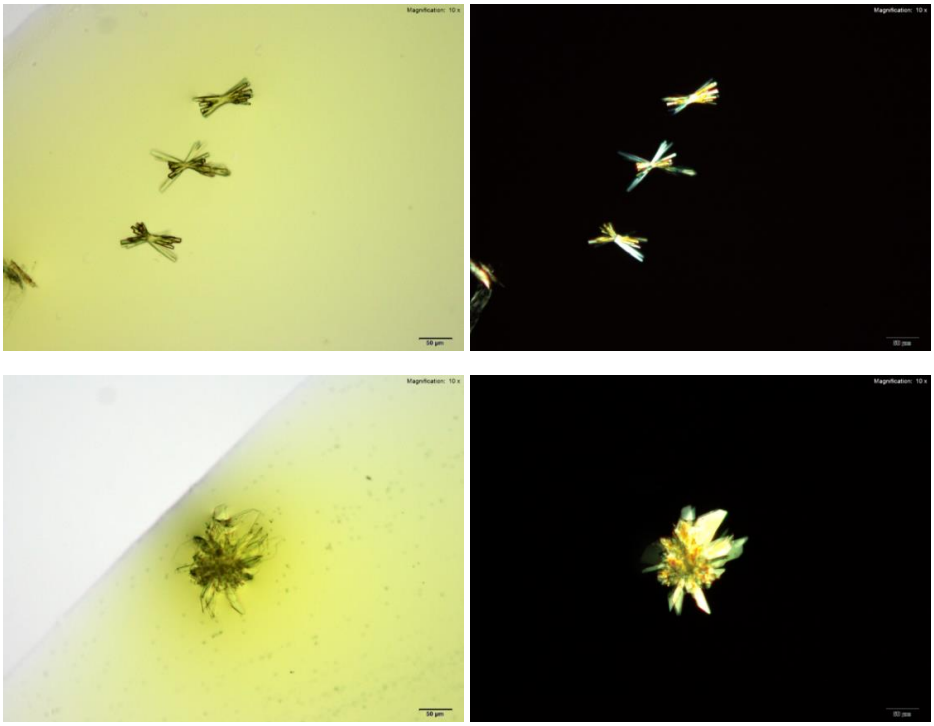
1. 3,4-Methylenedioxypyrovalerone (3,4-MDPV)



3,4-MDPV is one of the early generations of cathinones and has been studied extensively. It is a recreational substance that has stimulant properties but has also shown undesirable and often harmful physiological and behavioral side-affects. It is currently a DEA Schedule I controlled substance. Because of its structural similarity to 3,4-methylenedioxymethamphamine (MDMA), a phenyethylamine and a stimulant, 3,4-MDPV can also be considered a phenethylamine. It is generally found in powder form and ingested either orally or through the nasal passages.

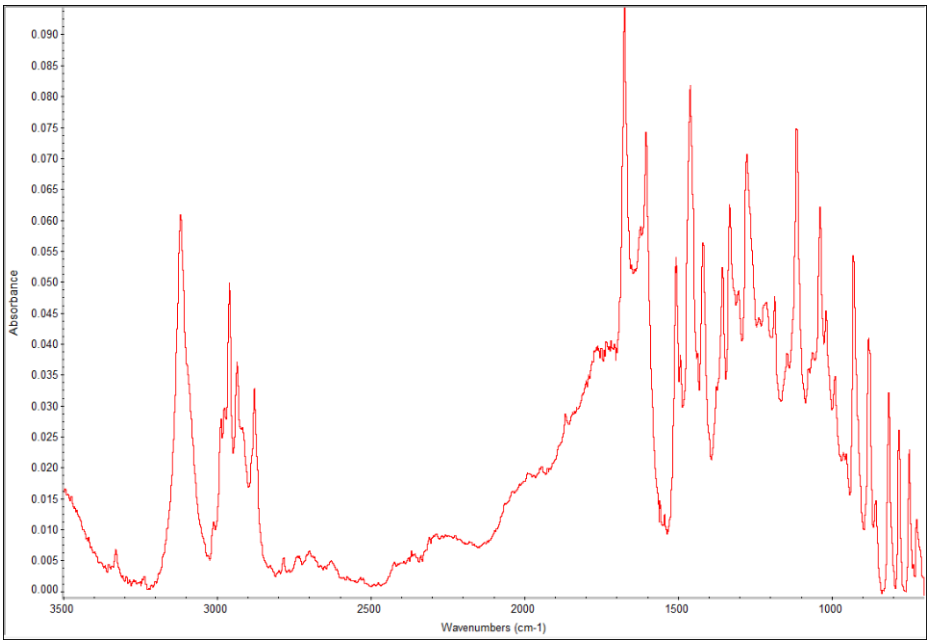
Additional reading:
DEA Fact Sheet- MDPV https://www.deadiversion.usdoj.gov/drug_chem_info/mdpv.pdf
WHO Expert Committee on Drug Dependence, 3, 4-MDPV Critical Review Report, 2014.
https://www.who.int/medicines/areas/quality_safety/4_13_Review.pdf
SWGDRUG Monograph http://www.swgdrug.org/Monographs/3,4MDPV.pdf
SOFT Monograph: https://www.soft-tox.org/files/designer_drugs/MDPV.pdf

5% Aqueous $HAuCl_4$

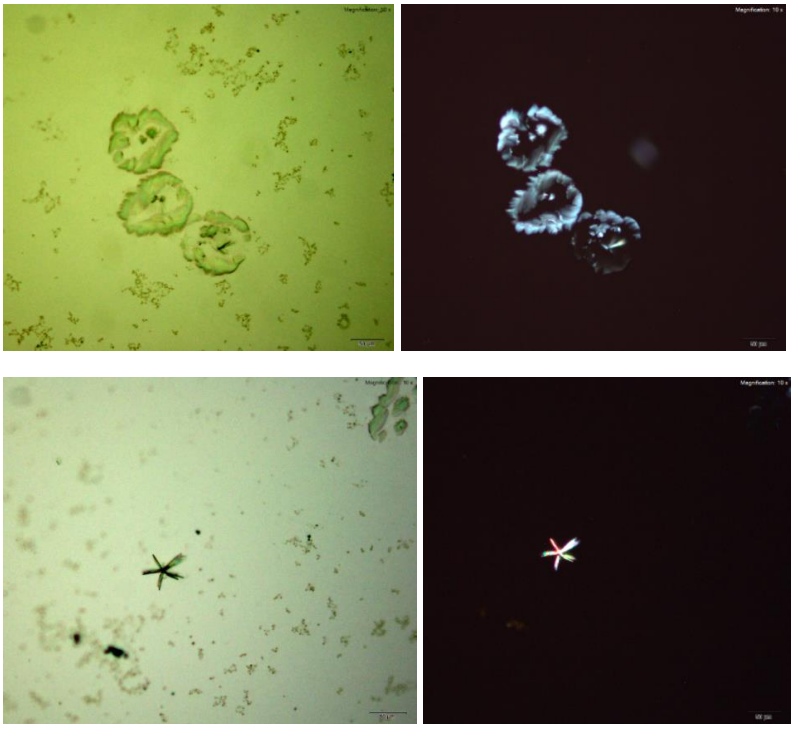


3,4- MDPV forms two types of crystals with aqueous gold chloride reagent 1) sheaves of short rods that are bright under crossed polars 2) tight clusters of plates and with angled edges. These features are better defined under crossed polars. The microcrystals that are clusters of thick plates offer good

maneuverability for infrared spectroscopy and the resulting transmission spectra exhibit spectral features that are a match for the pure substance.

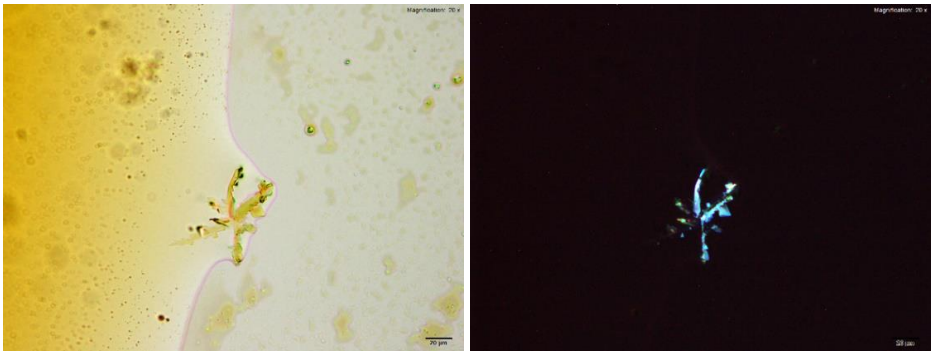


5% $HAuCl_4$ in 1:2 Concentrated H_2SO_4: H_20

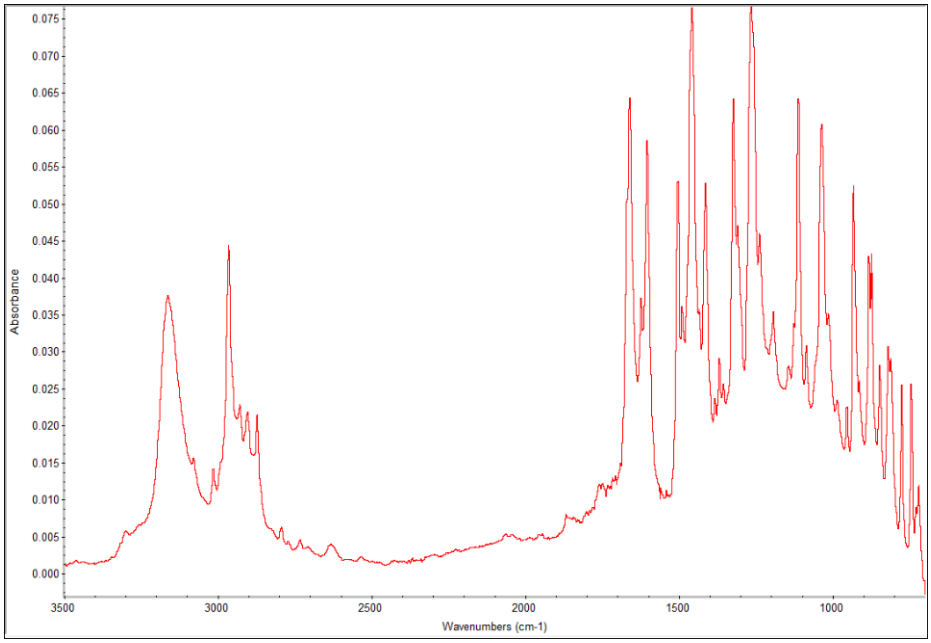


With this reagent, 3,4-MDPV forms a light leafy skin on the surface of the drop. This leafy growth is best visualized under crossed polars. At high concentrations of 3,4-MDPV, under this surface leafy formation, insect like clusters are also observed. This reagent and the observed crystal formation is not characteristic of 3,4-MDPV and therefore is not suitable test.

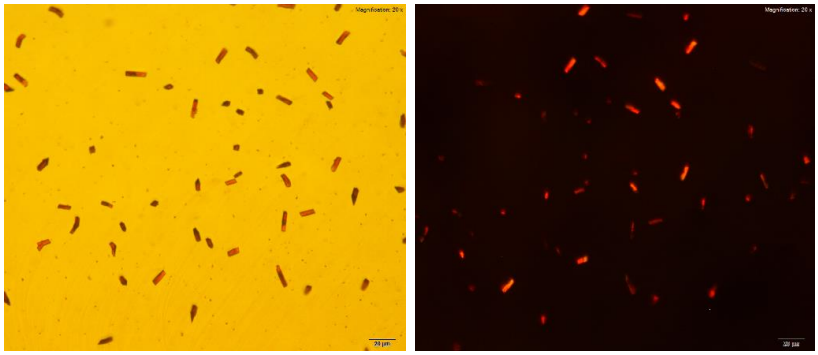
Gold bromide ($HAuBr_4$)



3,4-MDPV characteristic X-shaped clusters of stacked flat plates at the edges of the drops that appear bluish white under crossed polars. The clusters are friable and are do not yield themselves well for IR spectroscopy. However, when gathered together, the IR spectrum for the crystals is characteristic of 3,4-MDPV.



Platinic Bromide (H_2PtBr_6)

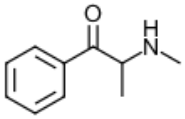


3,4-MDPV forms two crystal forms with the platinic bromide reagent. Both crystal forms are observed simultaneously and are shown in the first image. Thin, flat, squarish plates with angular ends are suspended in the drop. They may appear as needles or blades when oriented on their sides. They are bright under crossed polars and appear as orange, yellow or green plates. The second form of crystal are small, thick rectangular twinned tablets that are formed on the surface of the glass slide. They appear a deep orange under crossed polars. Both forms of crystals cannot be easily isolated from the reagent for IR microspectroscopy.

Overall recommended test:

Aqueous gold chloride reagent followed by infrared microspectroscopy of the resulting crystal.

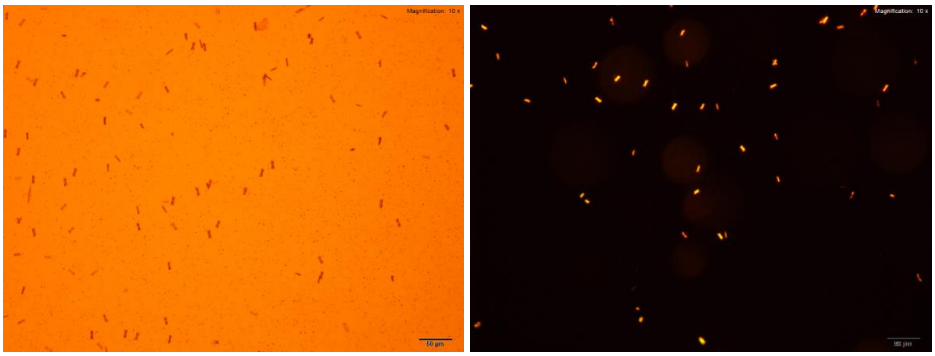
2. Methcathinone



Methcathinone, also known as ephedrone, is one of the earliest cathinones. Structurally it is beta-keto - methamphetamine and is a schedule I controlled substance. The derivates of methcathinone are more popular and methcathinone by itself is currently not encountered often in street drugs.

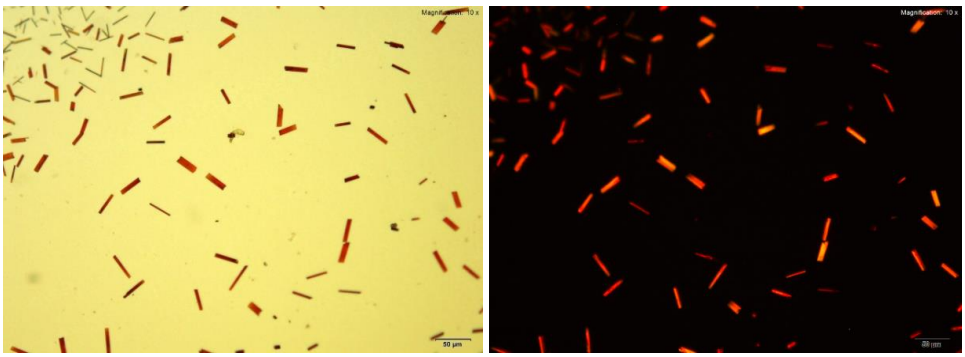
Additional reading:
https://drugs-forum.com/wiki/Methcathinone

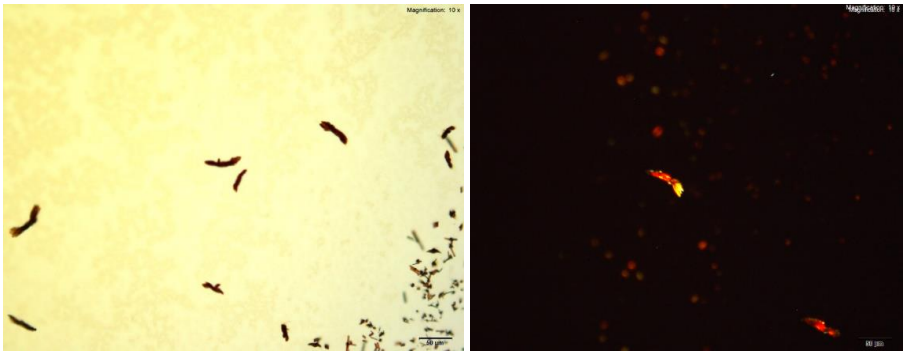
Gold bromide ($HAuBr_4$)



Methcathinone forms small orange x-shaped crystals with gold bromide reagent. The crystals grow abundantly even at 10 μg of methcathinone. The crystals are bright orange under crossed polars. However, magnification at 200x or greater is needed to observe the characteristic features.

Platinic Bromide (H_2PtBr_6)



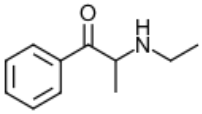


Two forms of crystals are observed for methcathinone with platinic bromide reagent. The first formed crystals are the abundant orange rectangular flat plates. Tight sheaves of short red needles form more slowly alongside the flat plates and both forms of crystals can be seen after 5 minutes of crystal growth. Both crystal forms are bright orange under crossed polars.

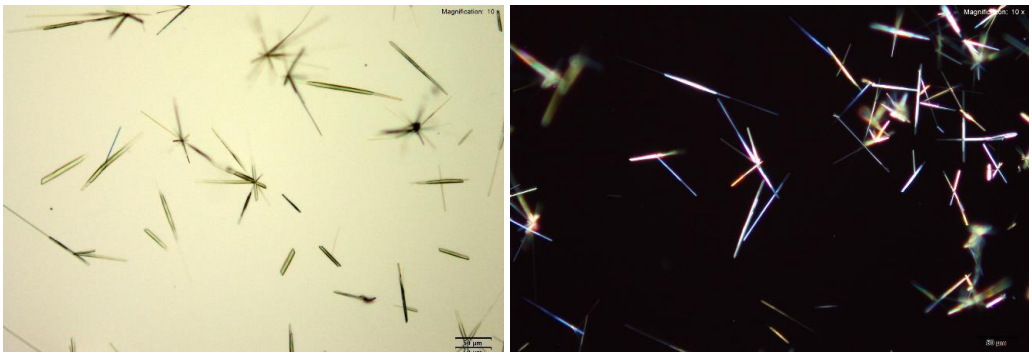
Overall recommended test:

The microcrystals formed by methcathinone are very small and difficult to study by IR microspectroscopy. For a standalone microcrystal test, either reagent may be used.

3. Ethcathinone

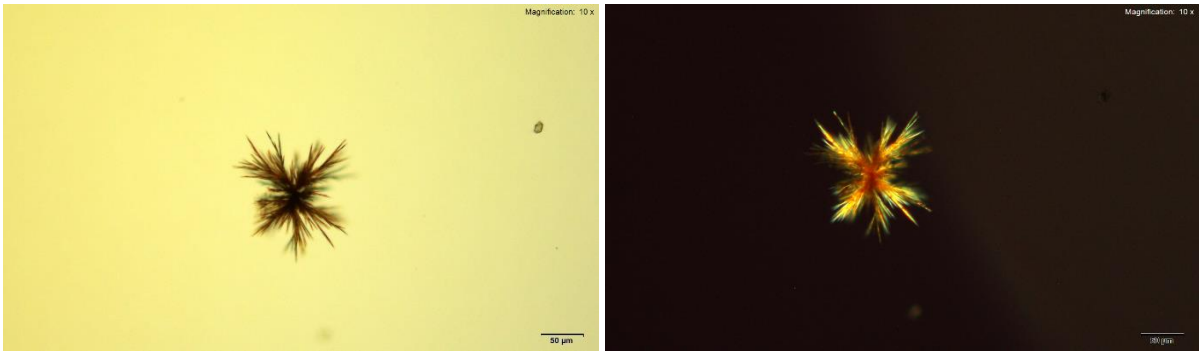


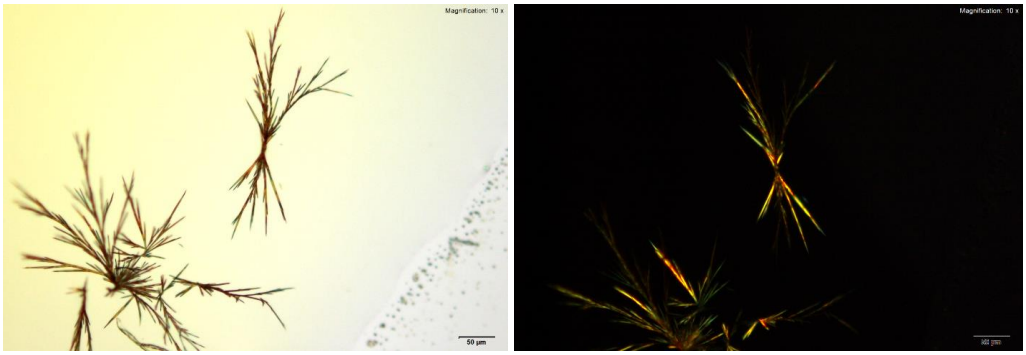
5% $HAuCl_4$ in 1:2 Concentrated H_2SO_4: H_20



In the acidic gold chloride reagent, ethcathinone, at amounts greater than 20 μg, slowly forms flat, pale yellow, straw-like crystals. The crystals grow either individually or in clusters. They are very bright under crossed polars.

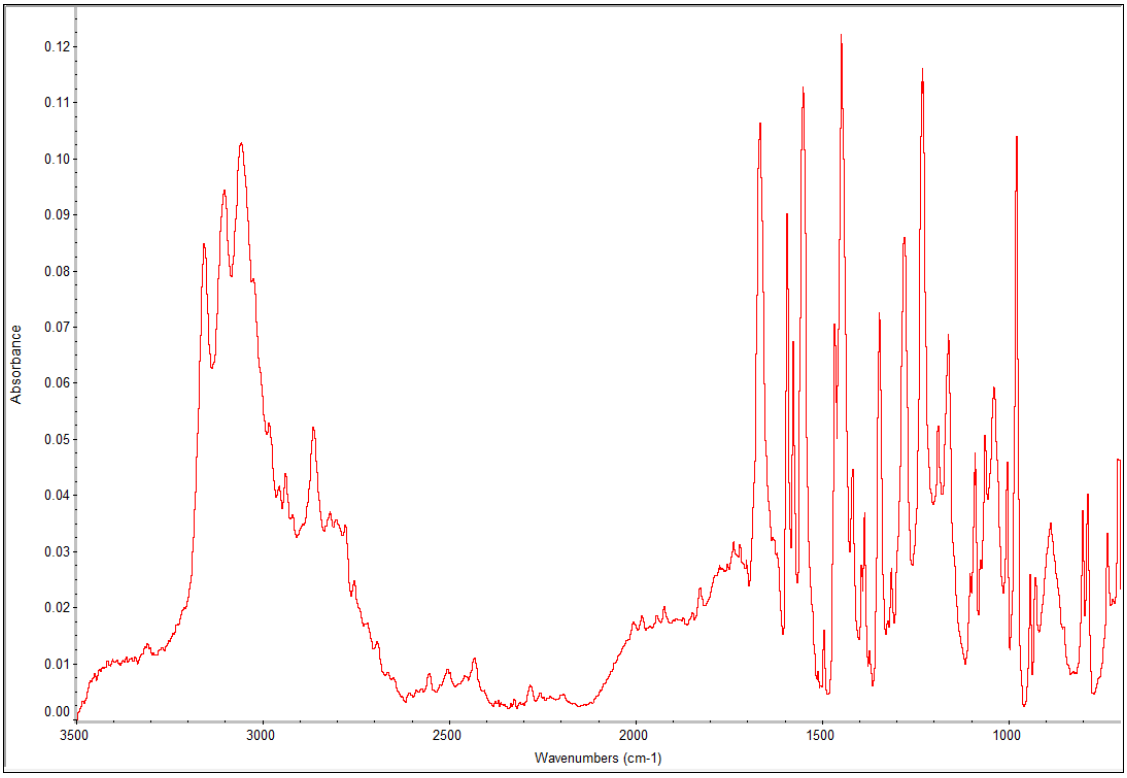
Platinic Bromide (H_2PtBr_6)





Distinct sheaves and dendrites of red needles are observed for ethcathinone and the platinic bromide reagent. The crystals could appear as tight clusters or loose large sheaves. Both habits are bright under crossed polars. With time (10 minutes), the crystal growth covers the entire drop even at amounts lower than 20 μg.

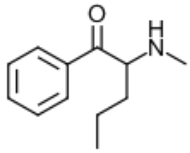
The abundant crystal growth makes this test suitable for IR spectroscopy. Information rich spectra that have spectral features consistent with the pure ethcathinone are obtained.



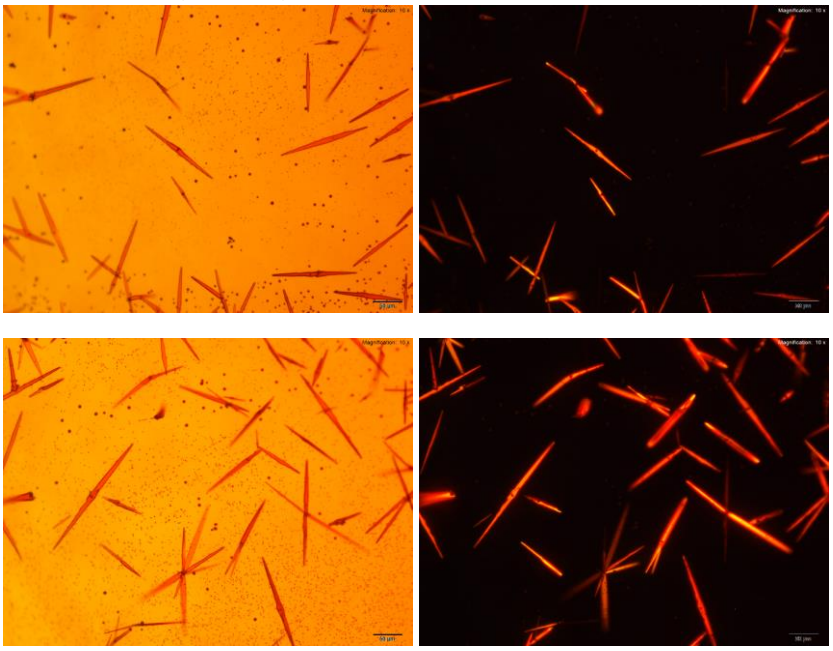
Overall recommended test:

The platinic bromide test is ideal for combined microcrystalline test and IR microspectroscopy

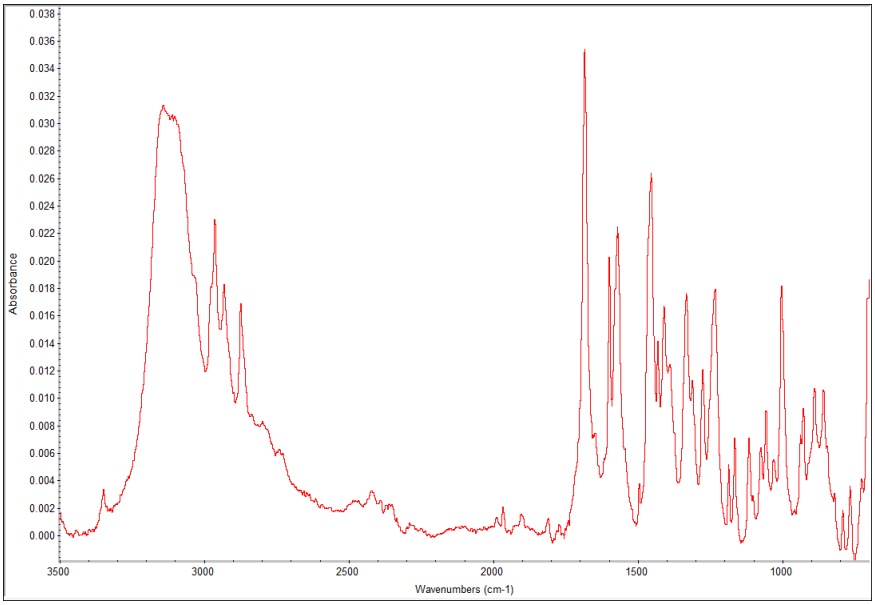
4. Pentedrone



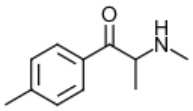
Gold bromide (HAuBr4)



Pentedrone reliably forms characteristic orange red spindles that are bright red under crossed polars with the gold bromide reagent. The crystals form abundantly and grow on the surface of the glass slide. They are difficult to displace from the slide for IR microspectroscopy but when done successfully, the resulting spectra are information-rich.

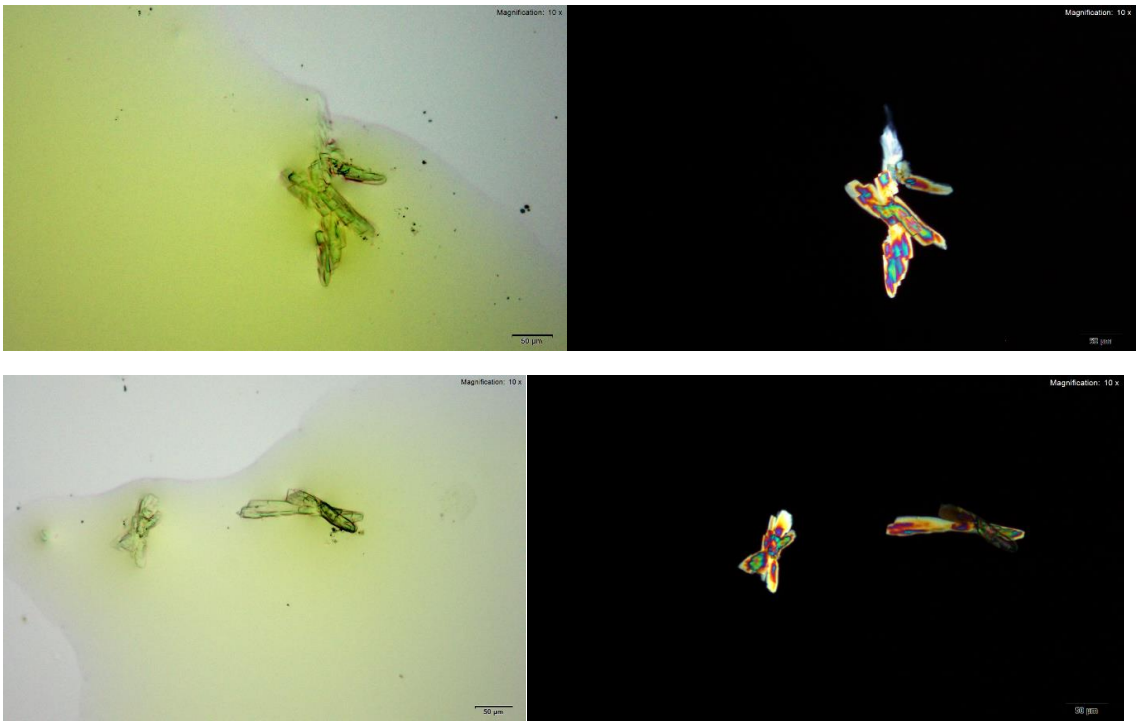


5. Mephedrone



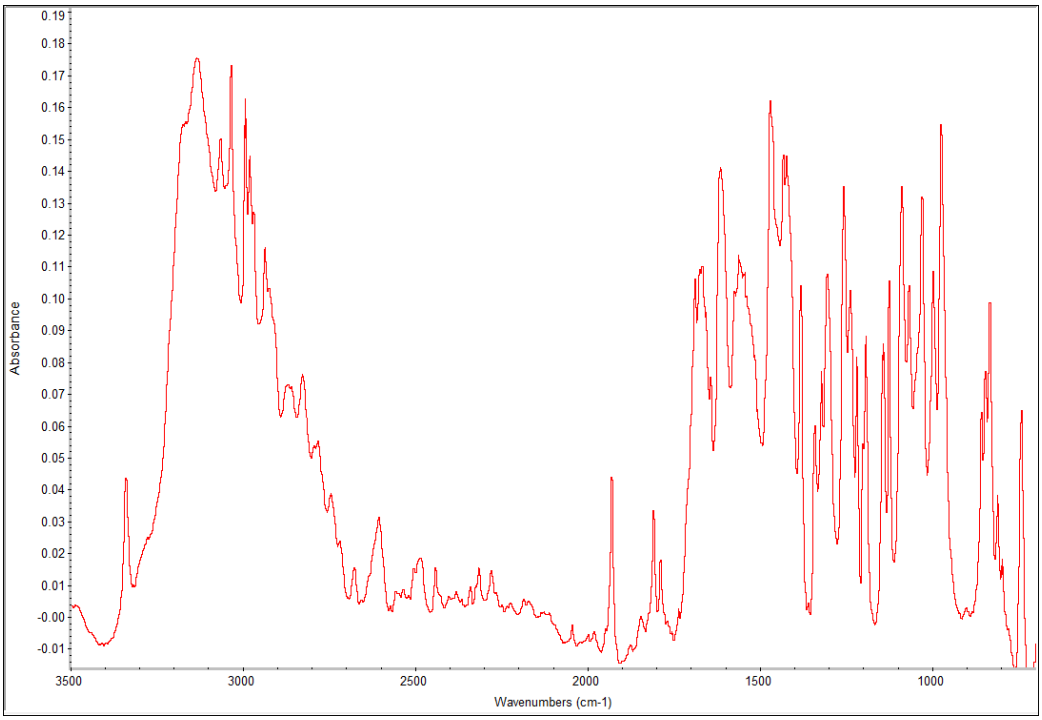
Additional reading:
SOFT Monograph https://www.soft-tox.org/files/designer_drugs/Mephedrone.pdf
SWGDRUG Monograph http://www.swgdrug.org/Monographs/4-methylmethcathinone.pdf

5% Aqueous $HAuCl_4$

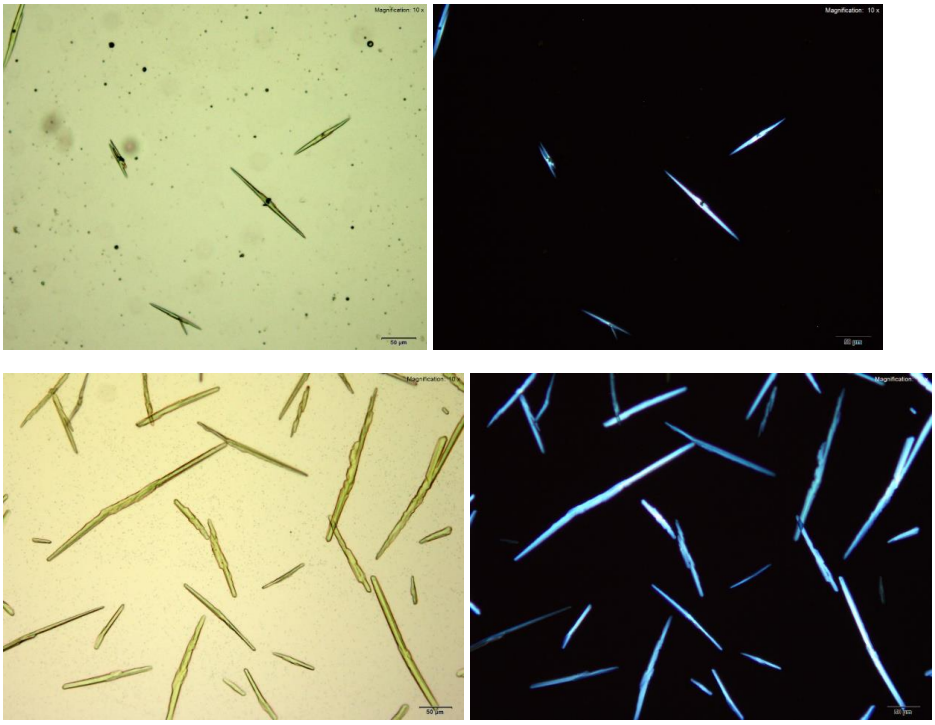


Mephedrone in aqueous and acidic solutions gives composites of thick, irregular tablets. These crystals are very bright under crossed polars with different parts of the composite going extinct at different times on rotation of the stage.

The size and thickness of the crystals makes them suitable for infrared microspectroscopy. The spectra obtained in transmission mode are information rich. They are representative of the mephedrone molecular structure while accounting for the shifts caused by mephedrone interaction with reagent.

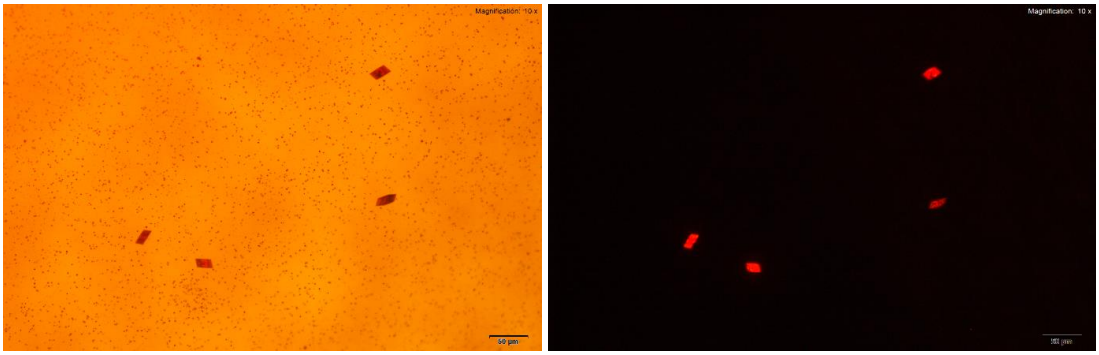


5% $HAuCl_4$ in 1:2 Concentrated H_2SO_4: H_20



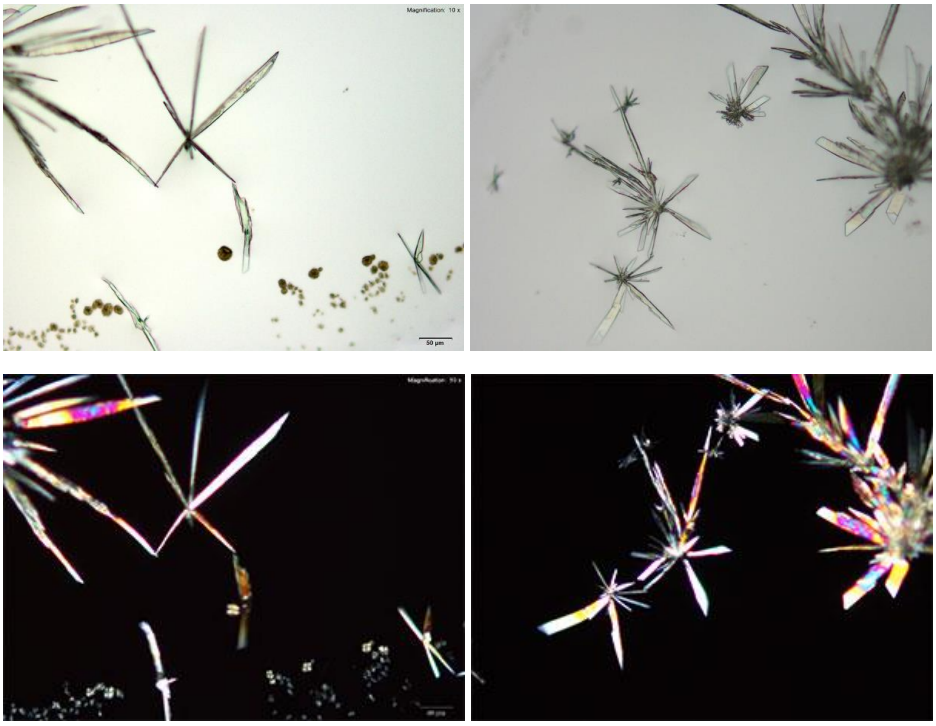
Long flat blades growing individually with sharp growing ends are observed for mephedrone with acidic gold chloride reagent. New blades grow from the growing end and after 10-20 mins of crystal formation time, the crystals appear as thick rods that are stacked end to end. All the crystals are distinctly blue under crossed polars.

Gold bromide ($HAuBr_4$)



In gold bromide reagent, mephedrone grows very short rod like prisms. The crystals are very small (10 µm) and take long to first appear (~20 mins) but are very distinct. They are similar to the crystals observed for butylone with gold bromide reagent.

Platinic Chloride (H_2PtCl_6)

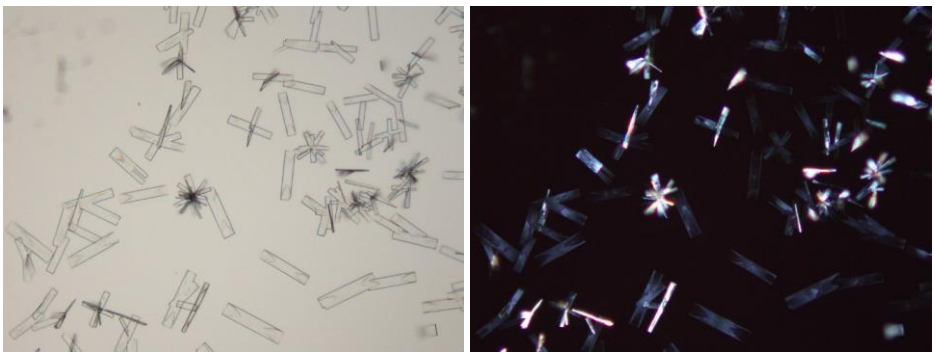


Mephedrone in aqueous and acidic solutions gives fans and rosettes of colorless, long and flat blades. The crystals grow abundantly and the best forms are observed in aqueous and 10% hydrochloric acid solutions. Another form of crystals that grow simultaneously are tight burrs of short, dark needles. The individual features of the burrs are observed under higher magnifications. The burrs precede the blades.

Platinic Bromide (H_2PtBr_6)

Small (10-20 µm) yellow octahedral prisms are observed with platinic chloride reagent and mephedrone. The crystals take long to grow (~25 min) and therefore this test is not ideal for characterizing mephedrone. However, other cathinones do not give this crystal shape with platinic bromide.

Mercuric Chloride (HgCl2)

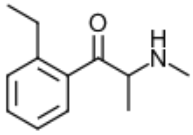


Mercuric chloride and mephedrone give characteristic colorless, rectangular flat plates that may grow individually or in clusters. These crystals grow quickly and require less than 10 µg of mephedrone.

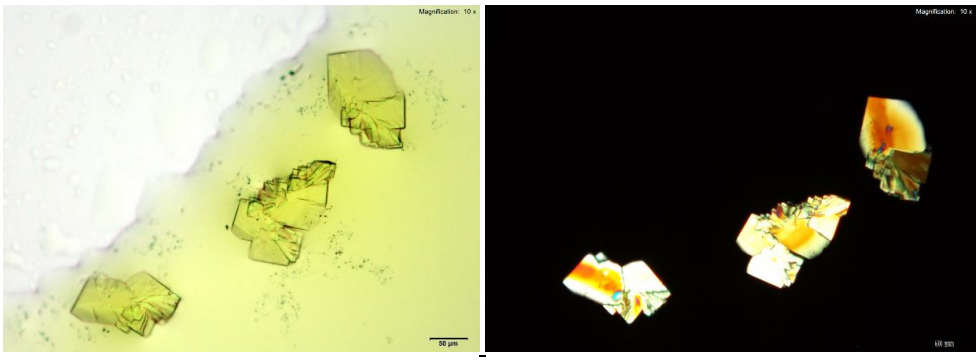
Overall recommended test:

The overall test for combined analysis of a crystal test with infrared microspectroscopy would be the 5% Aqueous $HAuCl_4$. The IR is information rich and can help determine if the chemical identity of the crystal though the shape of the crystal is not unique. For unique and easily recognizable crystals, the mercuric chloride test would be ideal.

6. 2-Ethylmethcathinone (2-EMC)

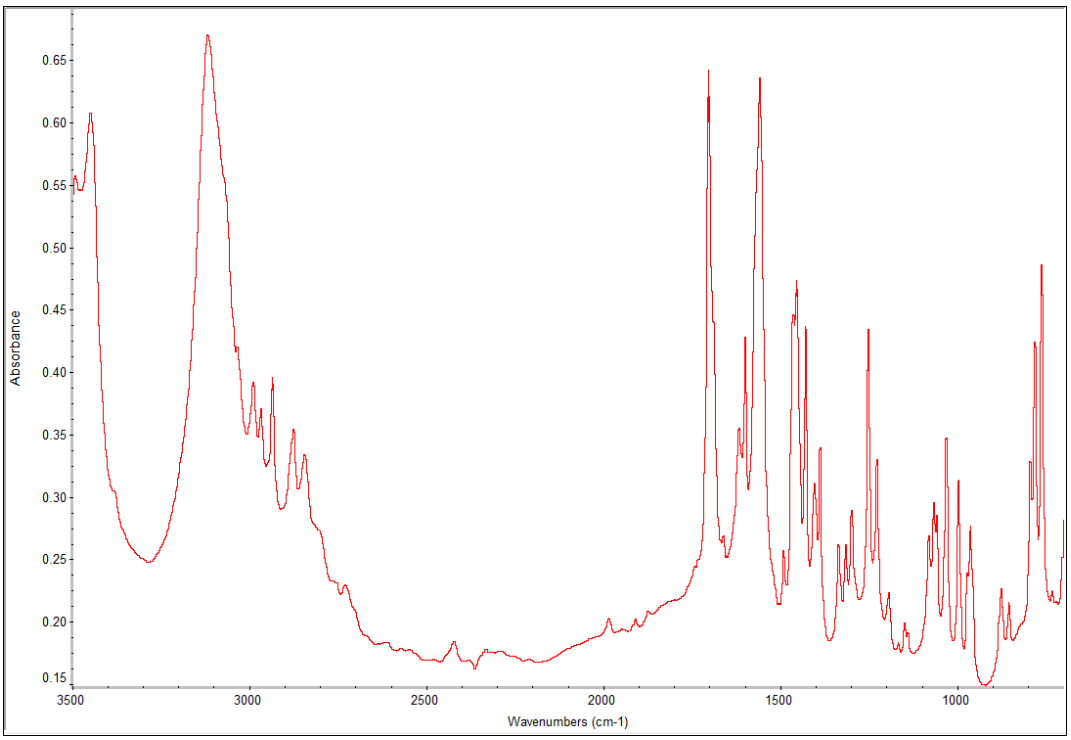


<u>5% Aqueous $HAuCl_4$</u>

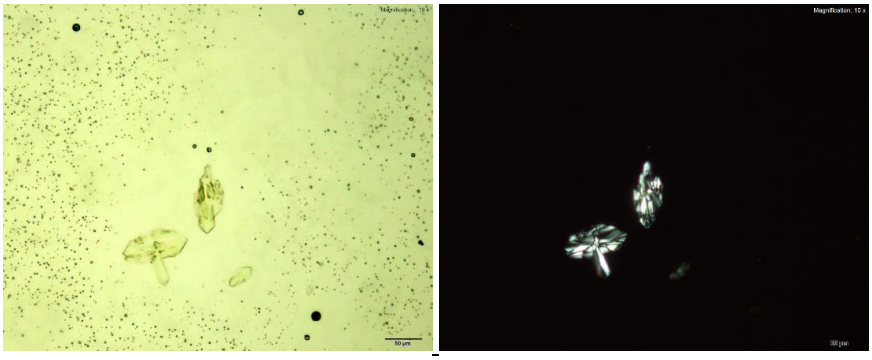


2-EMC with the aqueous gold chloride reagent gives characteristic large clear crystals. The crystals grow as large, thick tablets that are stacked. The crystals are very bright under crossed polars. 2- EMC residue (5 µg) can be dissolved in either water, 10% hydrochloric acid, or 10% acetic acid to achieve the described crystals.

The crystals are very well-suited for single crystal infrared spectroscopy. The easy to acquire IR spectra are information-rich with the crystal spectra being very similar to the drug standard.

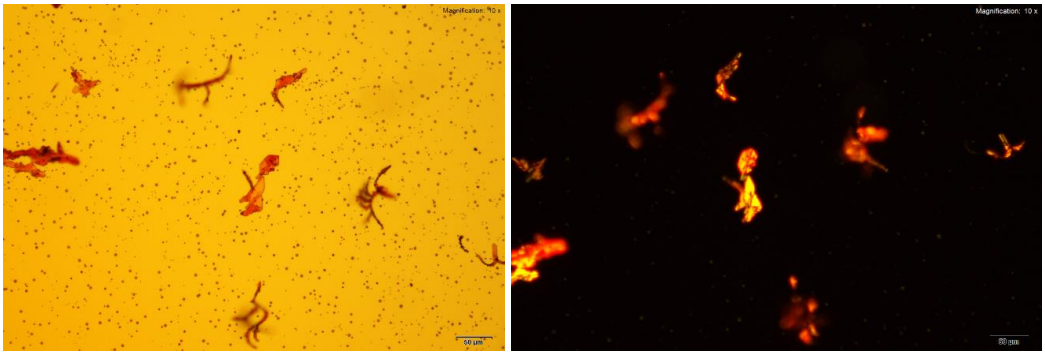


5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



2-EMC forms colorless prisms that form as twins. The drop initially oils very heavily and crystals form slowly (15-20 min) while clearing the oil. The crystals are not very bright under crossed polars and the edges of the crystals appear white against the dark background while the body of the crystals appear dark.

Gold bromide ($HAuBr_4$)

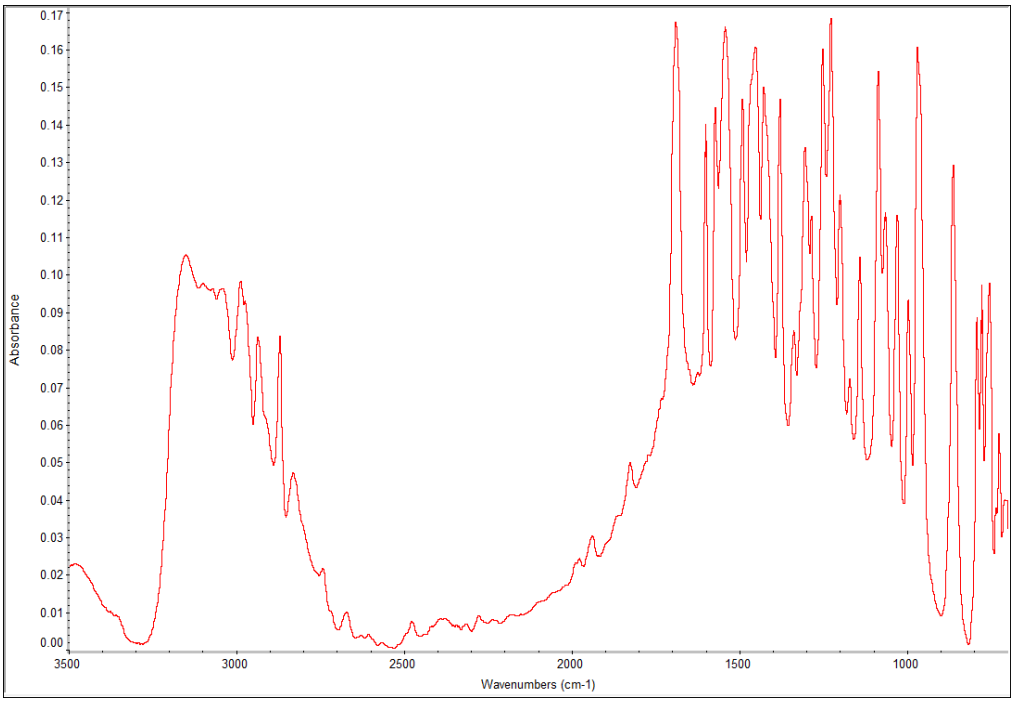


2-EMC with gold bromide grows characteristic red twig like growths. This shape is a composite of individual flat hexagonal plates that grow strung together in different orientations. This habit is more readily observed under higher magnifications. The individual plates appear bright in some orientations under crossed polars.

Platinic Chloride (H_2PtCl_6)

Under acidic conditions of 10% HCl, 2-EMC with aqueous platinic chloride forms thick, long, colorless prisms that grow end to end. The crystals display bright interference colors in crossed polars with different parts of the composite growing dark at different angles.

The crystals are very thick and are not ideal for transmission spectra but sufficient peak information can be obtained. Spectral quality is dependent on the crystal chosen for analysis.



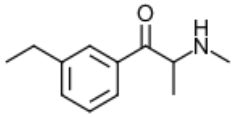
Platinic Bromide (H_2PtBr_6)

2-EMC forms crystals in two concentration dependent forms with platinic bromide reagent. The more common form of these crystals are the long blades with one angled edge. The crystal appears bright under crossed polars and goes extinct in some angles. The second form of crystals observed are sheaves of short red rods that are very bright under crossed polars.

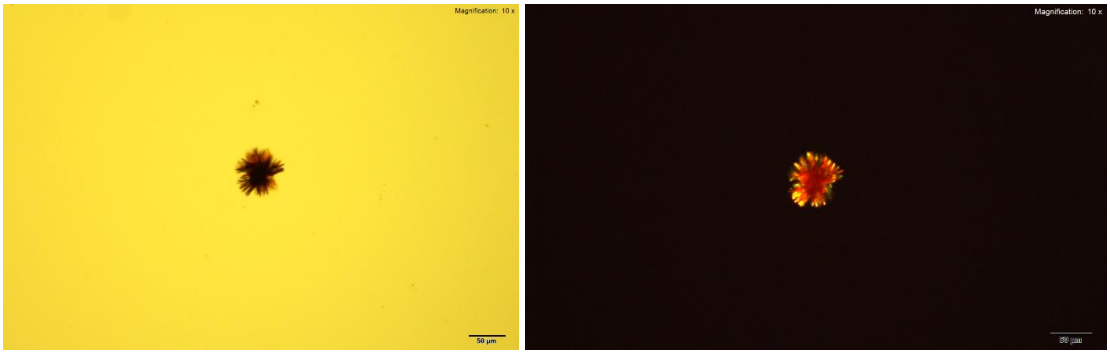
Overall recommended test:

The aqueous gold chloride reagent is the best reagent for both characteristic large 2-EMC microcrystals and infrared microspectroscopy.

7. 3-Ethylmethcathinone (3-EMC)



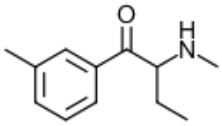
<u>Platinic Bromide (H_2PtBr_6)</u>



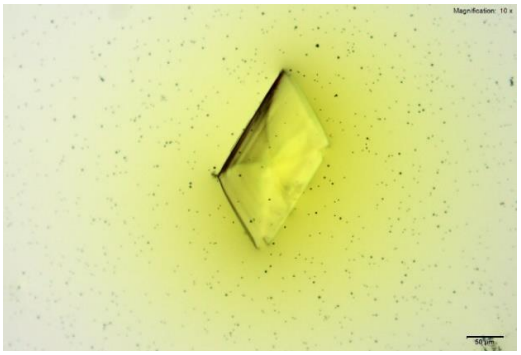
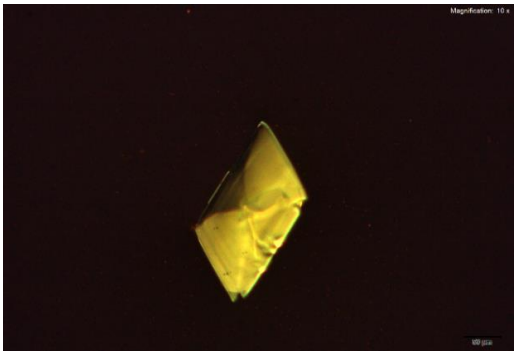
3-EMC forms tight clusters of short thin rods. The rods are red and very thin and look like needles. The needles are better visualized under higher magnifications. Under 100x magnification smaller clusters appear as dots. The clusters appear deep red spots under crossed polars.

3-EMC when combined with the other test reagents does not show crystal growth or when it does show some crystal formation, the crystals are not reproducible. Therefore, only one reagent is reported for 3-EMC.

8. 3-Methylbuphedrone

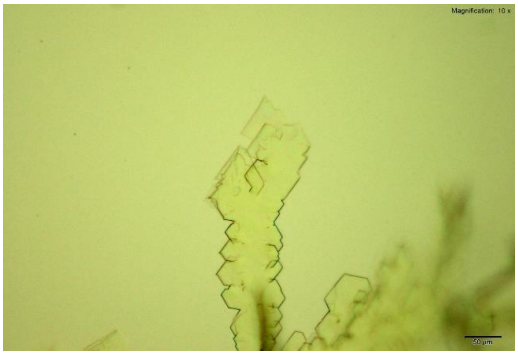
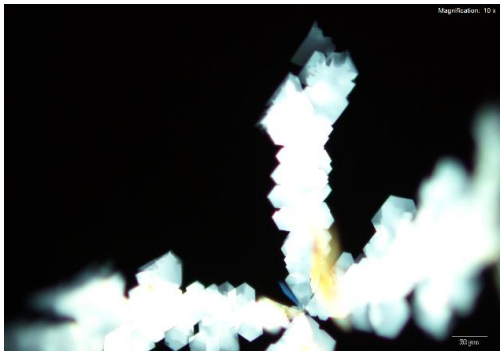


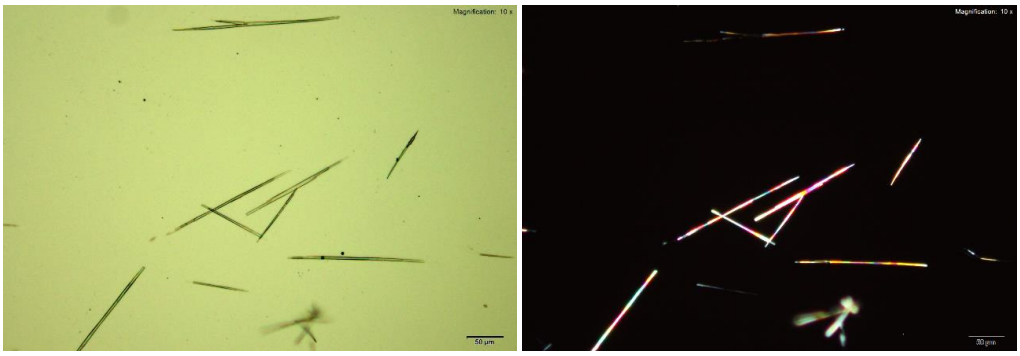
<u>5% Aqueous $HAuCl_4$</u>

Two forms of crystals are observed for 3-methylbuphedrone when mixed with aqueous gold chloride reagent. The substance, dissolved in water, 10% hydrochloric acid, or 10% acetic acid, gives yellow rectangular tablets when grown slowly at lower concentrations. The tablets when grown quickly may grow smaller and longer to appear as blades stacked on each other. Both forms appear very bright under crossed polars. The thick tablets do not give good IR spectra.

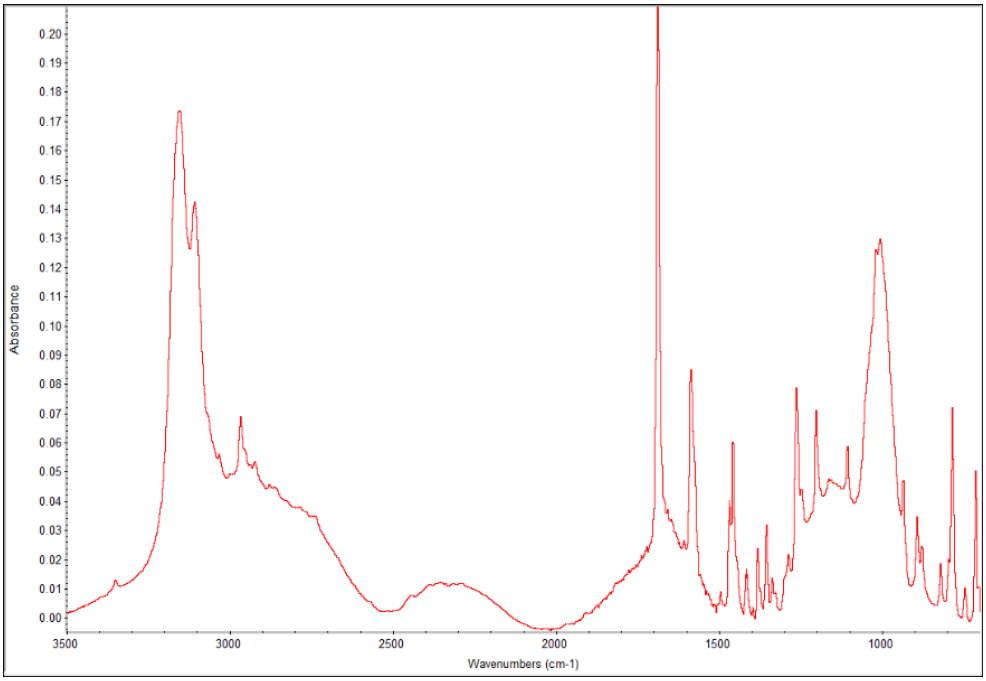
<u>5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20</u>

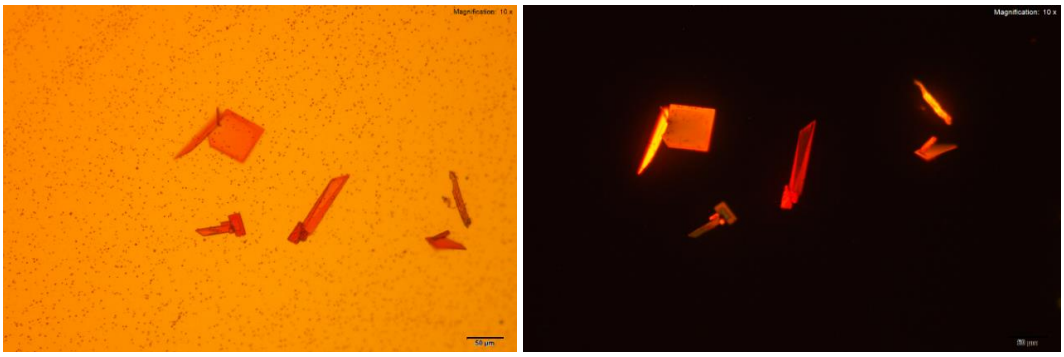


Two forms of crystals are observed with the acidic gold chloride reagent. The hexagonal plates are more prominent and grow end to end as large stacks. They appear bright white or bluish white under crossed polars. Flat thin blades that appear as needles when oriented on their sides are also observed. These are brightly colored under crossed polars.

The large flat hexagonal plates are suitable for IR microspectroscopy.

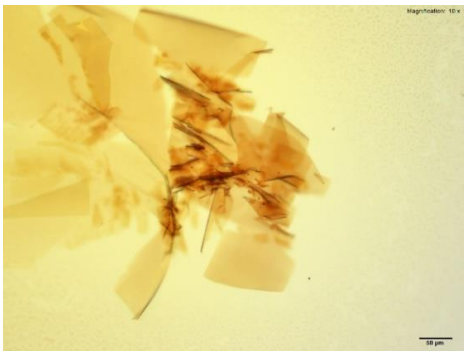
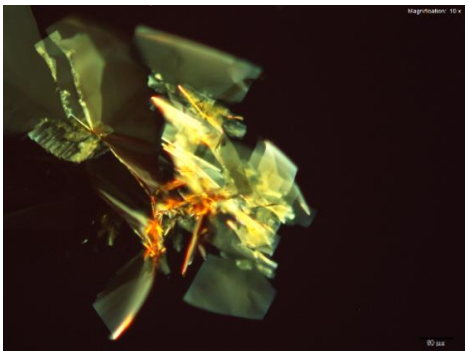
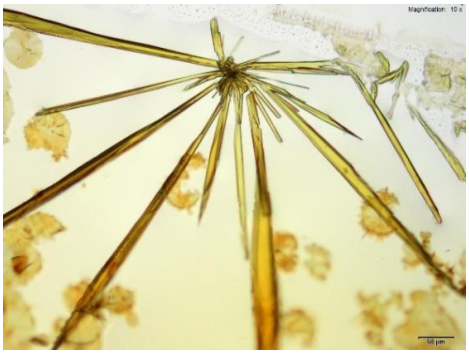


Gold bromide ($HAuBr_4$)



Flat orange square or rectangular plates are readily formed with gold bromide reagent. The thin plates are suspended in the drop in various orientations and are observed as blades, plates or needles. They appear bright orange under crossed polars.

Platinic Bromide (H_2PtBr_6)

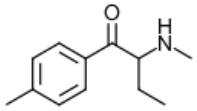





The first change seen when platinic bromide reagent is mixed with an aqueous solution of 3-Methylbuphedrone is the formation of thin floating skin. The skin is a bright greenish yellow under crossed polars. When the skin is disturbed, long blades arranged individually or in clusters are observed in the drop. The yellow blades appear very bright under crossed polars and are distinct from the skin.

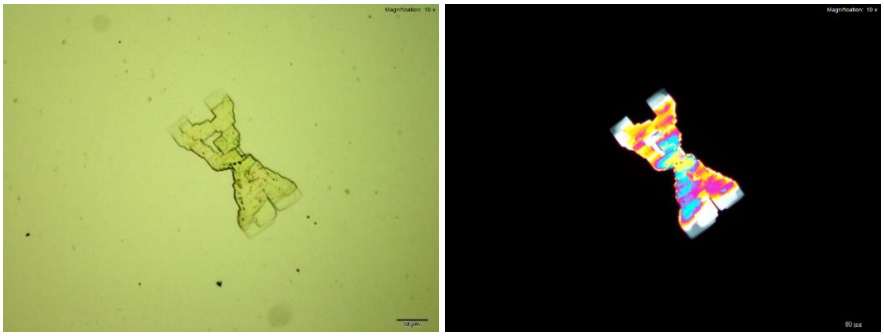
Overall recommended test:

The 5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20 reagent is recommended both for the characteristic microcrystals and the ability to maneuver them for IR spectroscopy.

9. 4-Methylbuphedrone

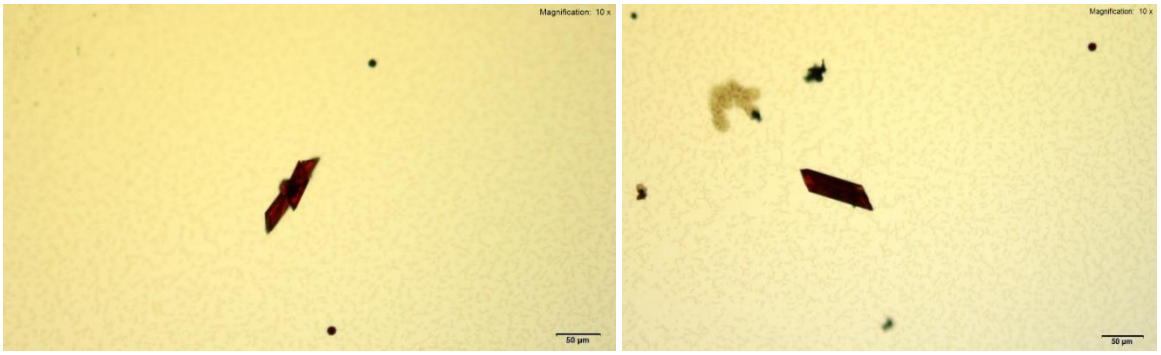


5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



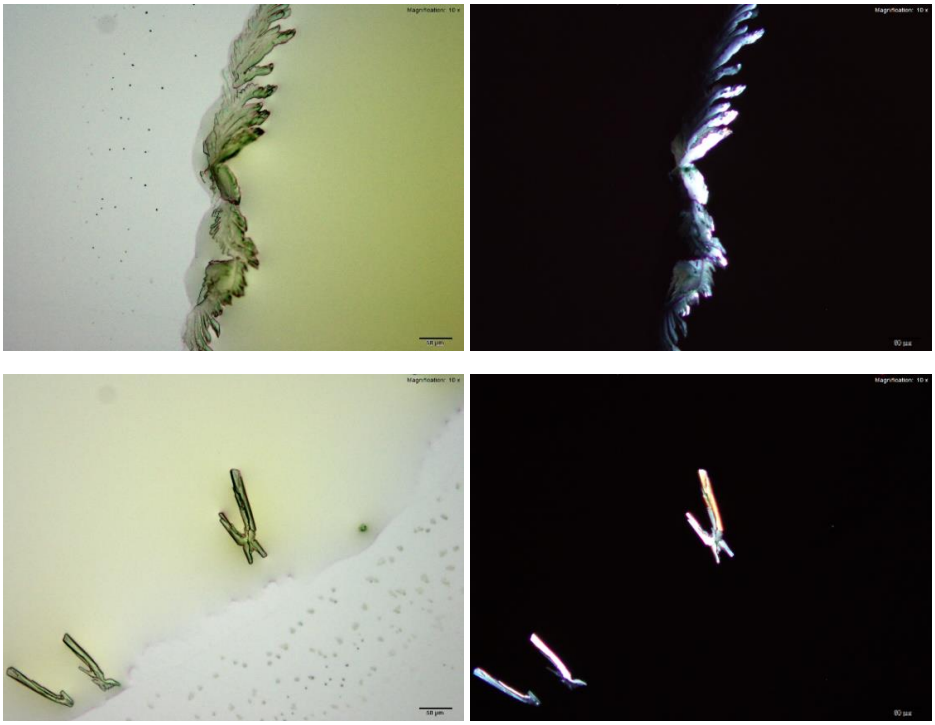
4-Methylbuphedrone forms colorless thick tablets that are arranged as X-shaped composites with the acidic gold chloride reagent. The overall formation is quite distinct. The composites are bright and multicolored under crossed polars. The composites, though large, are not easily maneuvered for IR spectroscopy because they fall apart into their individual thick tablets.

Gold bromide ($HAuBr_4$)

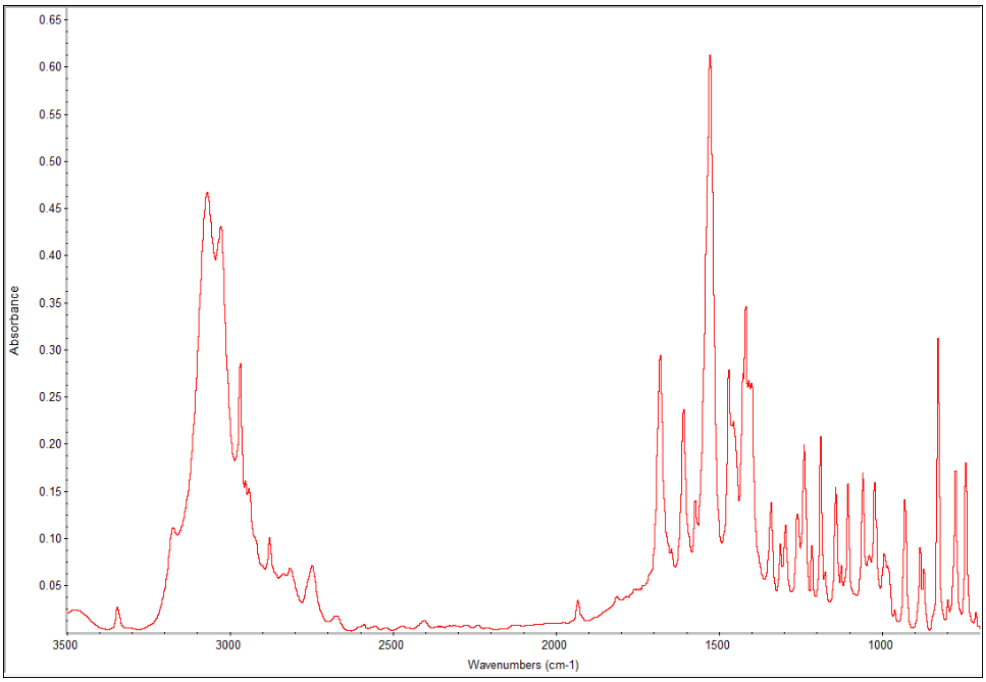


Gold bromide reagent gives distinct red rectangular tablets with 4-methylbuphedrone. The thick tablets grow individually or in pairs. The crystals take long to grow (~30 min) at 4-methylbyphedrone concentrations below 25 µg. The crystals appear bright red under cross polars and go completely extinct upon rotation of stage.

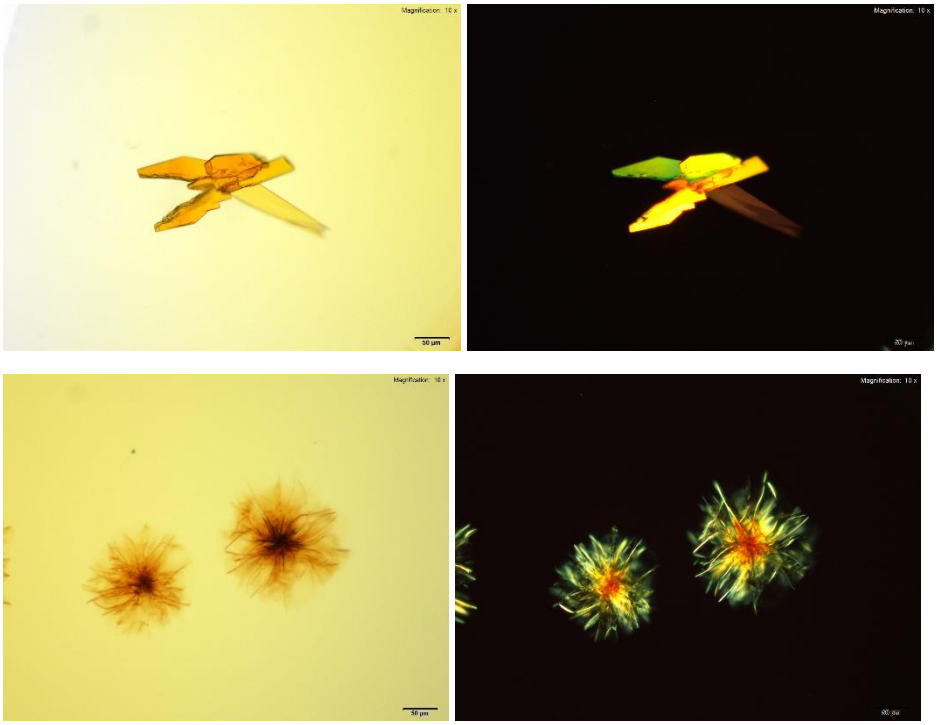
Platinic Chloride (H_2PtCl_6)



Yellow feathery crystals with serrated edges that grow as a crust slightly inside the reaction drop and composites of tablets are observed when 4-Methylbuphedrone dissolved in water or 10% acidic solutions is mixed with platinic chloride reagent. Crystals formed under aqueous are more characteristic and reproducible. The crystals are very bright under crossed polars. The crystals on the edge of the drop are amenable for IR spectroscopy and give information rich spectra.



Platinic Bromide (H_2PtBr_6)

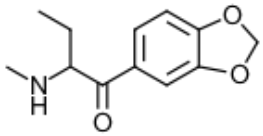


Platinic bromide reagent results in two distinct forms of crystals when mixed with platinic bromide reagent. The round, tight clusters of light, orange hairs are prominent. The centers of the clusters are very dark and the loose ends are bright under crossed polars. The composites of irregular thick yellow-orange tablets grow very large. These composites are very bright under crossed polars and different parts of the crystal go dark at different times. The crystals are very thick and do not give good IR spectra.

Overall recommended test:

For characteristic shapes crystal formations, the platinic bromide reagent is ideal. However, the crystals are not always suitable for IR spectroscopy. Flatter crystals that have grown sufficiently large but not overgrown are best for IR. For a combined test, the platinic chloride reagent is recommended.

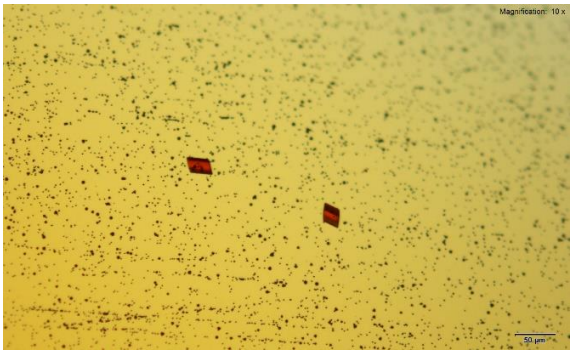
10. Butylone

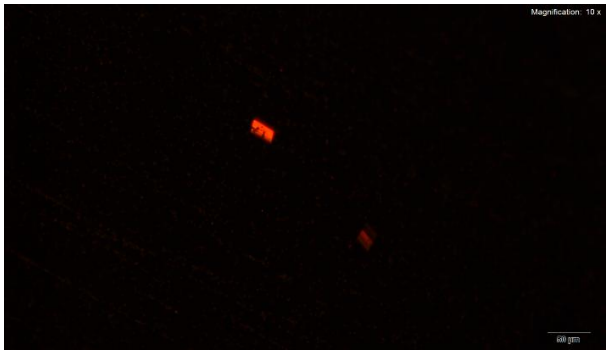


Butylone is a synthetic cathinone that is listed as a DEA Schedule I substance. It is commonly used in powder form and ingested orally or by snorting. It has commonly been observed as a mixture with its homology methylone rather than as a pure substance.

Additional reading:
Warrick et al., Lethal Serotonin Syndrome after Methylone and Butylone Ingestion, J Med Toxicol. March 8(1),65-68, 2012. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3550225/#Sec2title
DEA Special Testing and Research Laboratatory, Butylone Monograph, 2014, http://www.swgdrug.org/Monographs/butylone.pdf
SOFT Monograph: https://www.soft-tox.org/files/designer_drugs/Butylone.pdf

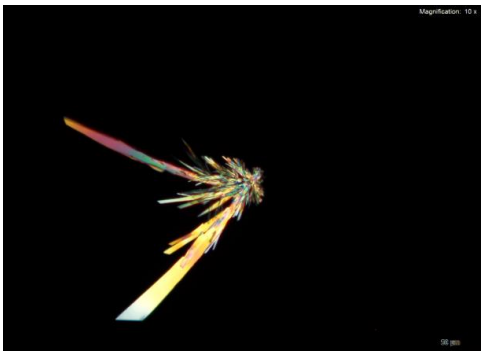
Gold Bromide ($HAuBr_4$)





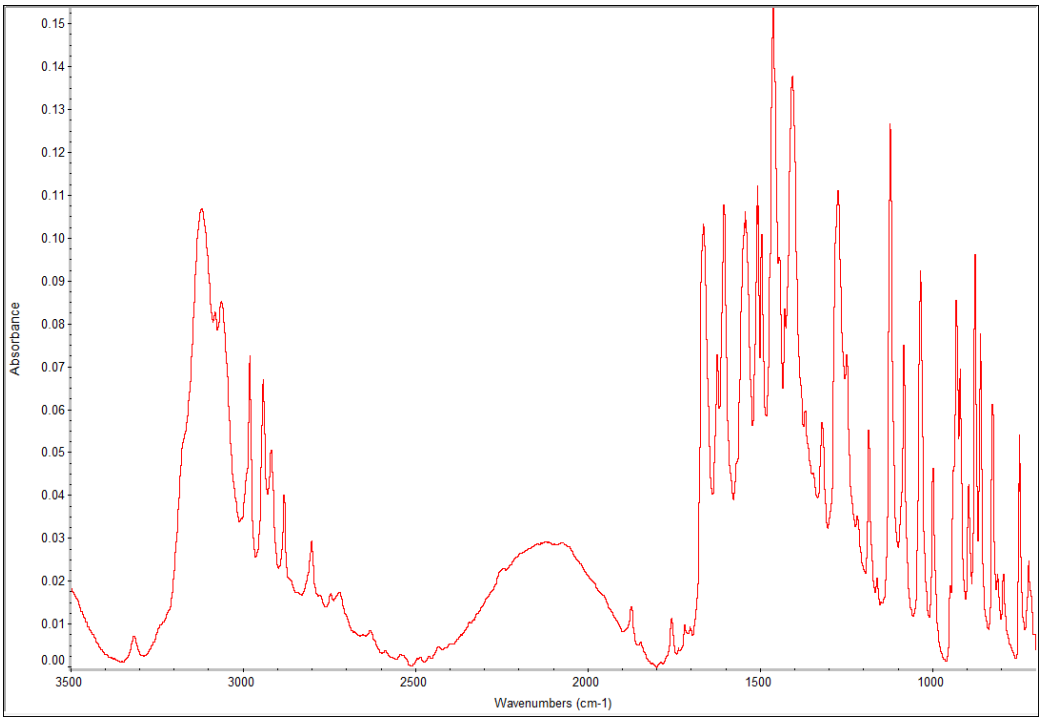
Butylone forms small (15-30 μm), red rod-like prisms that grown abundantly but very slowly (~15-20 min). Once crystal formation begins, the drop can easily be overgrown with several such prisms. The distinct red crystals are bright red under crossed polars and go completely extinct upon rotation of the stage.

Platinic Chloride (H_2PtCl_6)

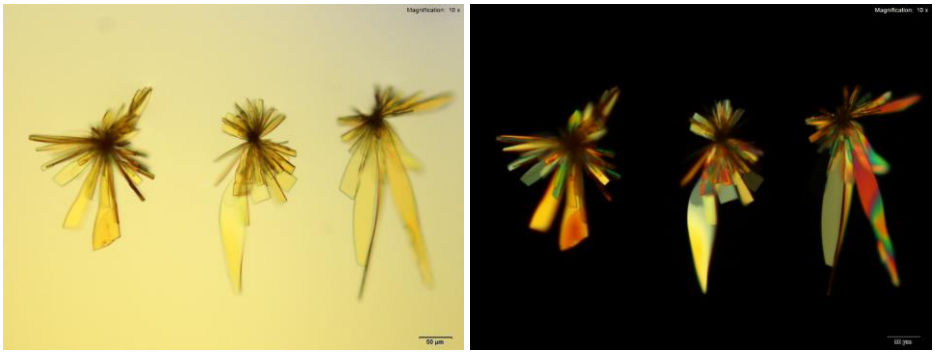


Platinum based reagents are the best reagents for butylone as the crystals are much larger than the gold bromide reagent crystals. The crystals formed with platinic chloride, in both aqueous and acidic solutions of butylone, are colorless tufts of long rectangular blades with shorter blades at the dense center of the tuft. The crystals are bright under crossed polars and exhibit a range of interference colors.

The crystals are easily studied with infrared microspectroscopy and give information rich spectra.



Platinic Bromide (H_2PtBr_6)

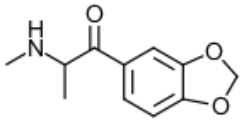


Butylone with the platinic bromide reagent forms large rosettes of yellow flat blades and tablets. The rosettes feature a few long blades with curved sides giving the rosettes a characteristic bow like appearance. The crystals are bright with various interference colors under crossed polars. The crystals grow abundantly within 10 minutes with butylone amounts as little as 5μg.

Overall recommended test:

Platinic chloride reagent with either aqueous or acidic solutions of butylone followed by infrared microspectroscopy of the resulting crystal.

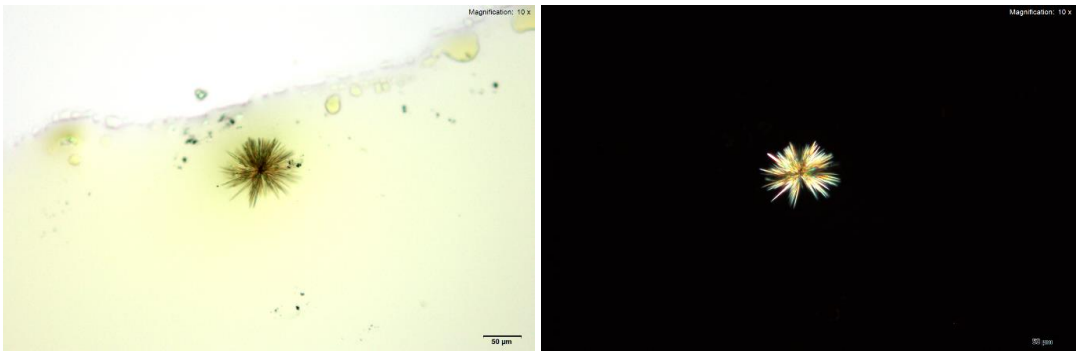
11. Methylone



Methylone is DEA Schedule I controlled substance. It is commonly used in powder form and ingested orally or by snorting. It has commonly been observed as a mixture with its homology methylone rather than as a pure substance.

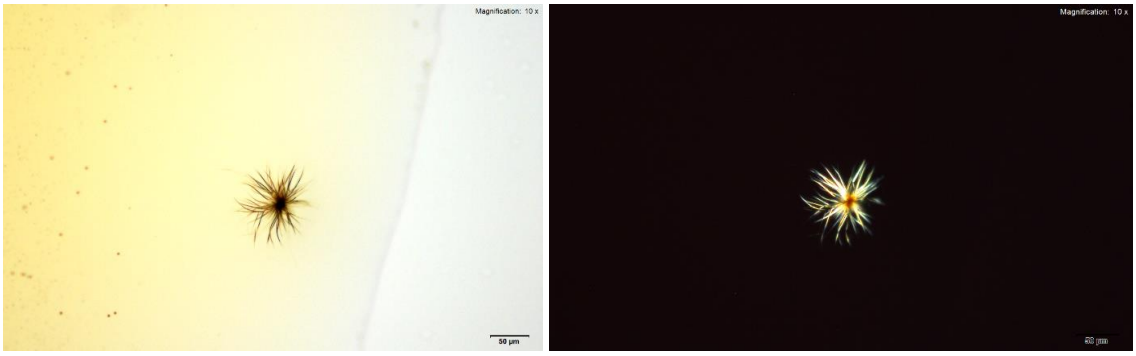
Additional reading:
Warrick et al., Lethal Serotonin Syndrome after Methylone and Butylone Ingestion, J Med Toxicol. March 8(1),65-68, 2012. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3550225/#Sec2title

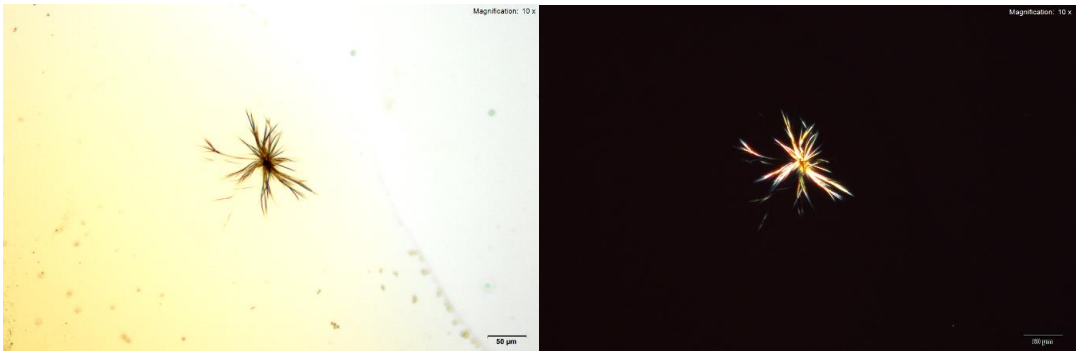
5% Aqueous $HAuCl_4$



Methylone in acidic (10% hydrochloric acid) solution when mixed with aqueous gold chloride reagent forms different sized burrs of colorless fine needles. The burrs appear dark in the center where the growth is dense. Under crossed polars, the burrs are very bright and appear yellow with extinct needles at 90°.

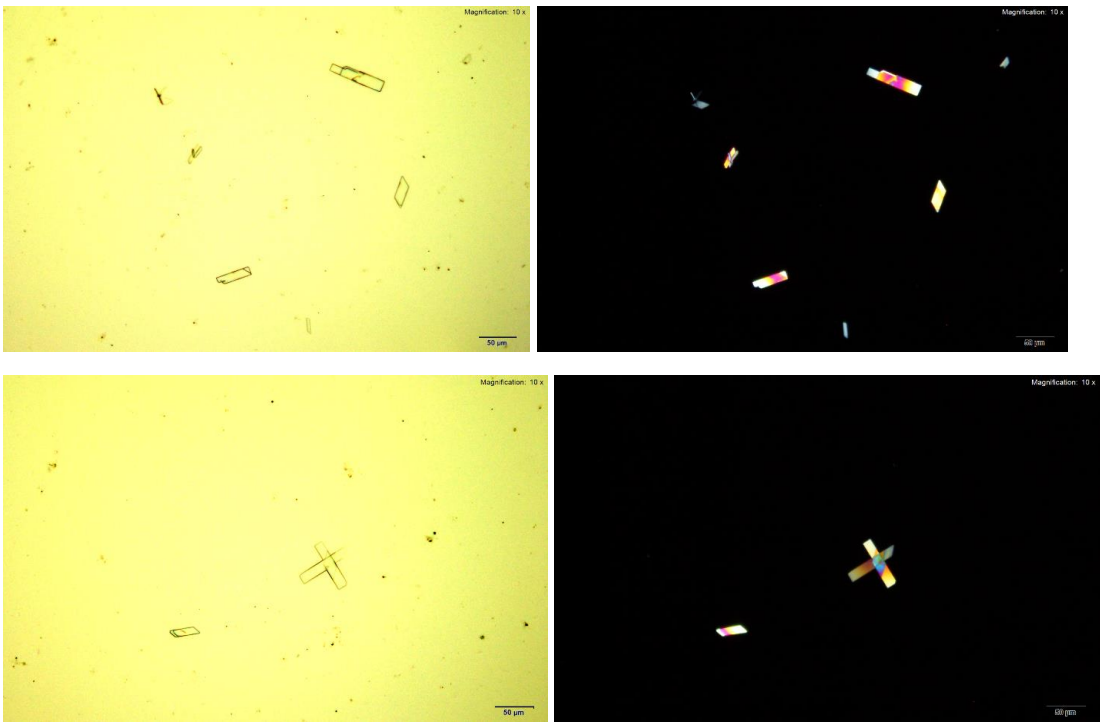
Gold bromide ($HAuBr_4$)





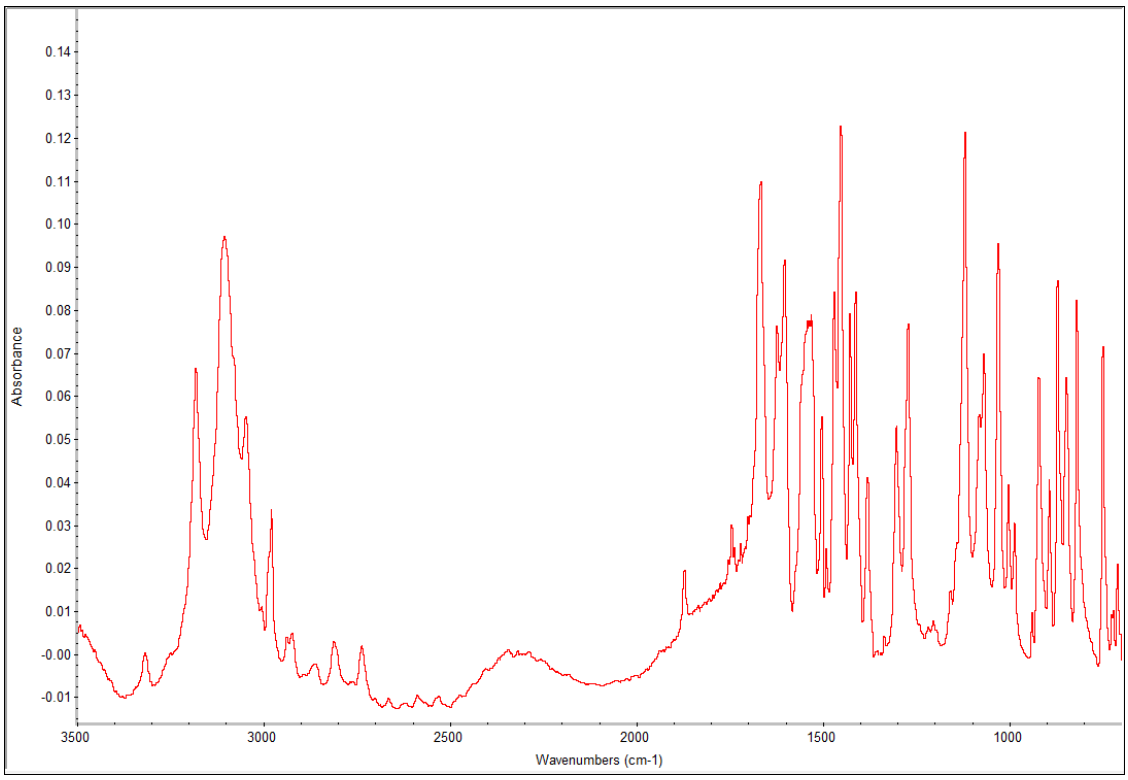
Methylone, in keeping with the crystal shape observed with gold-based reagents, forms needles with gold bromide reagent. The crystals are red, wispy, and are arranged in loose sheaves or rosettes. They range in size but are easily recognized as bright sheaves under crossed polars.

Platinic Chloride (H_2PtCl_6)



Methylone, as a 10% hydrochloric acid solution, with platinic chloride reagent forms colorless rectangular plates and tablets that may grow individually or in stacks. The crystals grow abundantly and in composites when methylone amounts are greater than 10 μg.

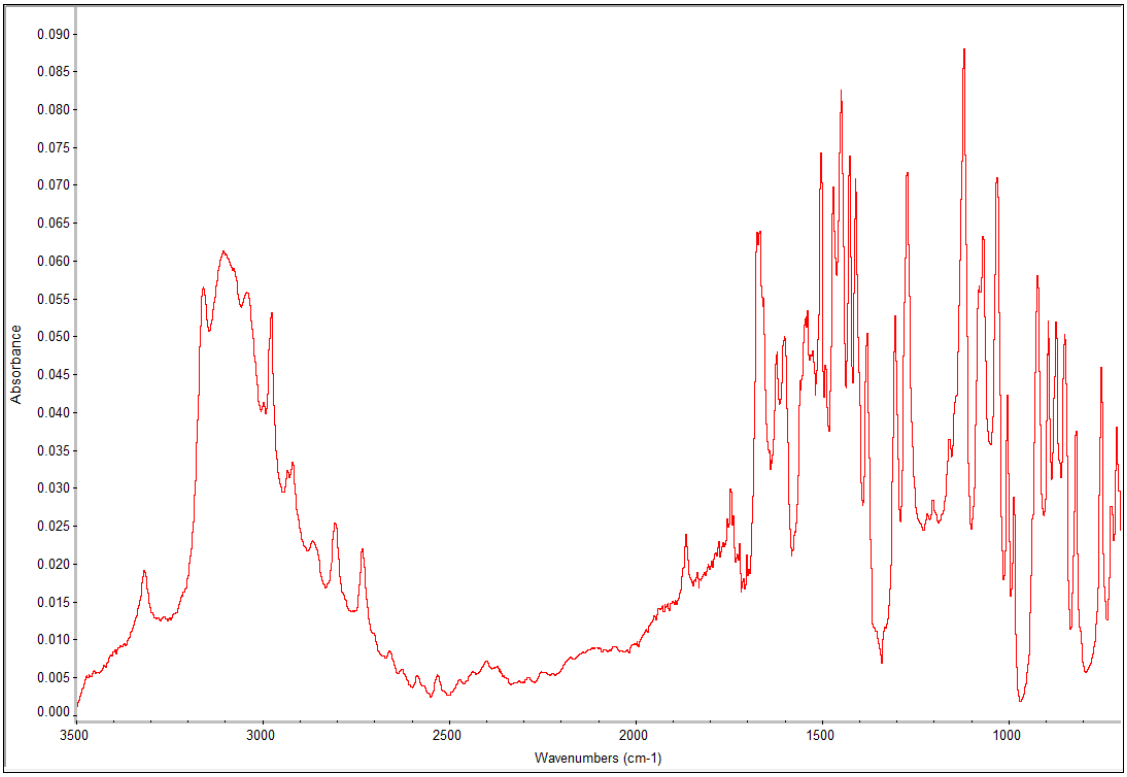
The crystals are amenable for IR microspectroscopy and yield information-rich spectra.



Platinic Bromide (H_2PtBr_6)

The crystals for methylone in platinic bromide reagent are thick, yellow prisms and tablets. The crystals are bright yellow under crossed polars with interference colors showing where the tablets are thick or stacked.

The tablets are easily studied by IR microspectroscopy. However, the thickness of the tablets causes distortions in the transmission IR spectrum. Crystals have to be chosen carefully to get spectra that are information-rich and consistent with methylone.

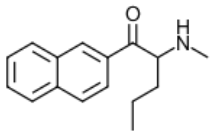


Overall recommended test:

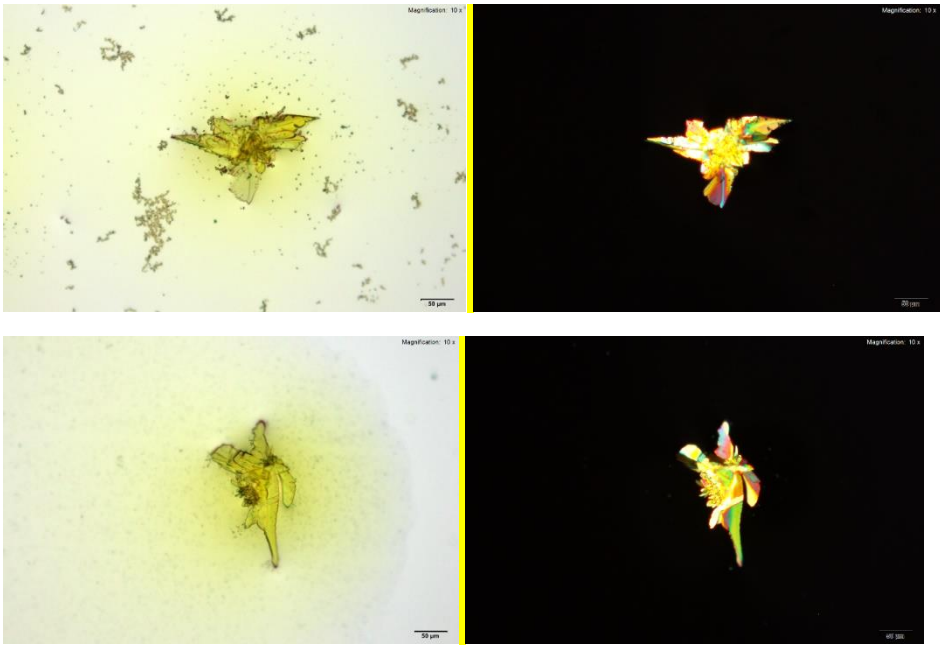
Platinum-based reagents are ideal for methylone. Both platinic chloride and bromide yield good crystals and information rich IR spectra. However, platinic chloride reagent is better for the combined ease of crystal growth and maneuvering the crystals for IR spectroscopy.

The homologs butylone and methylone, which are generally found in mixtures, can be differentiated from each other using a simple microcrystalline test using platinic chloride and the infrared spectra of each crystal also differ.

12. NRG-3

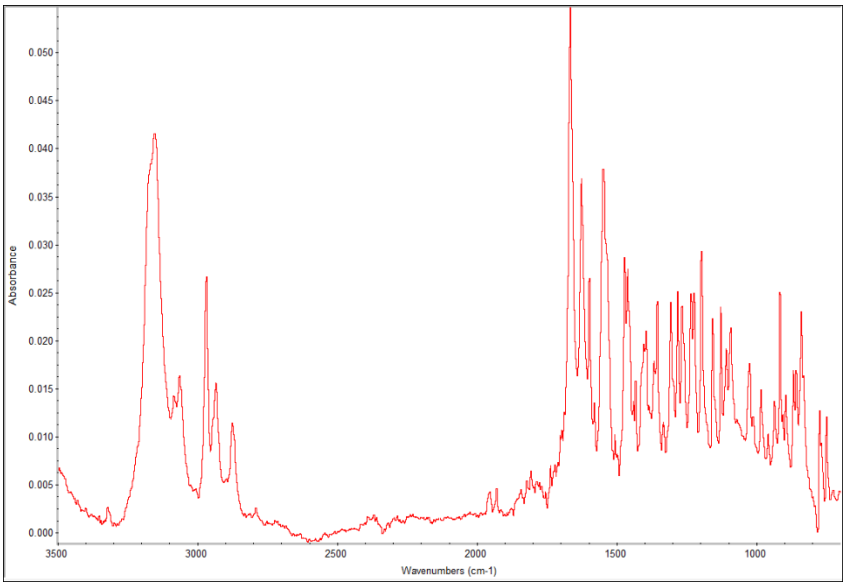


5% Aqueous $HAuCl_4$

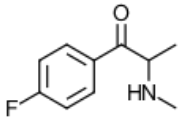


Tests for NRG-3 are not very sensitive. With 10-20 µg of the substance dissolved in water, 10% hydrochloric acid or 10% acetic acid, NRG-3 forms large composites of yellow plates and blades. However, the time taken for crystal formation is very slow with crystals fully forming after 1 hr.

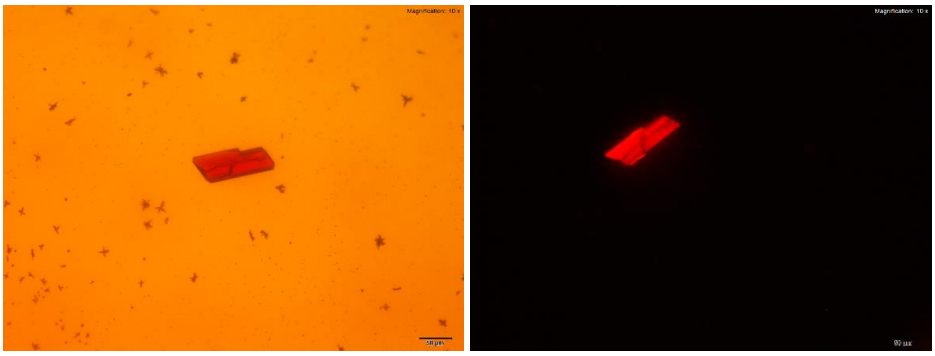
The IR spectrum of the crystal is representative of the crystal structure.



13. 4-Fluoromethcathinone

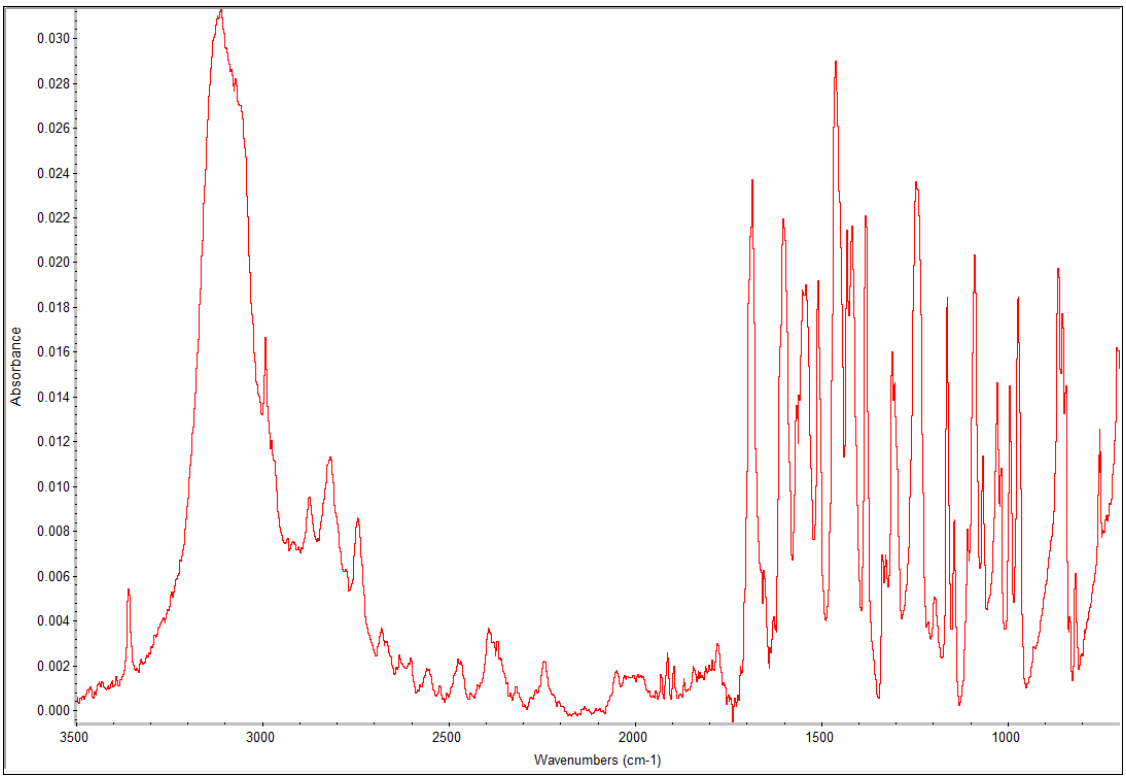


Gold bromide ($HAuBr_4$)

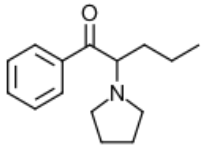


The most reliable test for 4-FMC is the gold bromide test. The substance forms long red serrated blades that start as short orange blades. The blades overtake the drop over time and can be readily identified. The blades are a deep orange to red under crossed polars.

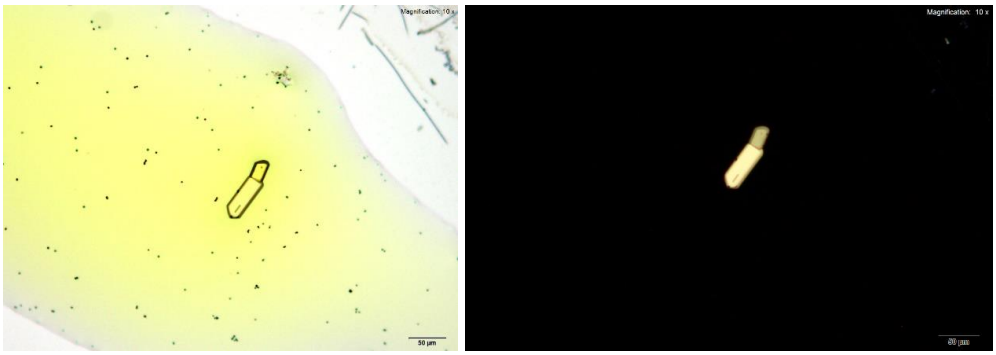
The blades can be easily maneuvered onto IR substrates to obtain information-rich spectra.



14. α-Pyrrolidinopentiophenone

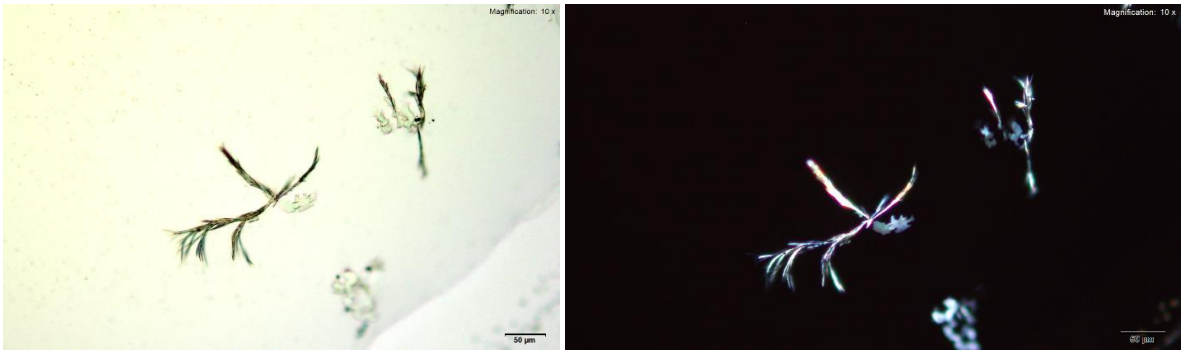


5% Aqueous $HAuCl_4$



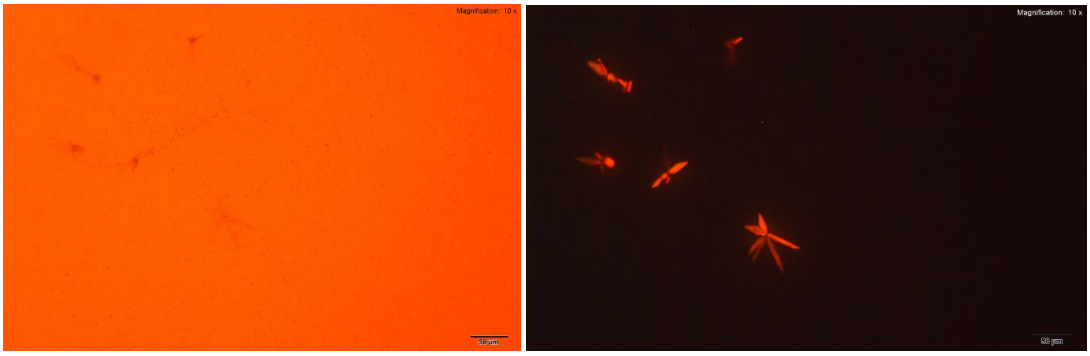
Prisms and tablets are formed in aqueous solutions of α-PVP with gold chloride reagent. The narrow prisms may be short with rounded growing ends. The prisms grow to look like long rods in time. Both forms of the crystal are very bright under crossed polars.

5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



A colorless leafy skin on the surface and branched tufts of dark needles are formed when α-PVP is mixed with the acidic gold chloride reagent. The skin precedes the needles but both formations can be observed together after 10 minutes of crystal growth.

Gold bromide ($HAuBr_4$)

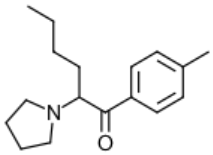


Clusters of thin, orange plates are observed with the gold bromide reagent. The orange crystals are best visualized in crossed polars. The crystals are bright and the characteristic clusters of plates can be observed.

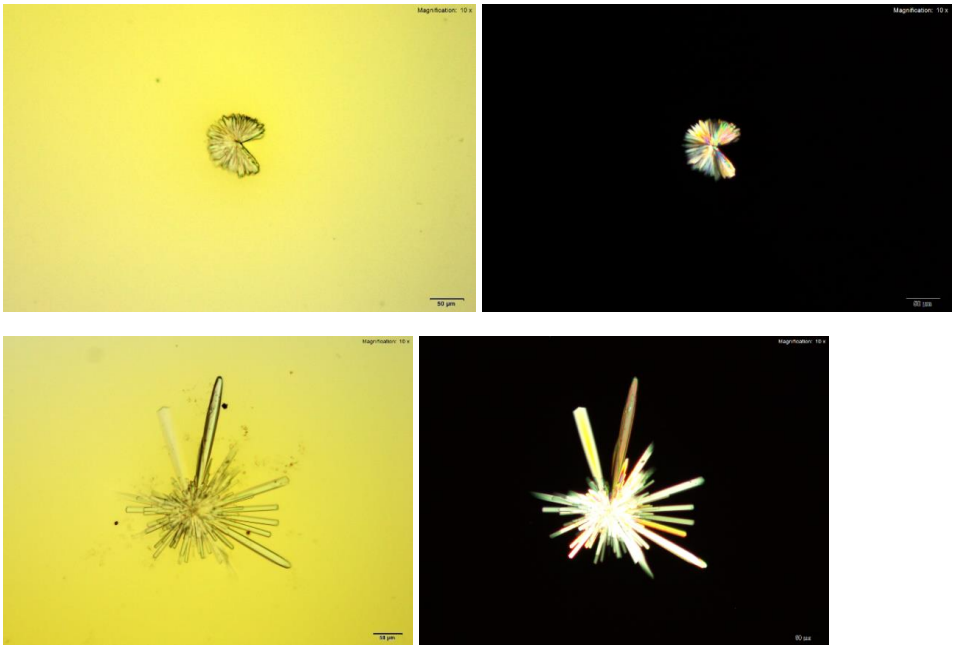
Overall recommended test:

The gold bromide reagent is the best reagent for characterizing α-PVP and forms crystals reliably. The crystals however are very fragile and though infrared spectra can be obtained, the spectra are not information-rich.

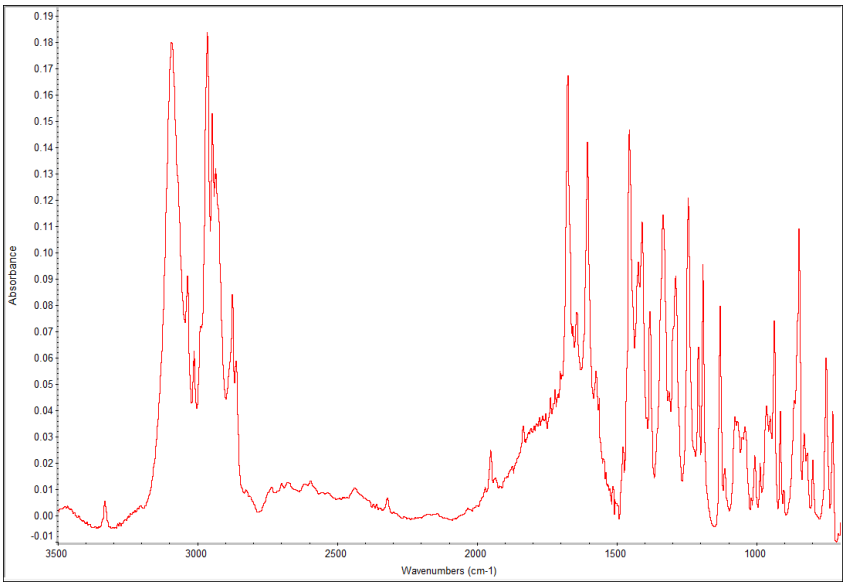
15. 4'-methyl-α-Pyrrolidinohexanophenone (MPHP)



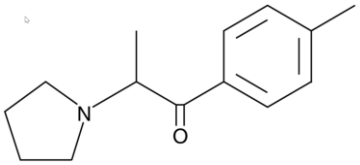
<u>Platinic Chloride (H_2PtCl_6)</u>



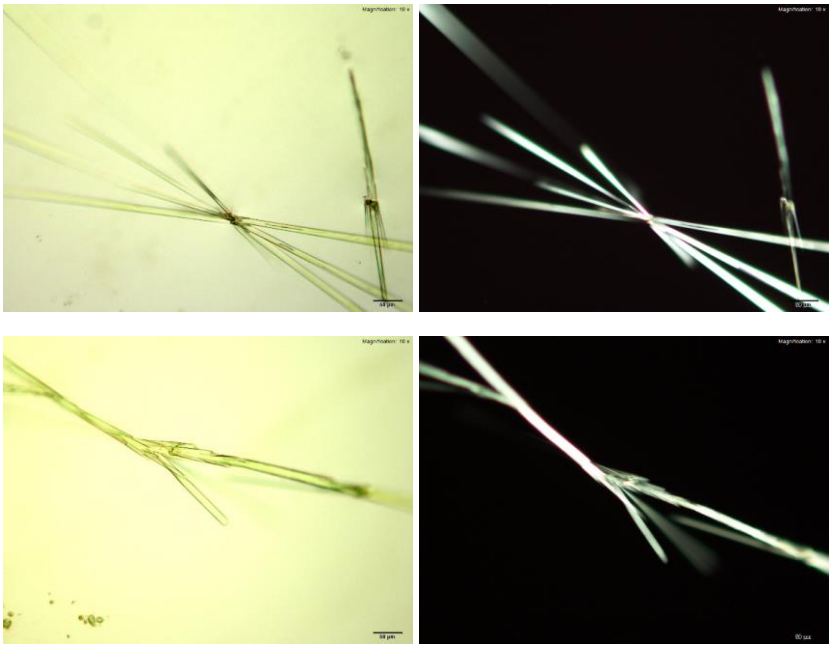
MPHP in aqueous and acidic solutions gives immediate growth of characteristic rosettes of thick rods. Some of the individual crystals in the rosette may grow long to appear as thick blades and end when they meet the growing edge of another crystal. The crystals are easily available for IR microspectroscopy and the resulting IR spectra are information-rich.



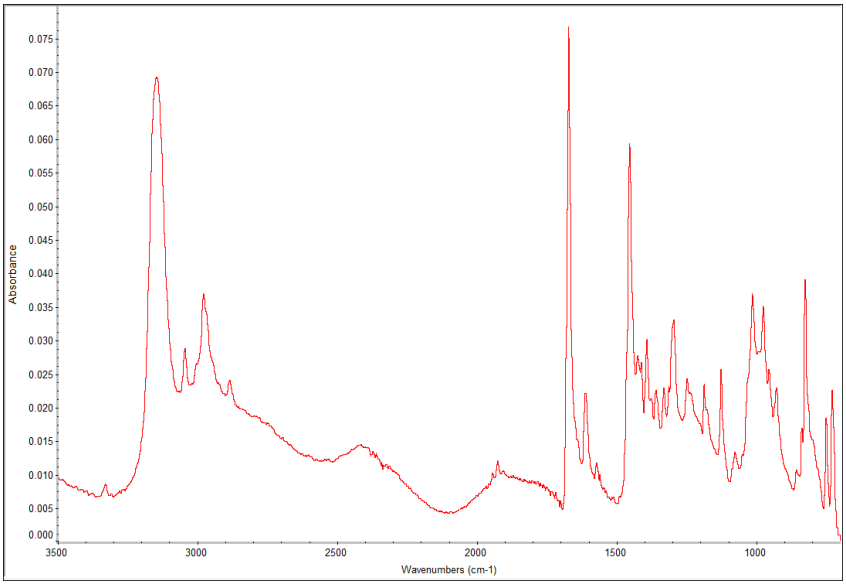
16. 4'-methyl-α-Pyrrolidinopropiophenone (4-MePPP)



5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20

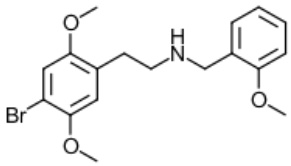


MPHP in aqueous and acidic solutions gives immediate growth of characteristic rosettes of thick rods. Some of the individual crystals in the rosette may grow long to appear as thick blades and end when they meet the growing edge of another crystal.Information-rich IR spectra can be obtained from the crystals when they are chosen carefully.



Phenethylamine Class

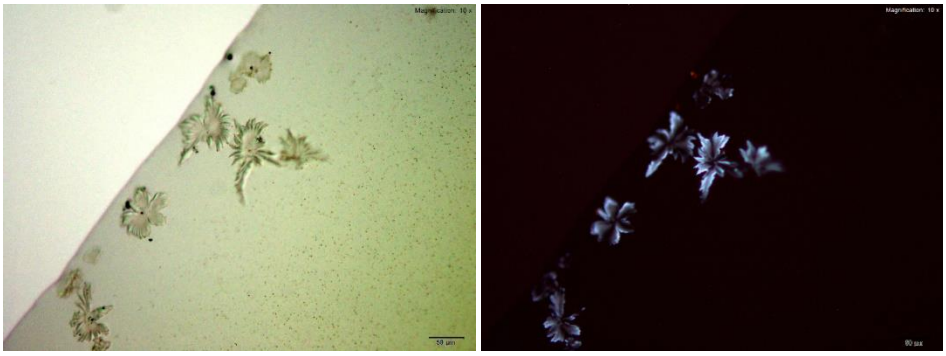
1. **25B-NBOMe**



5% Aqueous $HAuCl_4$

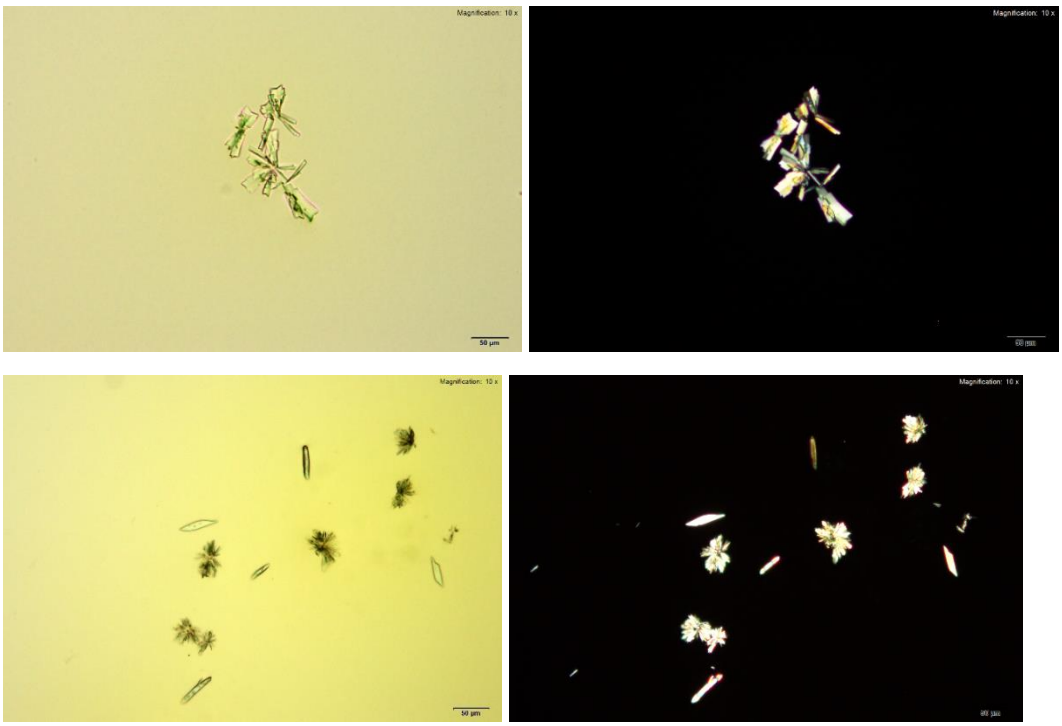
25B-NBOMe in aqueous or acidic solutions forms yellow dendrites with comb like arms when mixed with gold chloride reagent. The shape of the crystal is better observed under acidic conditions where the formations are larger with distinct features of the combs.

5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



Colorless, light floral dendrites of 25NBOMe are formed with the acidic gold chloride reagent. The crystals are formed like a floating skin at the edge of the drop and can be best visualized under crossed polars.

Platinic Chloride (H_2PtCl_6)

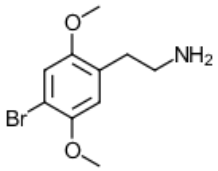


Clusters and rosettes of colorless, irregular plates are observed for 25B-NBOMe with platinic chloride reagent. Aqueous and acidic solutions of 25B-NBOMe give slightly different overall crystal habits. The clusters are larger and held together in aqueous solutions whereas in acidic solutions the crystals are observed as loose clusters and individual colorless plates and blades may be observed alongside the clusters.

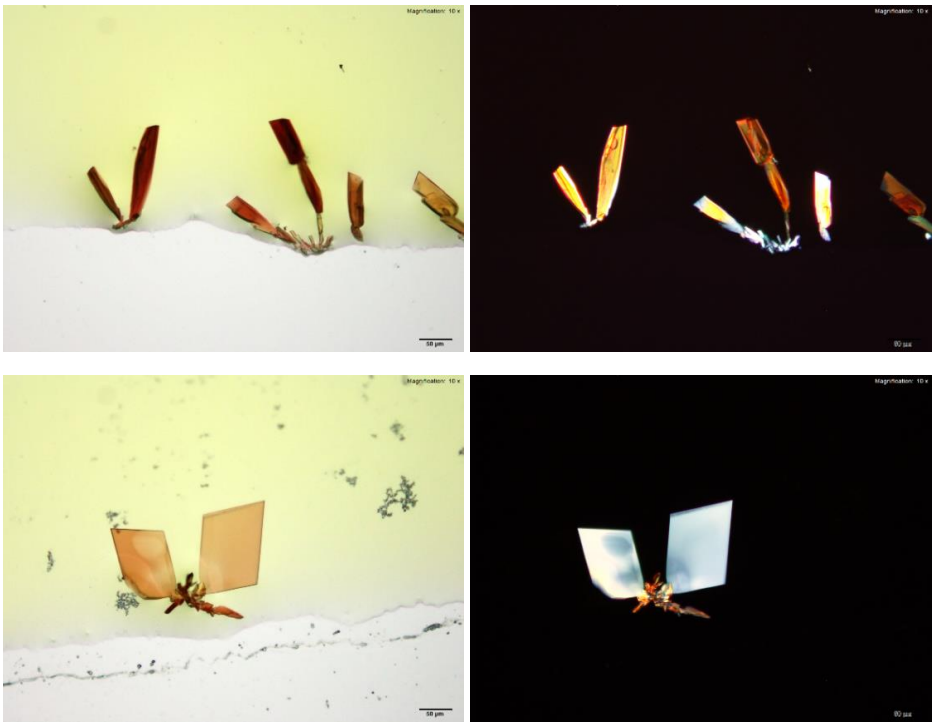
Overall recommended test:

The microcrystals observed for the three reagents discussed are not suitable for IR microspectroscopy. For microcrystalline test, the platinic chloride reagent mixed with acidic solutions of the substance is recommended.

2. 2C-B

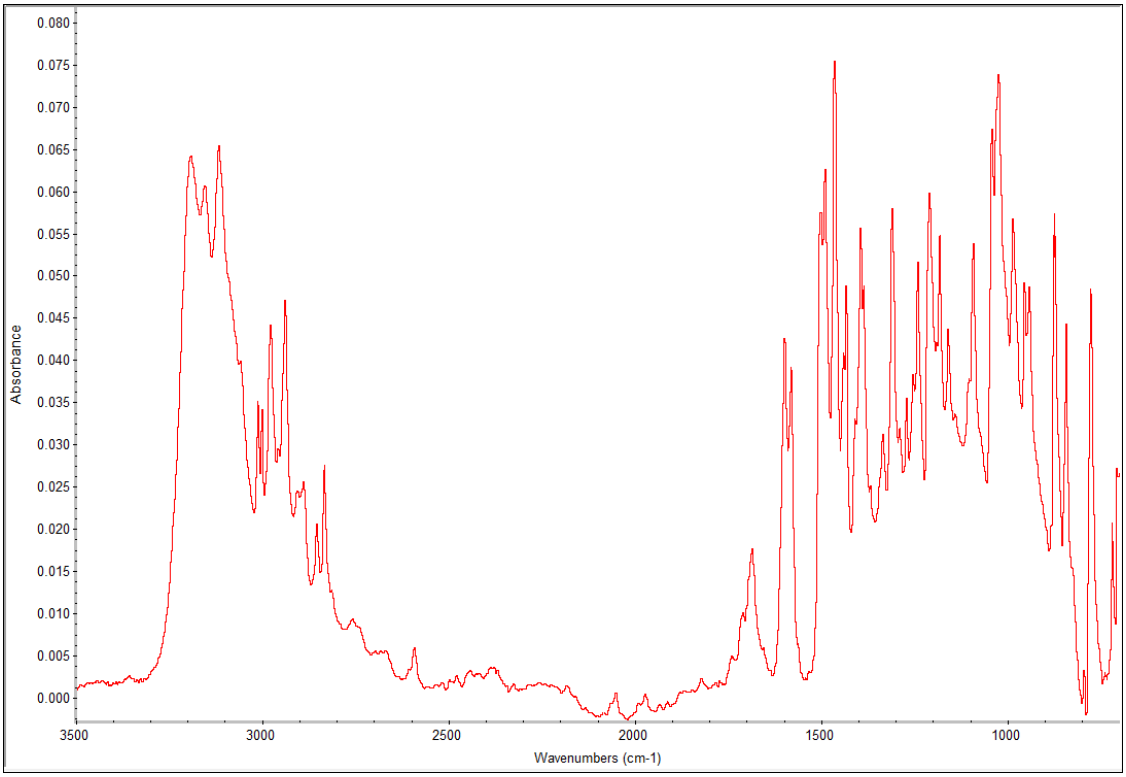


<u>5% Aqueous $HAuCl_4$</u>

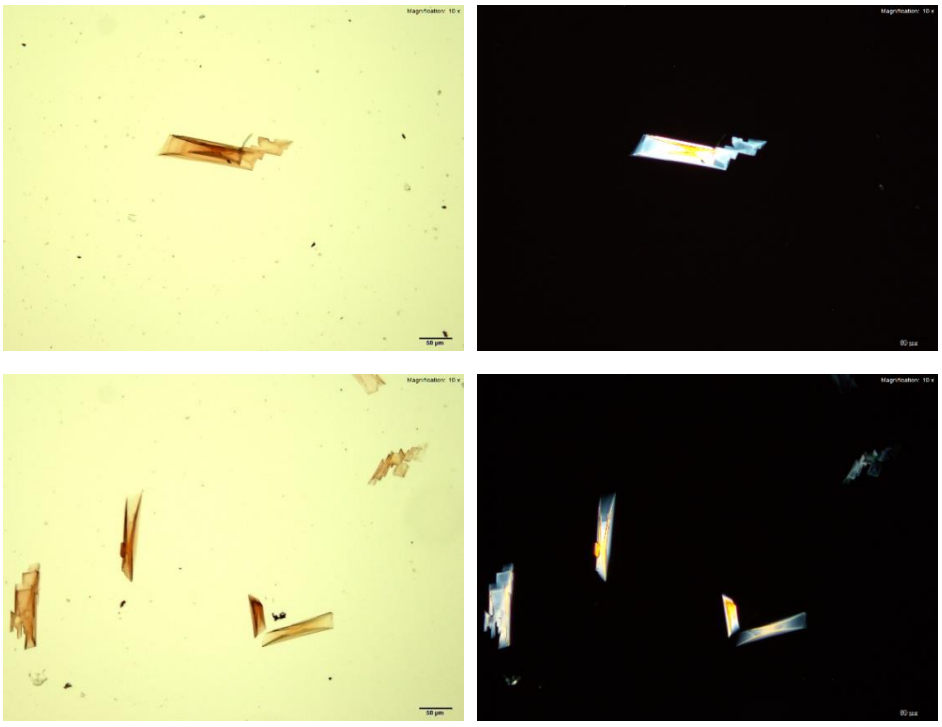


2C-B in aqueous solutions or acidic solutions of 10% hydrochloric acid or 10% acetic acid forms red-deep orange thick blades and large plates. The blades grow from the edge into the drop either individually or in clusters. The blades may be stacked on each other giving a serrated appearance to a long blade. The blades are very bright under crossed polars and appears in various colors depending on their thickness.

The plates and blades are suitable for IR microspectroscopy when they grow large but not very thick. Characteristic IR spectra are obtained.



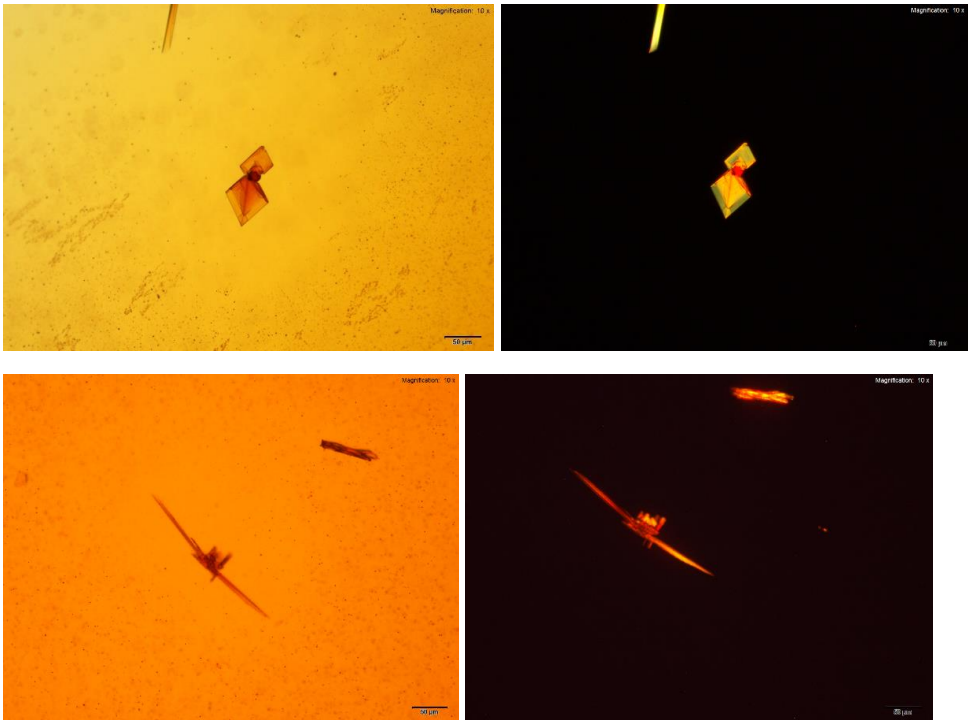
5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



2C-B in acidic gold chloride reagent forms red rectangular tablets. The crystals are very distinct and grow as composites of individual tablets and plates. The composite grows very large with time. Under crossed

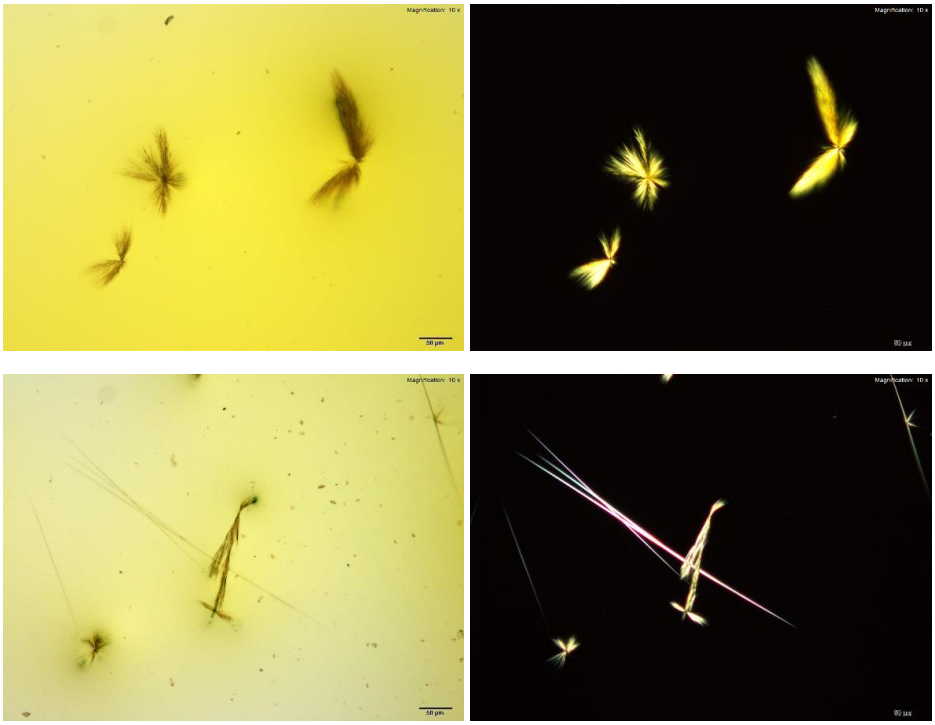
polars, the crystals appear white to blue on the thinner edges and yellow to orange as the thickness increases.

Gold bromide ($HAuBr_4$)



Gold bromide reagent gives distinct red prisms and tablets that generally grow individually or in small composites. The prims are suspended in the drop and can appear as needles or blades when oriented on their sides. The crystals are very bright under crossed polars.

Platinic Chloride (H_2PtCl_6)

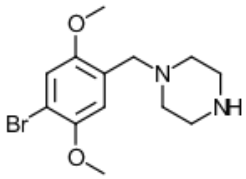


2C-B in aqueous, 10% hydrochloric acid, or 10% acetic acid solutions forms long yellow needles and clusters of short needles. The long needles appear either individually or in loose clusters whereas the short needles appear as tight sheaves or clusters. The tighter the cluster, the darker the growth appears. Both forms of crystals may be observed simultaneously. The darker clusters appear very bright under crossed polars and the individual needles are very faint.

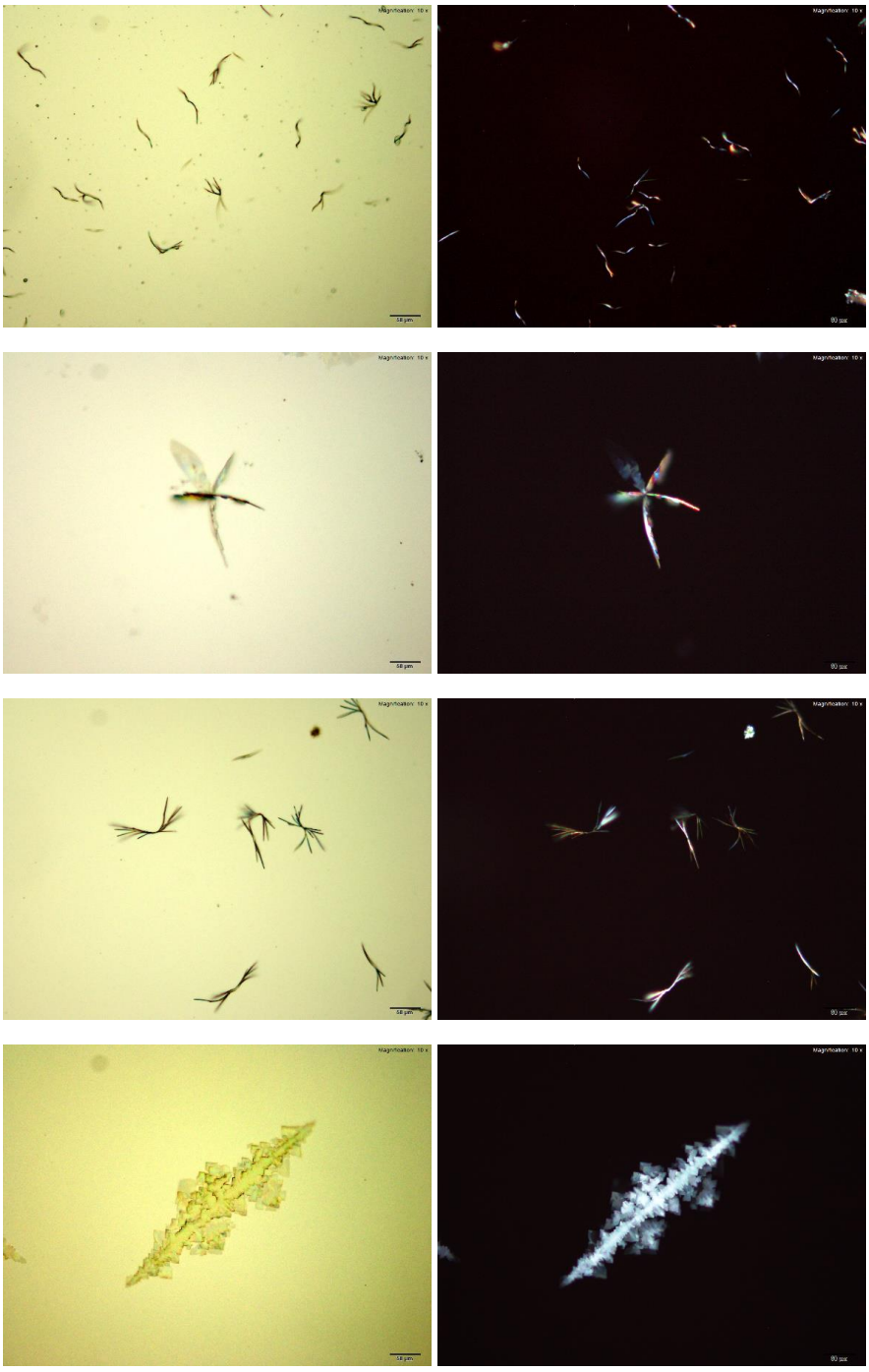
Overall recommended test:

2C-B gives characteristic crystals with gold-based reagents and these crystals can also be studied with IR microspectroscopy. The aqueous gold chloride reagent is ideal, among the gold-based reagents, for obtaining reproducible IR spectra because of the availability of large crystals.

3. 2C-B-BZP

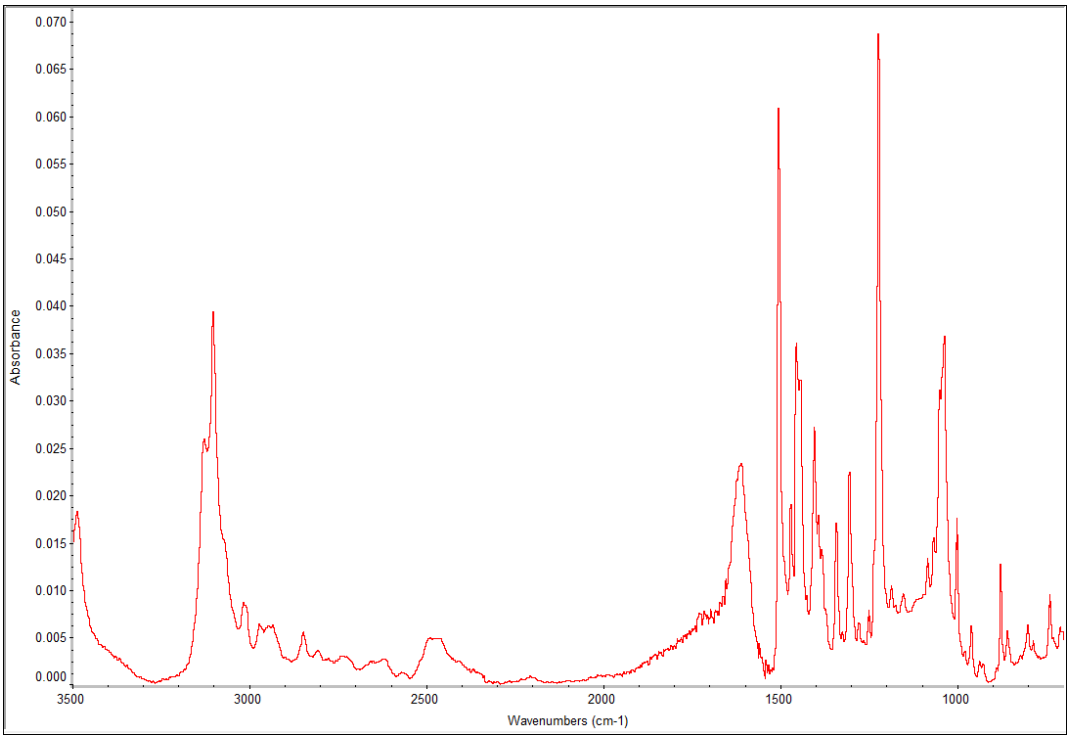


5% Aqueous $HAuCl_4$



2C-B-BZP forms various forms of clusters of short dark needles and insects. Light crosses that grow as large composite blades are also observed. Both forms are observed in aqueous and acidic solutions of 2C-B-BZP. The blades are bright under crossed polars whereas the needles are very faint.

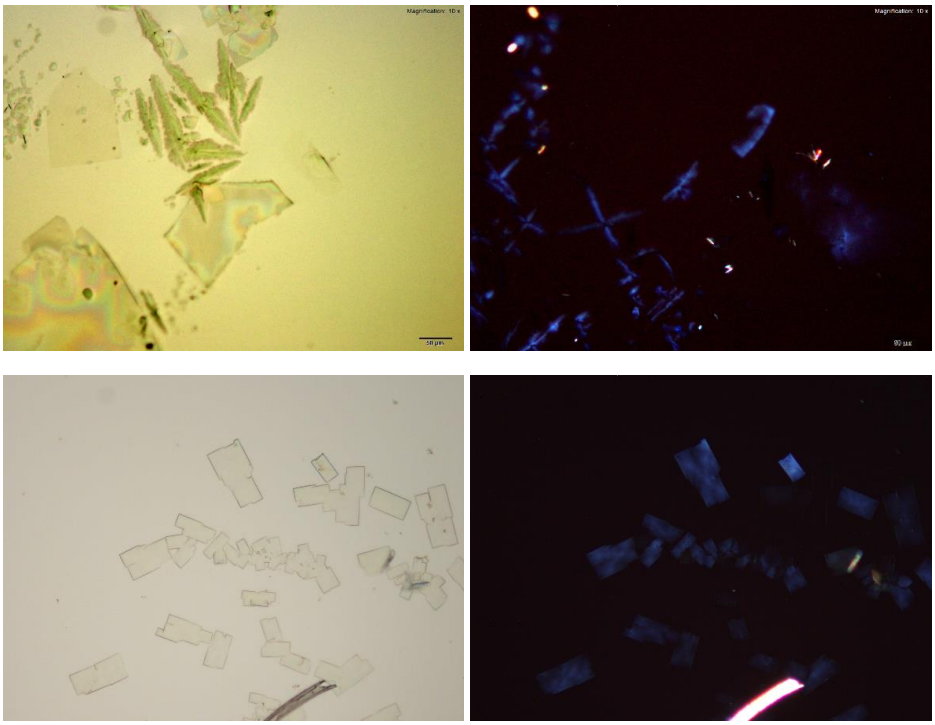
The large composites are suitable for IR microspectroscopy.



Gold bromide ($HAuBr_4$)

2C-B-BZP with gold bromide reagent forms individual yellow, light crosses that float in the drop. The crosses are bright yellow under crossed polars. The crosses are not visible under crossed polars when they are oriented along the plane of the slide.

Platinic Chloride (H_2PtCl_6)

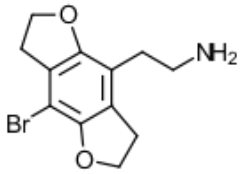


2C-B-BZP with the platinic chloride reagent first forms a large skin on the drop. The skin is actually an accumulation of large thin fragile plates. Under the skin, more substantial clear rectangular plates are observed. Both forms are faint and appear light bluish white colored under crossed polars. The individual crystal is better visualized under crossed polars.

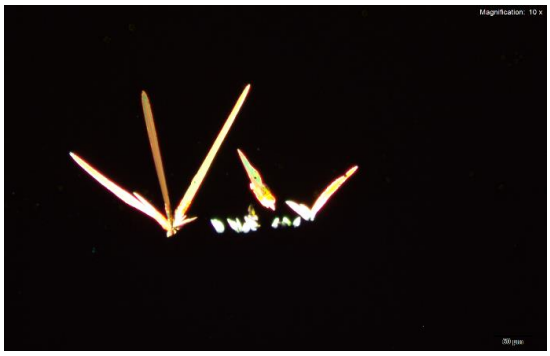
Overall recommended test:

Though there are multiple forms of crystals observed with the aqueous gold chloride reagent, it is the recommended test for 2C-B-BZP because of the availability of the large blades composites for IR microspectroscopy.

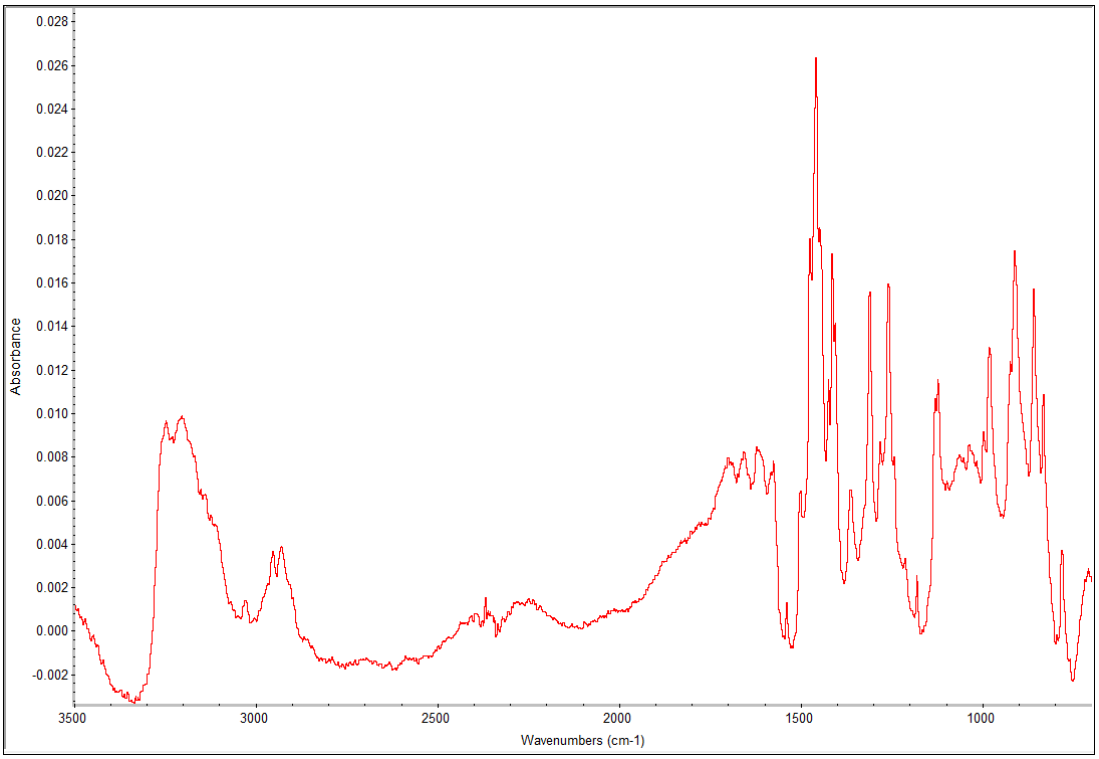
4. 2C-B-FLY



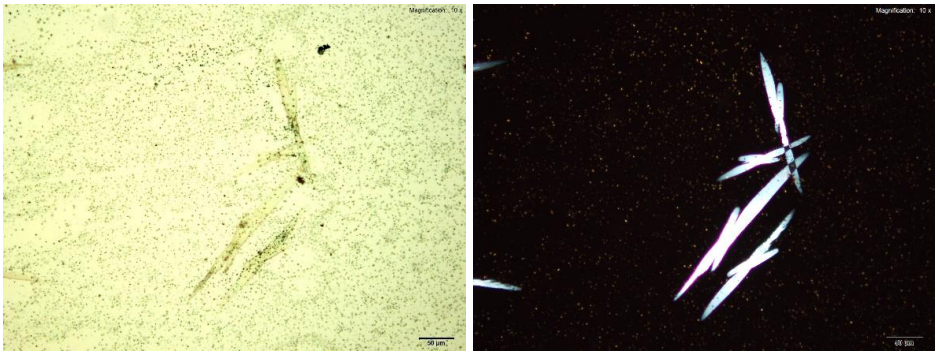
5% Aqueous $HAuCl_4$



2C-B-FLY as a 10% hydrochloric acid solution mixed with aqueous gold chloride reagent grows characteristic long red blades with a tapered growing end. The blades are very bright and yellow-orange in color under crossed polars. Aqueous and acetic acid solutions of 2C-B-FLY give slow and inconsistent crystal formation.

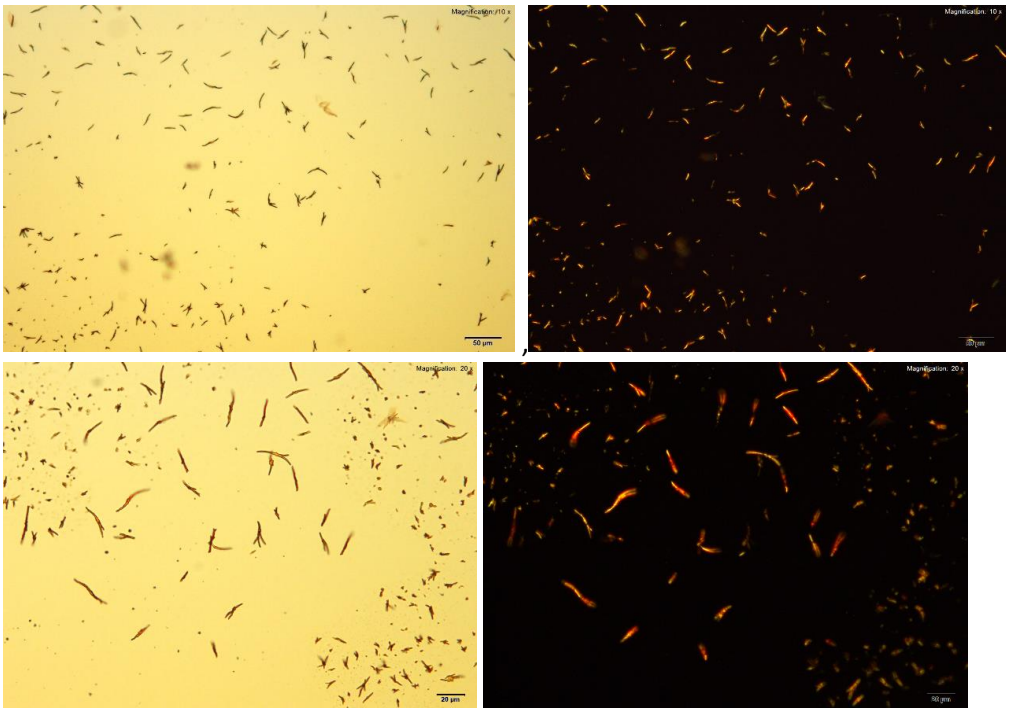


5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



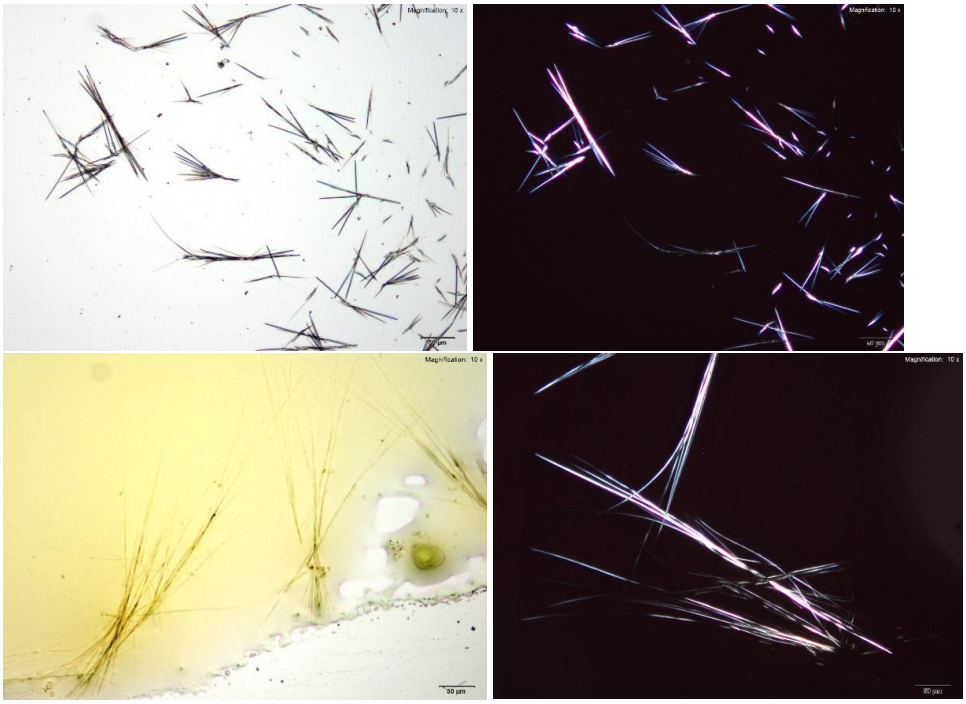
In the acidic gold chloride reagent, colorless, flat long blades that grow individually or in X-shaped habit are observed for 2C-B FLY. The colorless blades are not very apparent in brightfield visualization but are very bright under crossed polars.

Gold bromide ($HAuBr_4$)



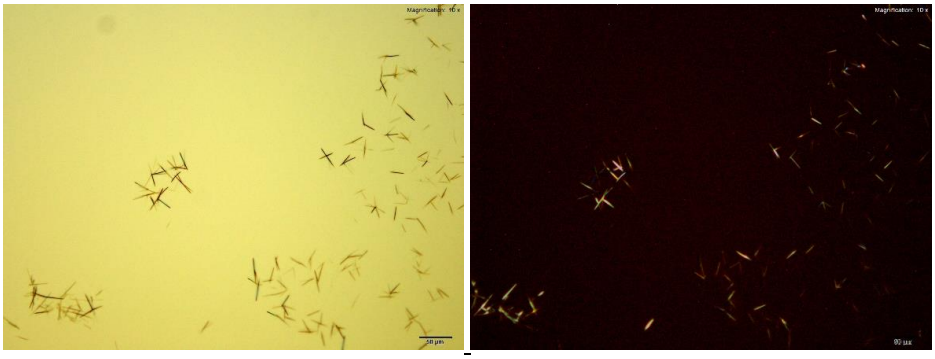
With gold bromide reagent, very small red rods are observed for 2C-B-FLY. These rods grow abundantly and the drop may appear to have precipitated immediately upon adding reagent. The wavy short rods are better visualized under higher magnifications.

Platinic Chloride (H_2PtCl_6)



Long colorless-yellow needles are observed for 2C-B-FLY in aqueous and 10% acetic acid solutions. When dense clusters of these long wispy needles are formed, the clusters appear dark. The needles are very bright under crossed polars. When a 10% hydrochloric acid solution of the substance is used for the test, the crystals formed are darker, shorter and broader.

Platinic Bromide (H_2PtBr_6)

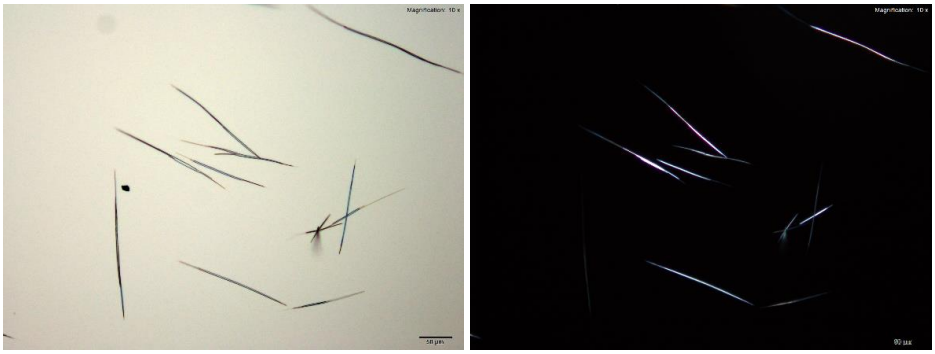


Clusters of short red needles are observed for 2C-B-FLY with platinic bromide reagent. The light clusters grow abundantly. Under crossed polars, the crystals are bright but their visibility and color is dependent on their orientation in the cluster.

Mercuric Iodide (MgI_2)

In mercuric iodide reagent, feathery clusters of short needles are formed for 2C-B-FLY. The needles appear bright under crossed polars.

Mercuric Chloride ($MgCl_2$)

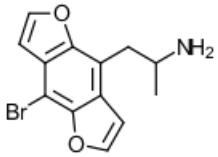


2C-B-FLY with mercuric chloride reagent gives long, flat and thin blades that may grow individually or in clusters. The colorless blades when oriented on their sides look like dark needles and appear dark. The blades appear white and bright under crossed polars.

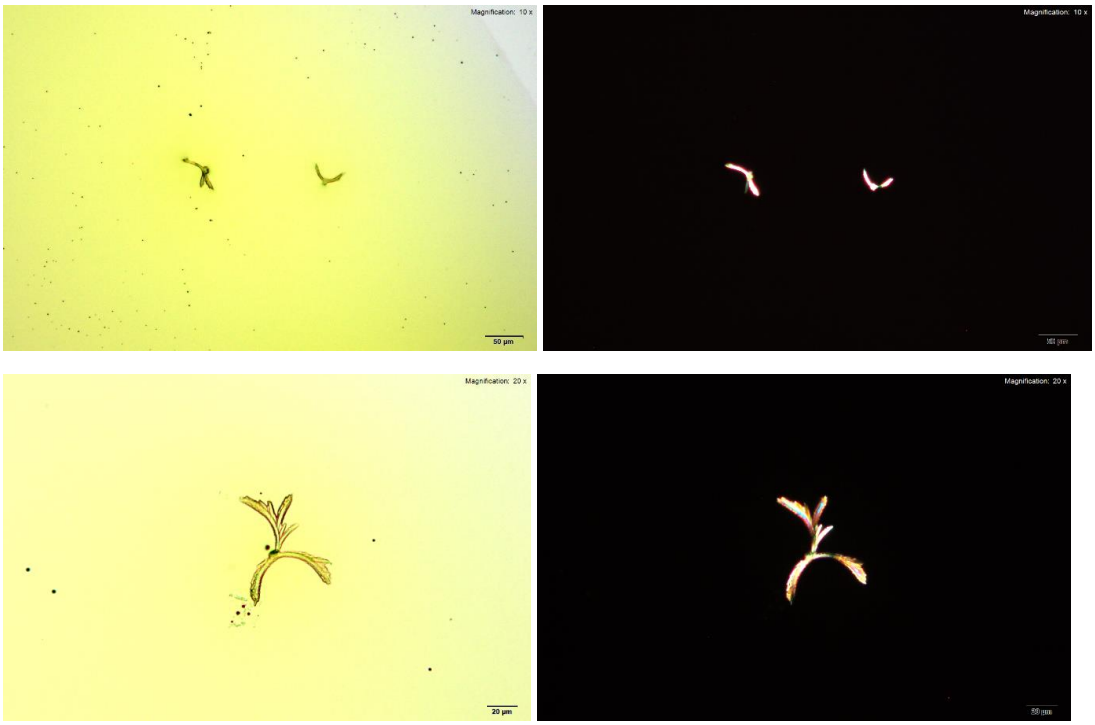
Overall recommended test:

2C-B-FLY forms crystals with several reagents and each of these are abundantly formed and quite distinct. However, the crystals with the aqueous gold chloride reagent are most suitable for IR microspectroscopy.

5. **Bromo-DragonFLY**

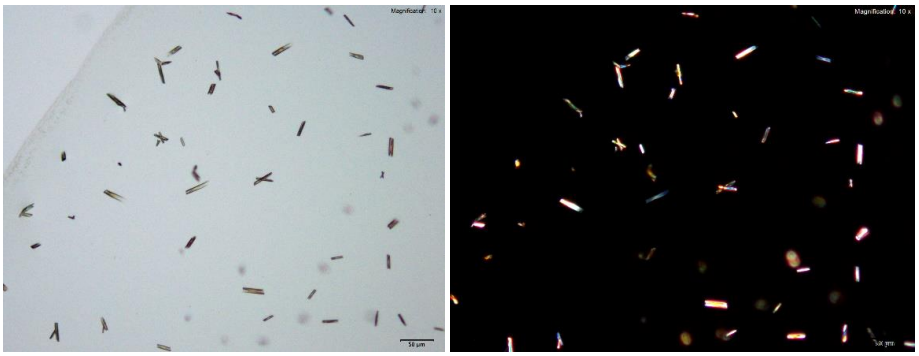


5% Aqueous $HAuCl_4$



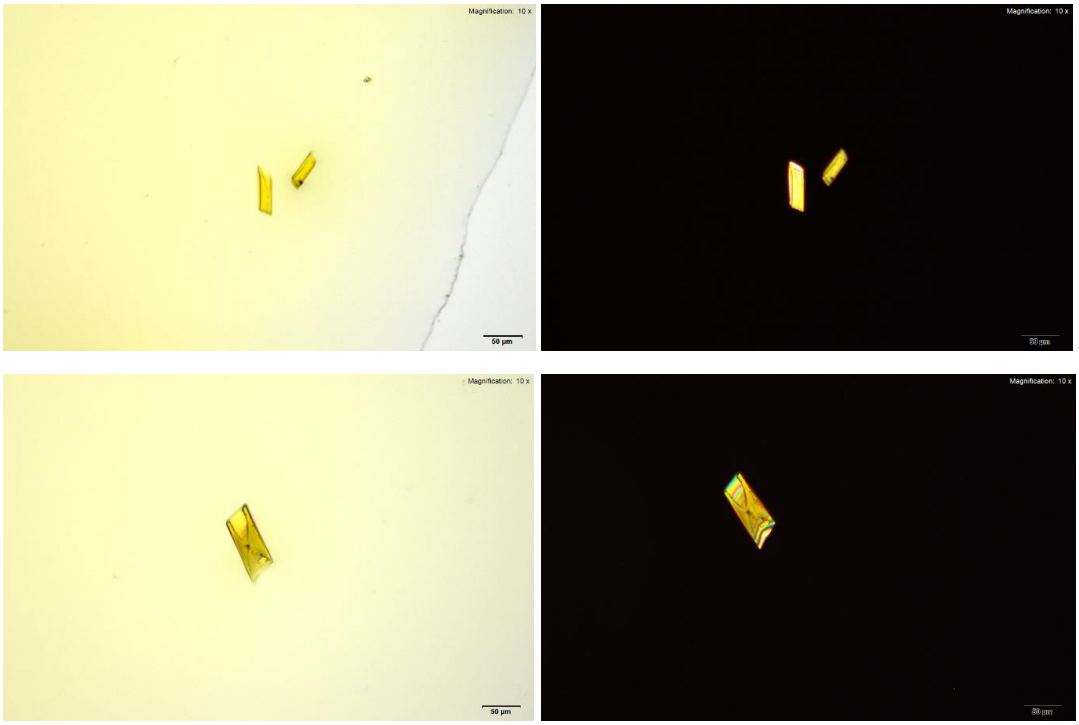
Bromo-DragonFLY in 10% hydrochloric acid solution gives small brown branched combs. These structures are best observed under crossed polars or higher magnifications. The crystals take 15 minutes to grow after reagent addition with Bromo-DragonFLY amounts less than 10 µg.

5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_20



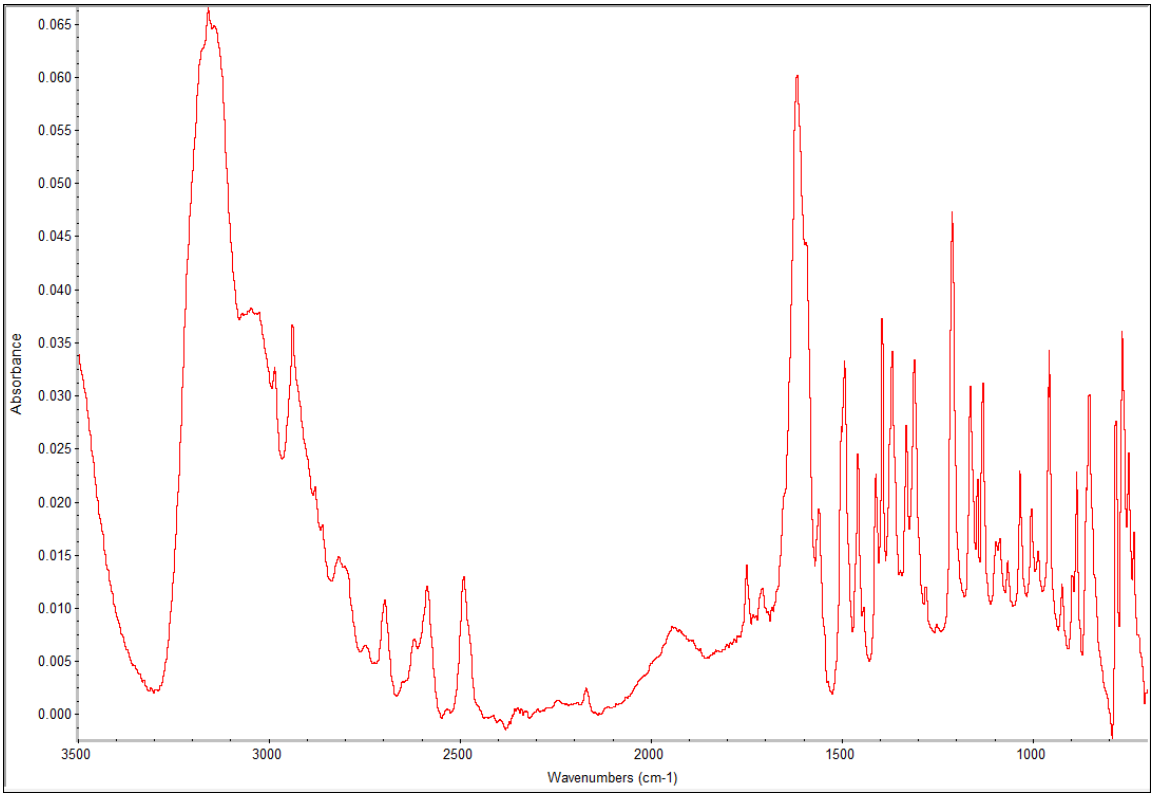
In acidic gold chloride reagent, Bromo-DragonFLY, gives reddish brown rectangular flat plates and small feathery crosses. These two crystals are concentration dependent and the plates are most commonly observed. They grow abundantly and are very bright under crossed polars.

Platinic Chloride (H_2PtCl_6)

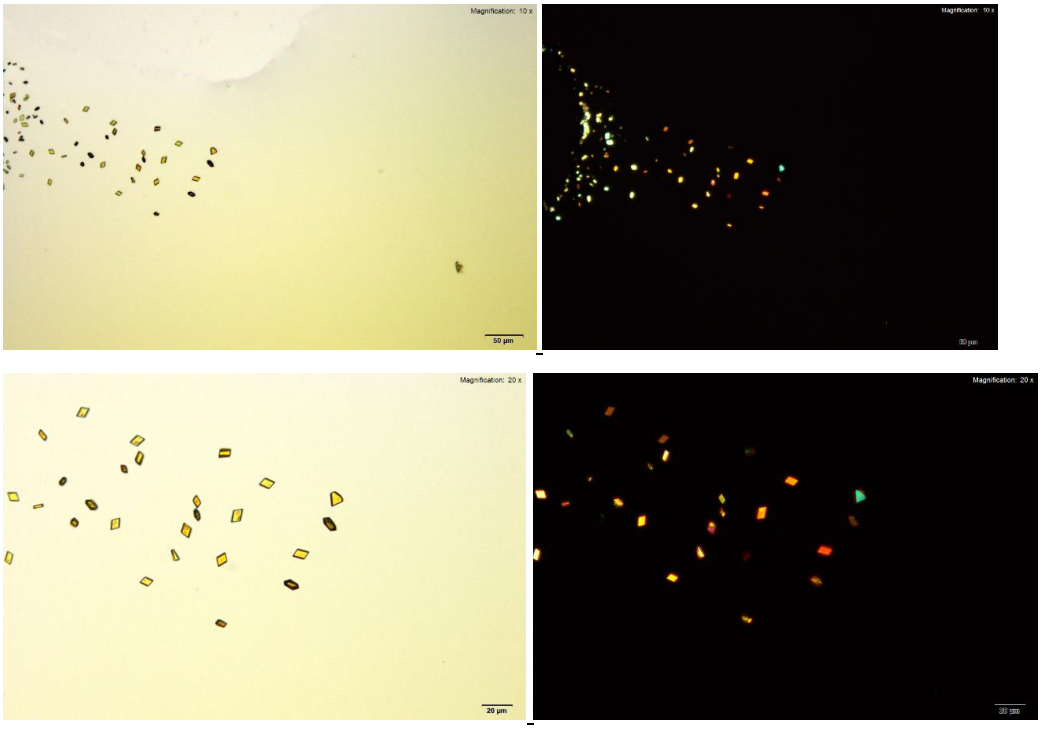


Bromo-DragonFLY in aqueous and acidic solutions, gives characteristic thick, yellow, rectangular prisms with the platinic chloride reagent. The prisms grow individually and with straight edges but can have uneven growing ends. The size of the prisms is concentration dependent and at lower than 5 µg of the substance the prisms grow very large.

These large crystals are easily studied by IR microspectroscopy and give information-rich spectra representative of the reference Bromo-DragonFLY spectra.

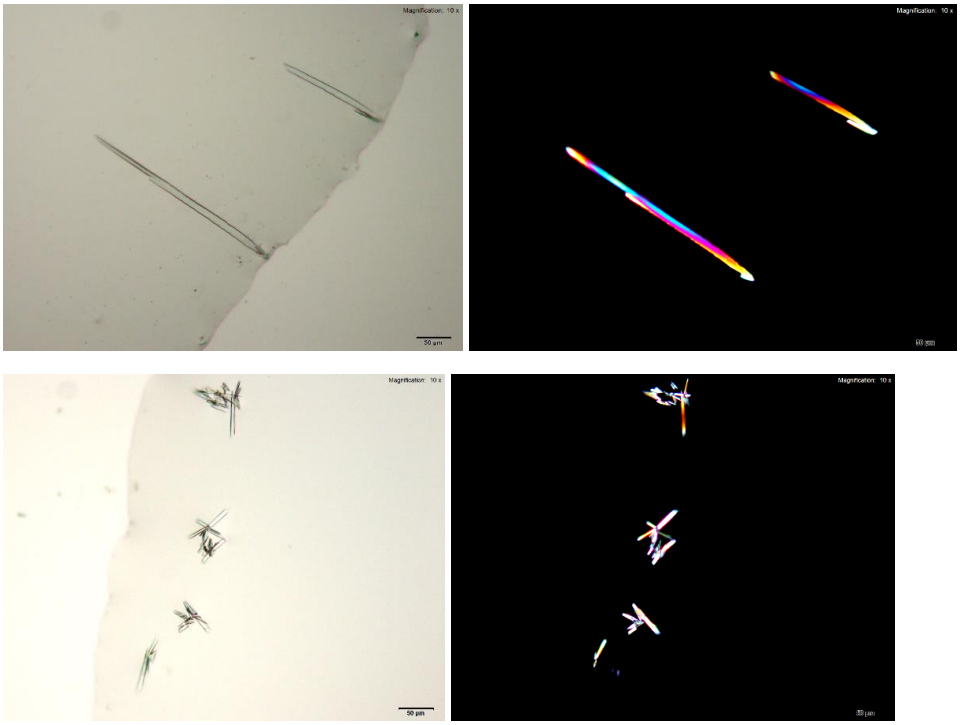


Platinic Bromide (H_2PtBr_6)



Crystals observed for Bromo-DragonFLY with platinic bromide are somewhat similar to those observed with platinic chloride. The tablet-like yellow prisms are shorter and thicker. Variations to the prisms include pyramids and shards.

Mercuric Chloride ($MgCl_2$)



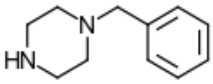
Mercuric chloride reagent gives colorless, long, flat blades that grow into the drop from the edge of the drop. When the blades grow in the drop, they grow as loose clusters of short blades. Both forms of the crystal grow quickly and are readily observed under brightfield and crossed polars.

Overall recommended test:

Though characteristic crystals are observed for several reagents, the platinic chloride test is ideal because of the comparatively large crystal size and their suitability for IR microspectroscopy.

Piperazine Class

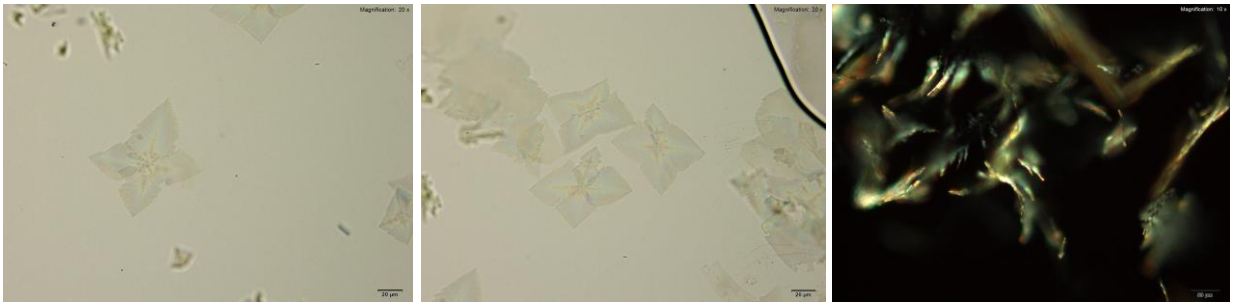
1. **BZP**



5% Aqueous $HAuCl_4$

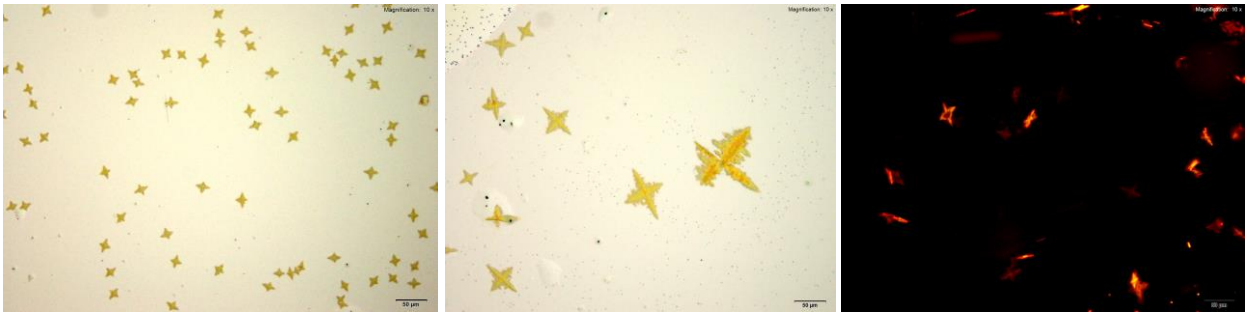
BZP forms colorless, large, light square plates. The plates float in the drop and are best photographed with a coverslip. The square plates appear light blue under crossed polars but only when they are suspended in the drop and not in the same plane as the glass slide. This test is very sensitive and can easily overgrow at concentrations of BZP as low as 1 µg.

5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_2O



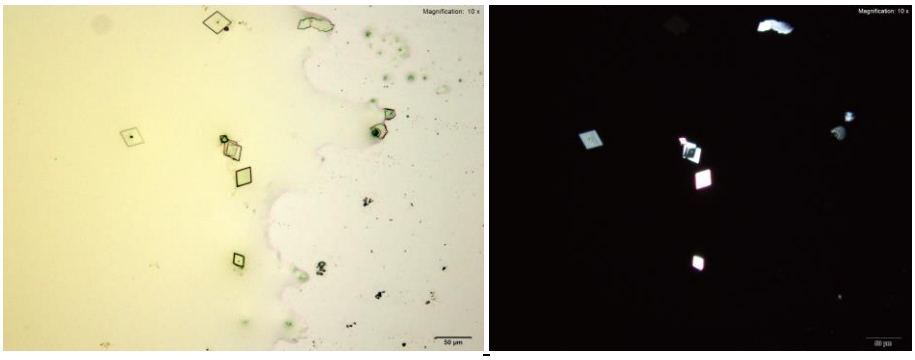
With the acidic gold chloride reagent, the reaction is very similar to the aqueous gold chloride reagent. The flaky square plates are large with indented sides to appear as a cross with equal sides. The crystals appear yellow under crossed polars. The crystal habit is best observed and photographed with a coverslip.

Gold Bromide ($HAuBr_4$)



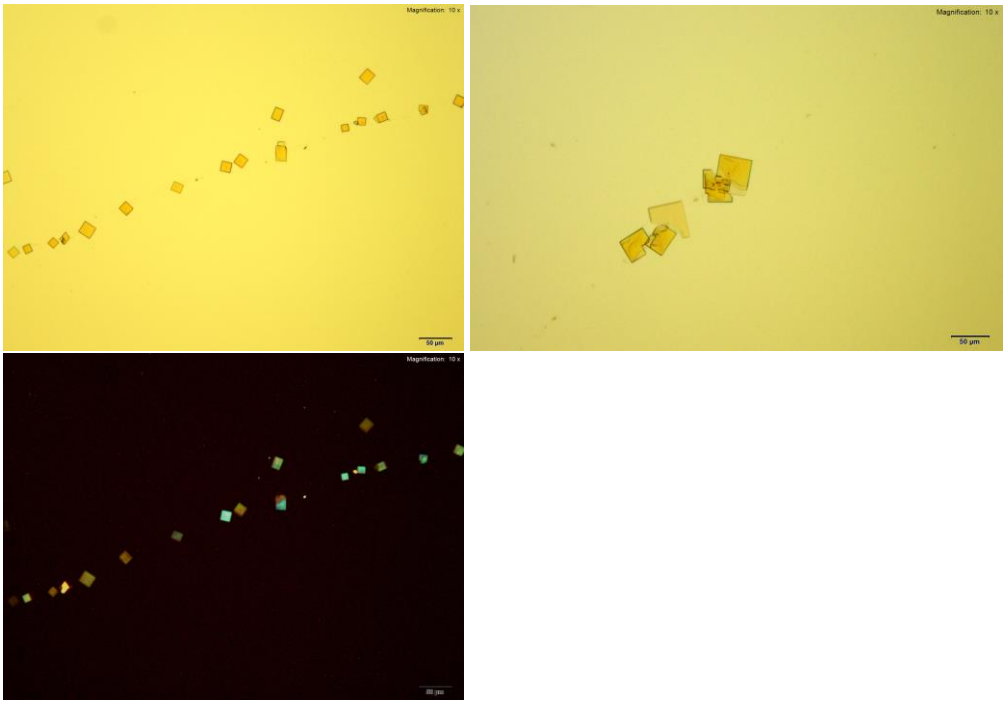
Gold bromide reagent forms yellow crosses with serrated edges that grow abundantly. The test is very sensitive and the size of the crosses depends on the concentration. The crosses appear orange under crossed polars and can be easily visualized unless they oriented flat along the plane of the slide.

Platinic Chloride (H_2PtCl_6)



BZP forms small, colorless square plates in the drop with the platinic chloride reagent. These plates can also be observed on the edges of drop with deformations. The size of the plates differs depending on if 10% hydrochloric acid or 10% acetic acid was used to make the BZP test solution. Platinic chloride reagent by itself crystallizes as square plates on standing. They appear very similar to the drug-reagent crystal observed in this test. However, the drug-reagent plates can be differentiated from the reagent crystals under crossed polars. The reagent crystals are dark or faintly visible under crossed polars while the drug-reagent crystals appear bright white or yellow.

Platinic Bromide (H_2PtBr_6)

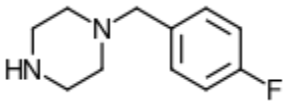


BZP with platinic bromide reagent forms yellow plates similar to the platinic chloride reagent. The crystals form abundantly and increase in numbers when the slide is scratched with a glass rod. The slower growing crystals are larger and can have deformities because of adjacent crystals. The crystals appear bright under crossed polars.

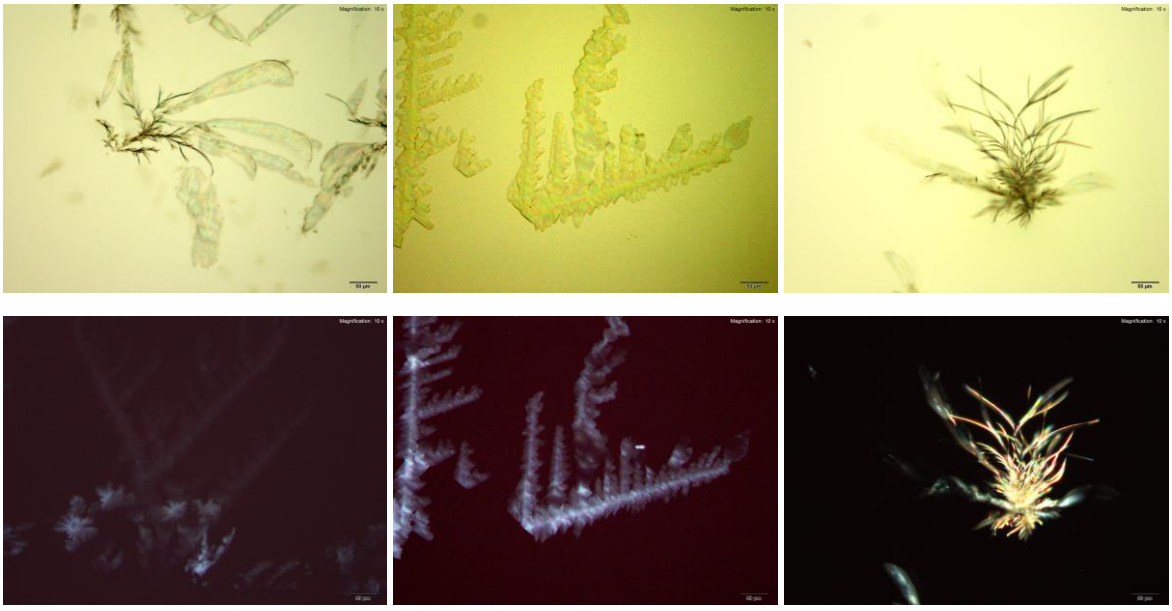
Overall recommended test:

Gold-based reagents are best for BZP. None of the crystals are sturdy enough for IR microspectroscopy so a combined test is not recommended under these crystal formation conditions.

2. **4-FluoroBZP**

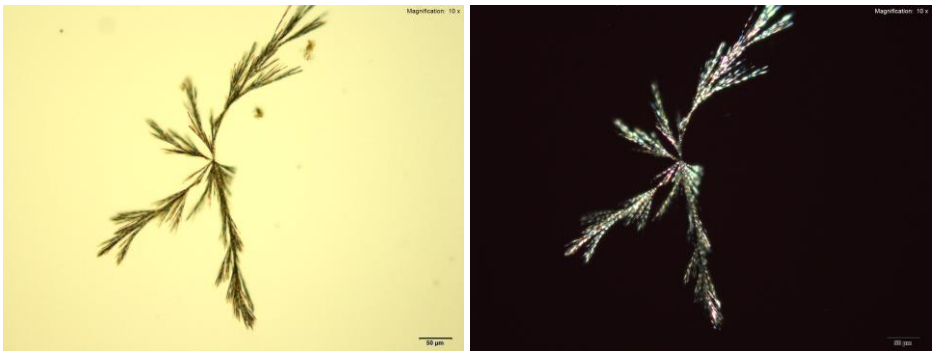


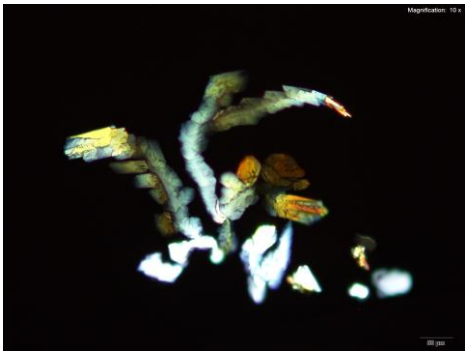
5% Aqueous $HAuCl_4$



4-FluoroBZP when dissolved in acidic solutions forms feathery blades. With 10% hydrochloric acid, the branched blades are more prominent. The blades are very faint in crossed polars. With 10% acetic acid, a combination of feathery blades and ribbons are observed. The ribbons are very bright under crossed polars.

Platinic Chloride (H_2PtCl_6)



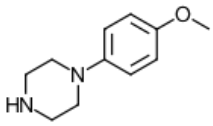



Platinic chloride forms two types of crystals with 4-FluoroBZP. The long, branched sheaves of dark crystals appear faint under crossed polars. The second form of crystals are thick hexagonal crystals that appear as composites in straight or branched chains. The hexagons are very distinct and appear very bright under crossed polars. This test is very sensitive and crystals form reliably.

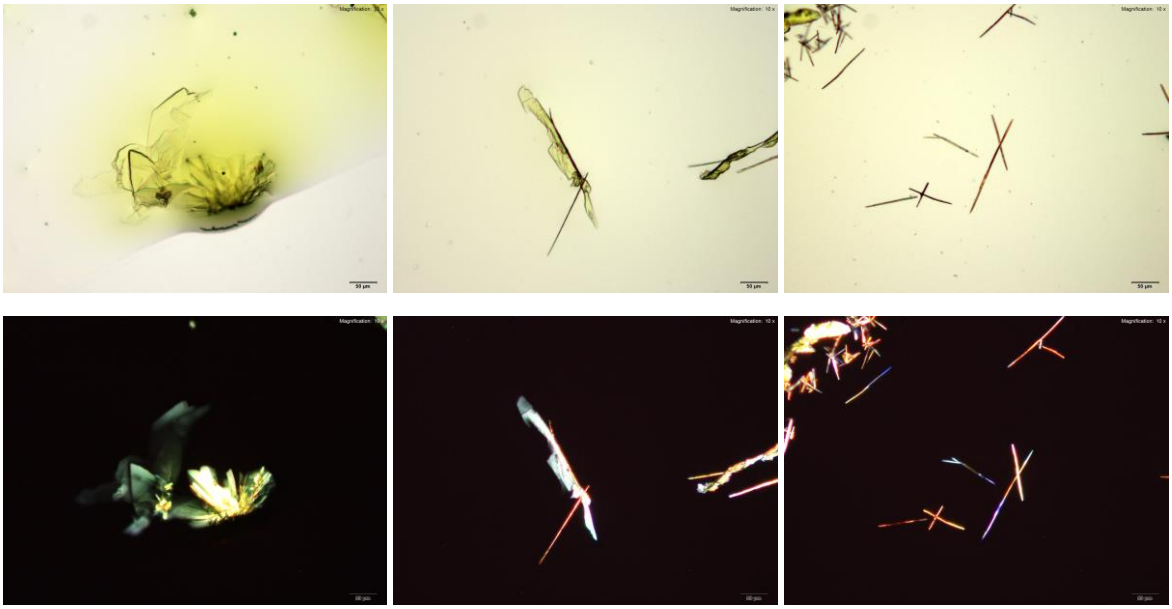
Overall recommended test:

Both the gold chloride and platinic chloride reagents form reliable and distinct crystals with 4-FluoroBZP. Only the hexagons formed with platinic chloride are sturdy enough for IR microspectroscopy. However, the crystal composite is not easily maneuvered and information-rich spectra are not reproducibly obtained.

3. 1-(4-Methoxyphenyl) piperazine (4-MeOPP)



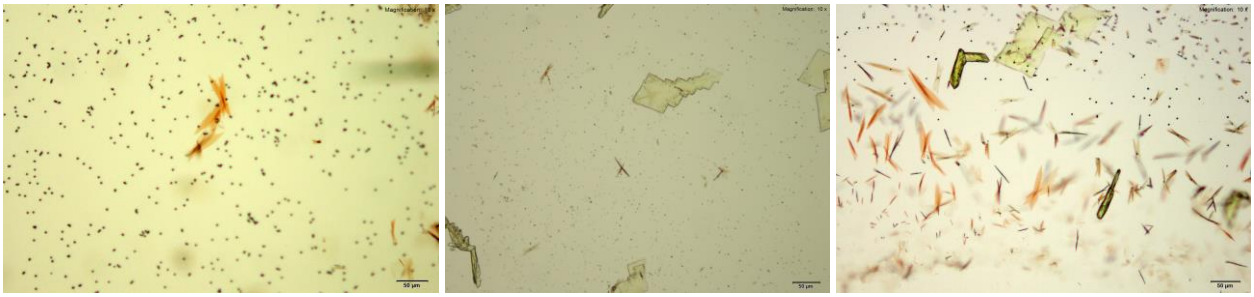
5% Aqueous $HAuCl_4$

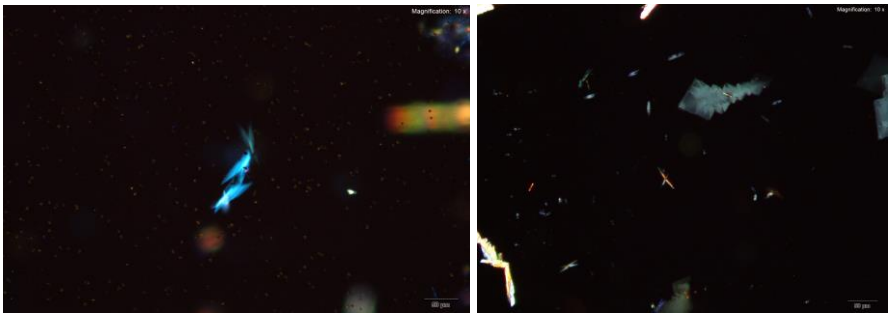


With the aqueous gold chloride reagent, two forms of crystals are observed for 4-MeOPP and their formation is concentration dependent. Clusters of thick irregular tablets are formed at the edge of the drop. These are colorless in bright field and bright yellow under crossed polars. The other form of crystals observed are long red rods that are arranged either individually or in loose clusters. A composite of these forms may also be observed.

Orange crosses similar to the ones observed with the acidic gold chloride reagent may also be observed at high concentrations when the reaction occurs very quickly.

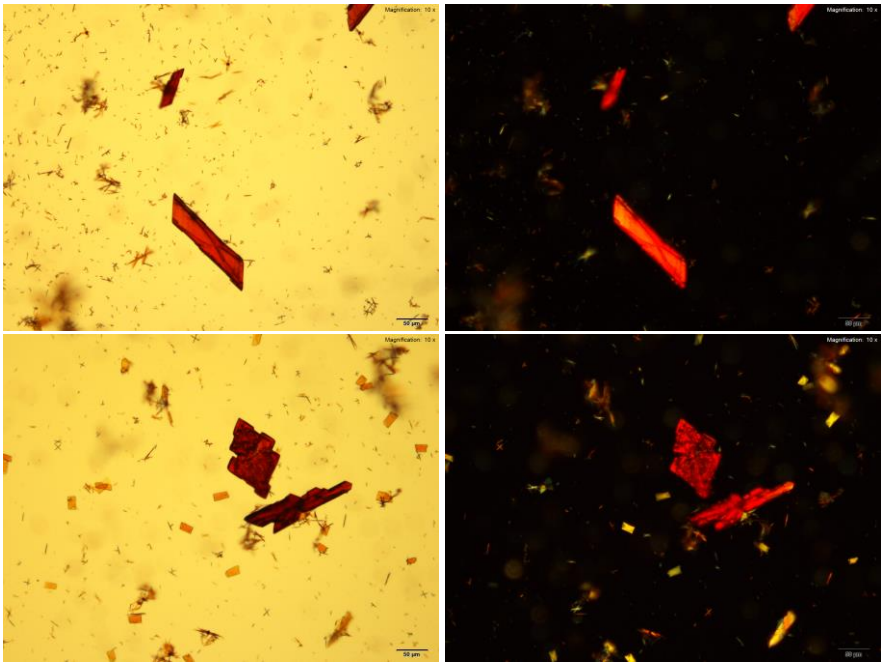
5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_2O





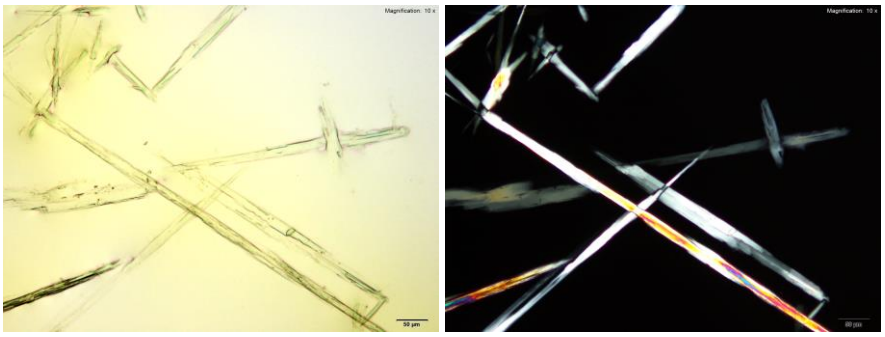
The first crystals observed with the acidic gold chloride reagent are light, feathery orange crosses. The crosses form so quickly that the drop becomes orange as soon as the reagent is mixed in. The drop slowly clears up as colorless thick irregular plates form at the expense of a few orange crosses. The orange crosses appear blue to yellow under crossed polars while the stacks of plates are faint white.

Gold Bromide ($HAuBr_4$)



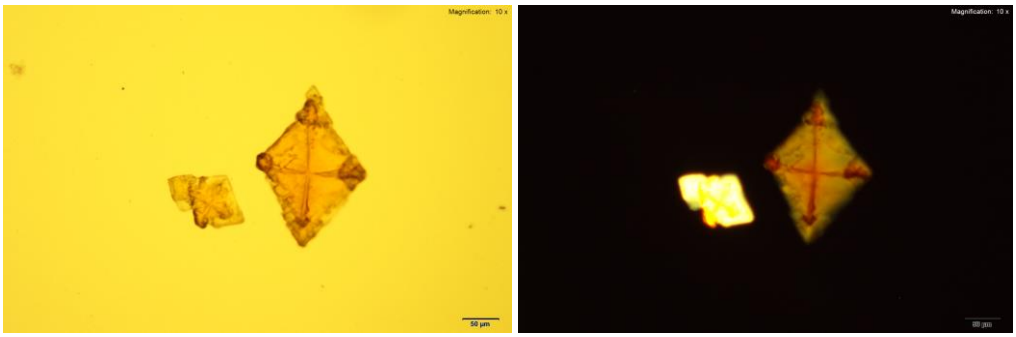
Gold bromide with 4-MeOPP gives three forms of concentration dependent crystals. The immediate formation is of light orange crosses that form abundantly. As with all gold-based reagents, the drop immediately becomes orange with the formation of the crosses when the substance is mixed in. The flat, distinct, orange rectangular plates are formed next alongside the crosses. Large irregular red, tablets grow fewer in number and grow slower. All three forms are bright under crossed polars.

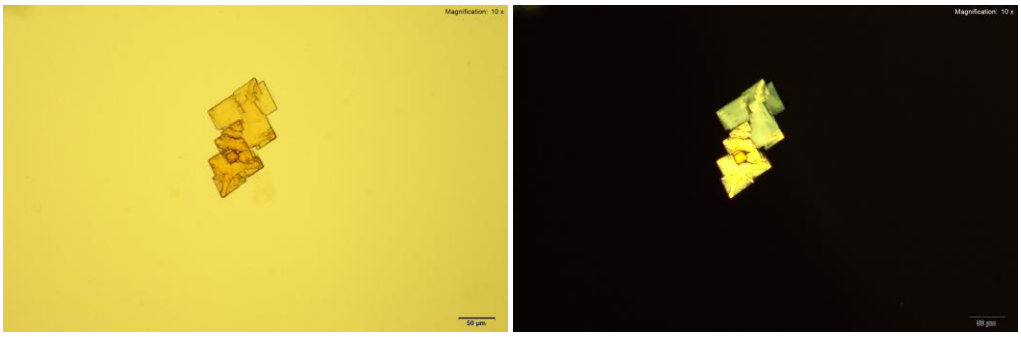
Platinic Chloride (H_2PtCl_6)



4-MeOPP in aqueous or acidic solutions forms long, colorless straw like blades with the platinic chloride reagent. These grow abundantly in the drop. They appear white or in various colors depending on their orientation in crossed polars. At high concentrations, shell-like structures are formed at the edge of the drop. These are unique to this drug-reagent combination. The shell-like crystal has an outer dark edge with a colorless layer that follows. At the core of the crystal are short dark needles arranged in loose clusters. Embedded in the needle clusters are small purple prisms that can be observed at high magnifications. The entire crystal form appears faint white under crossed polars with contrast between different features.

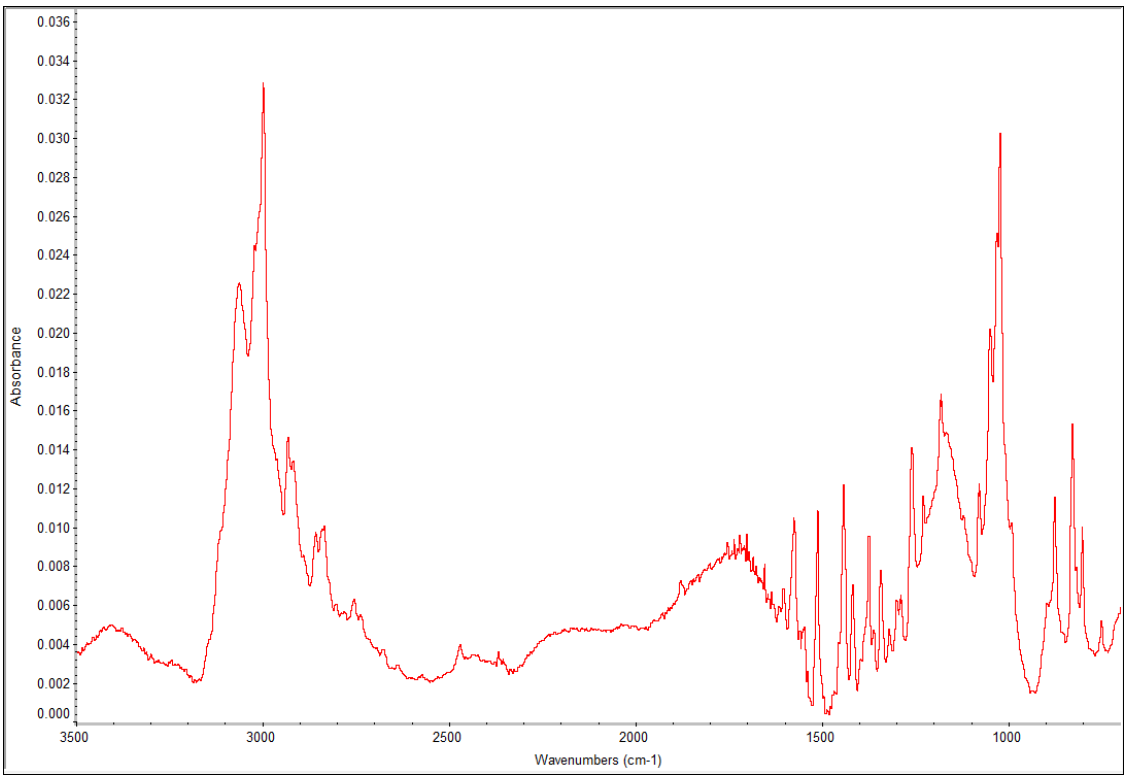
Platinic Bromide (H_2PtBr_6)





Large yellow, square to rectangular tablets that either grow individually or in composites are characteristic of 4-MeOPP with platinic bromide regent.

The large crystals are suitable for IR microspectroscopy.

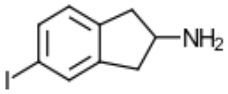


Overall recommended test:

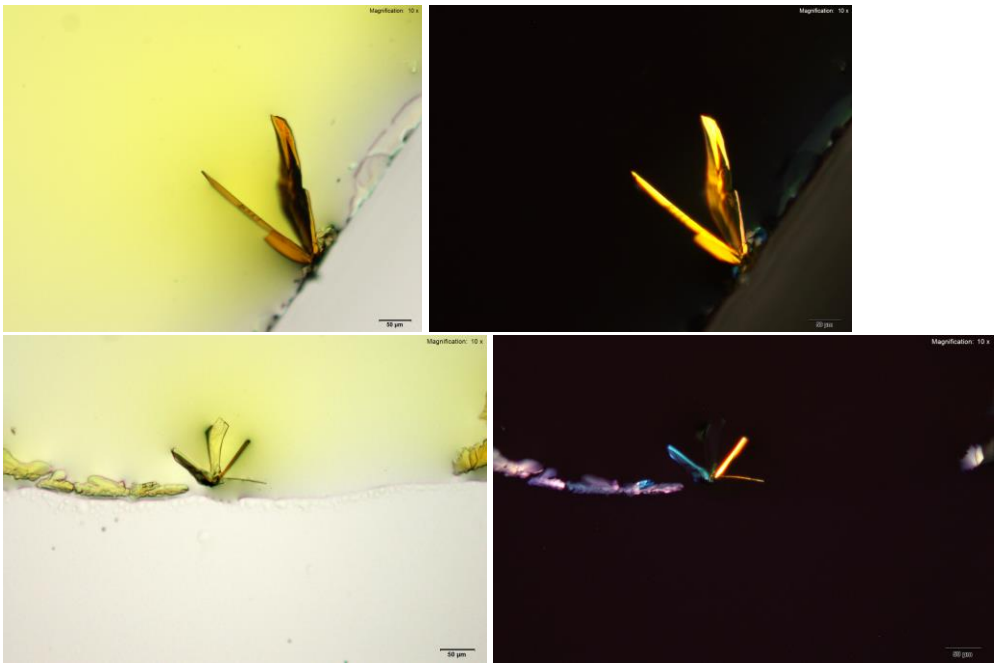
4-MeOPP gives characteristic, easily recognizable crystals with several reagents. The crystal forms observed with the platinic chloride reagent are most distinct. If choosing a combined microcrystalline test and IR microscopy approach, the platinic bromide reagent is recommended.

Aminoindane Class

1. **5-IAI**

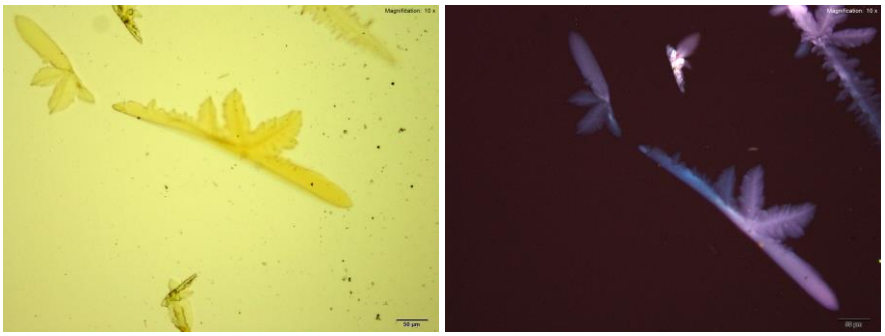


5% Aqueous $HAuCl_4$



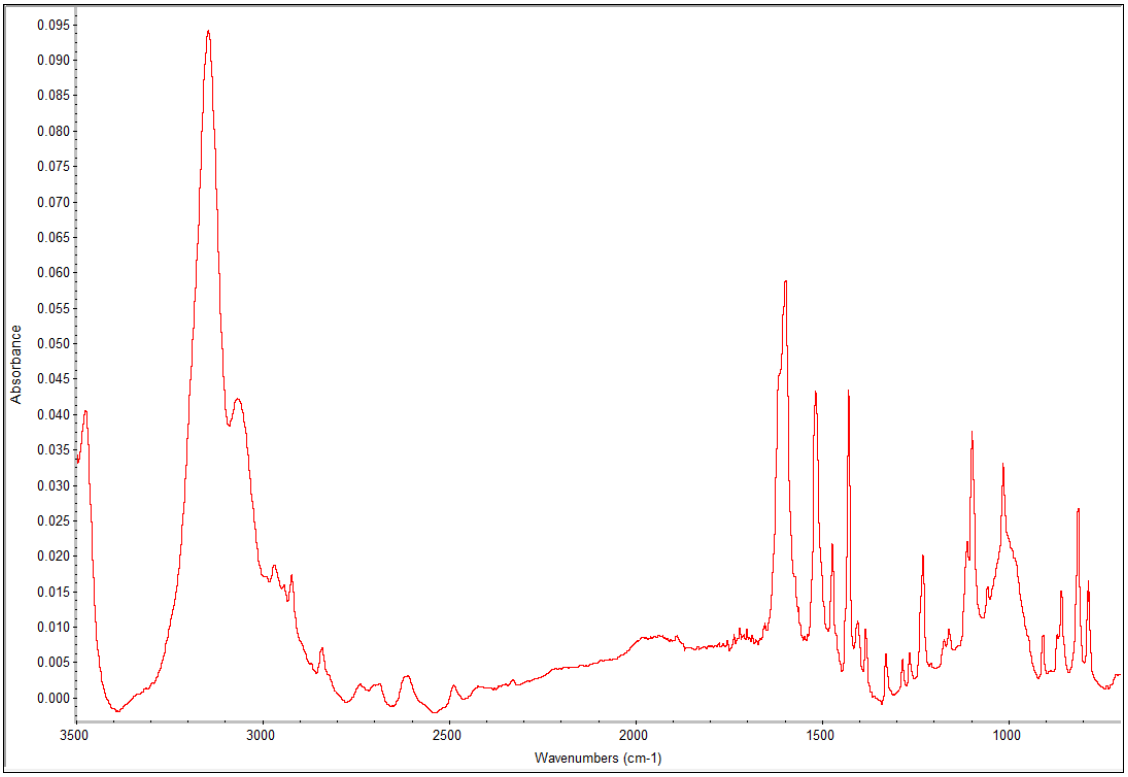
With aqueous gold chloride reagent 5-IAI forms long, thick yellow blades with angled edges. An aqueous solution of 5µg of the substance when mixed with gold chloride gives the most reliable formation. The blades are very bright under crossed polars and can appear yellow to blue depending on the orientation. Also formed along the edges of the drop is a thick leafy crust that appears a light purple under crossed polars.

5% $HAuCl_4$ in 1:2 concentrated H_2SO_4: H_2O

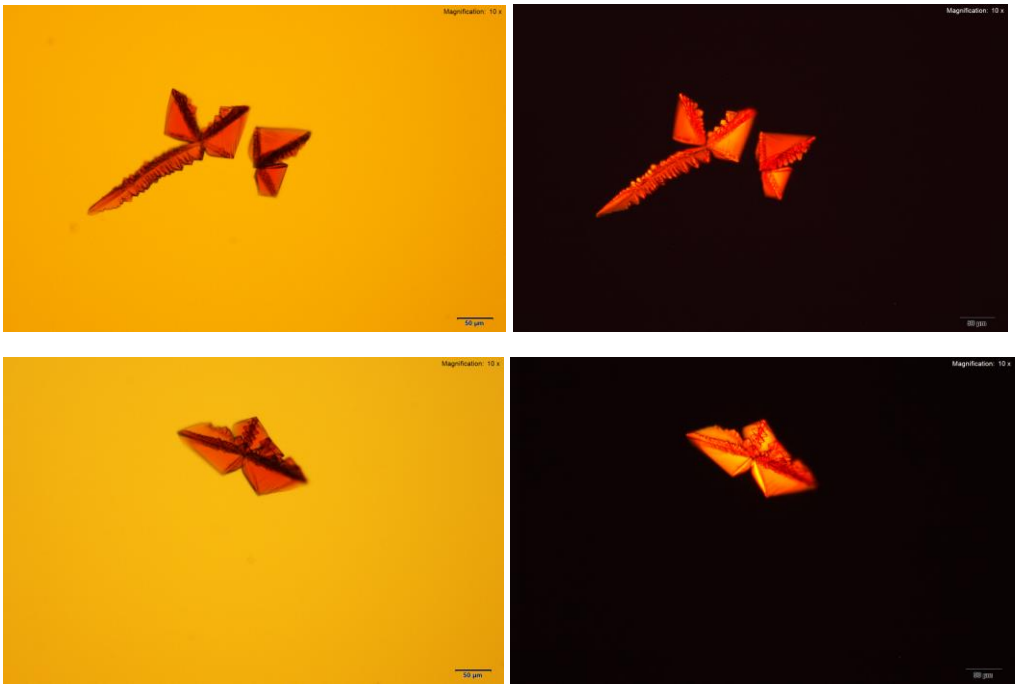


In the acidic gold chloride reagent, 5-IAI forms distinct long blades with serrated edges. The blades are generally branched with one long blade and shorter branches. The blades appear blue to light purple to white under cross polars.

The crystals obtained are sturdy and amenable for IR microspectroscopy.

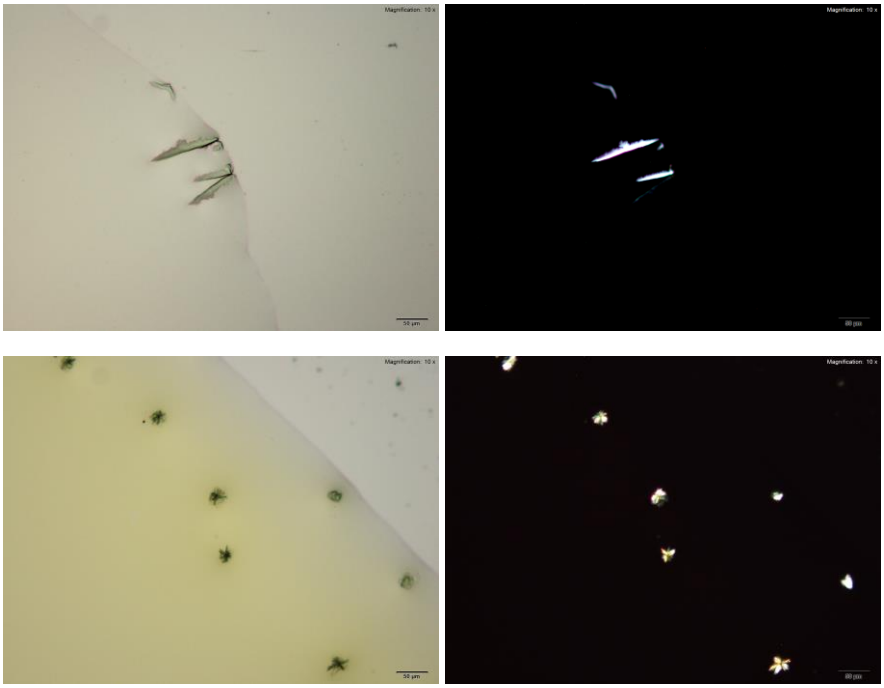


Gold Bromide ($HAuBr_4$)



The crystals formed with a 5-IAI and gold bromide reagent are distinct, very thick, red blades with serrated edges and sharp triangular heads. The crystals grow reliably and fully in 15 -20 minutes. The crystals do not grow abundantly but 2-3 large crystals will be observed in a reaction drop.

Platinic Chloride (H_2PtCl_6)



The crystals with platinum based reagents for 5-IAI are not as characteristic with the gold based reagents. The reactions observed with aqueous platinic chloride very small clusters that appear as dark dots in the drop or light leafy blades at the edge of the drop.

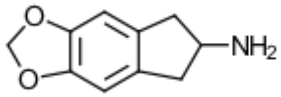
Platinic Bromide (H_2PtBr_6)

5-IAI and platinic bromide reagent make very light feathery X-shaped faint yellow crystals. The crystals appear white to bright yellow under crossed polars. The crystals are suspended in solution and are best photographed with a cover slip.

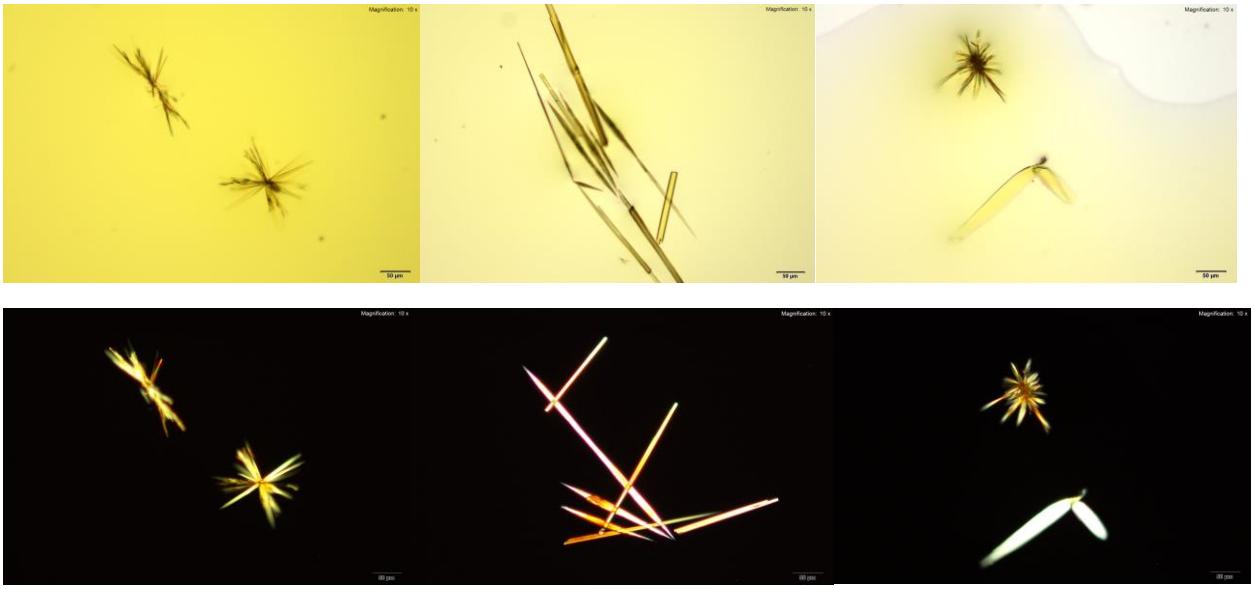
Overall recommended test:

The gold bromide reagent gives the most distinct crystals for 5-IAI but the crystals are very thick and do not easily give informative IR spectra. However, the spectra can be obtained when the crystals are chosen carefully. The acidic gold chloride reagent is recommended for a combined test because of the distinct crystals observed and their suitability for IR microspectroscopy.

2. MDAI

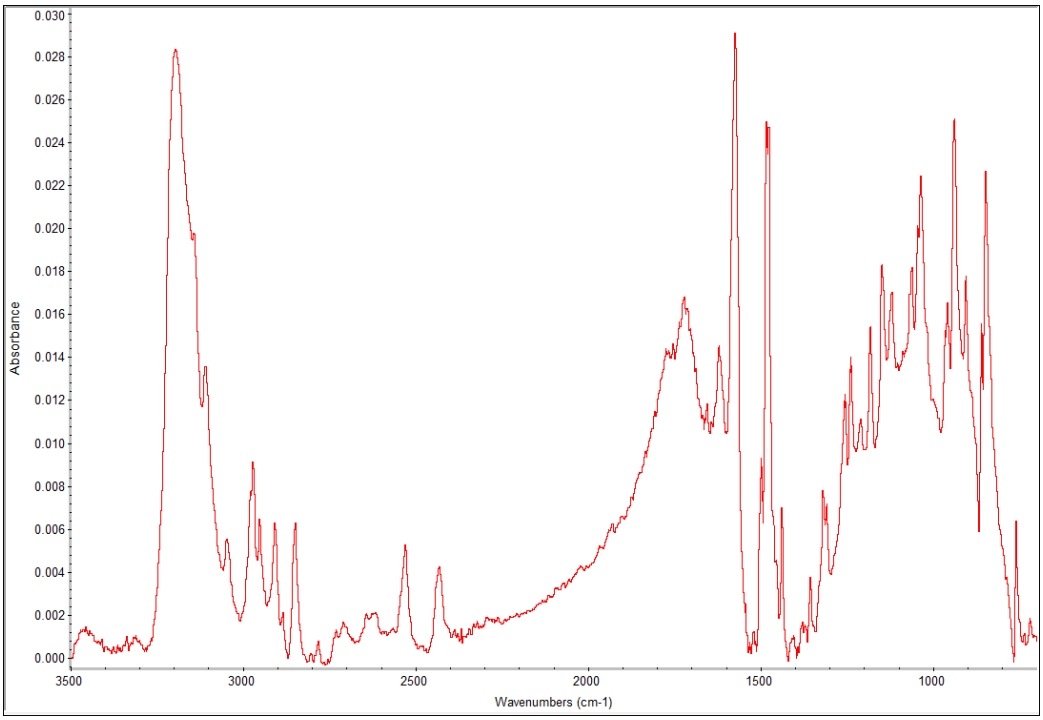


Platinic Chloride (H_2PtCl_6)

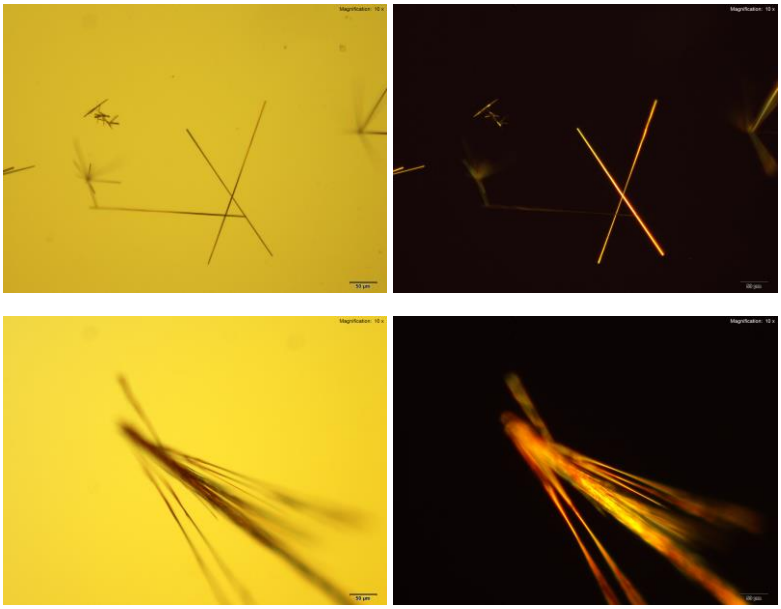


MDAI forms thin flat blades with platinic chloride reagent. In aqueous solutions, the crystals are arranged as clusters of thin yellow blades with pointed tips. These blades appear bright yellow under crossed polars. The blades grow in loose clusters with longer blades in hydrochloric acid solutions. With platinic chloride reagent and MDAI dissolved in 10% acetic acid, the blades in the cluster are broader and larger.

The clusters hold together very well and can be readily studied by IR microspectroscopy. The spectral quality can be improved with careful selection of crystals and pushing multiple crystal forms together.



Platinic Bromide (H_2PtBr_6)



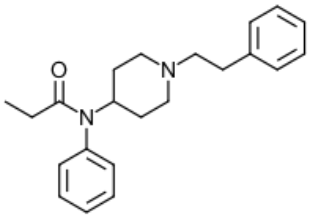
MDAI with the platinic bromide reagent forms long, smooth needles that grow individually or arranged in clusters. The abundant crystals are bright under crossed polars are very characteristic for MDAI.

Overall recommended test:

The platinic bromide crystals are very distinct and easily recognizable but the needles are very thin and do not perform well with IR microspectroscopy. The needles formed with platinic chloride allow for collection of IR spectra because they are thicker and form tighter clusters.

Opioid Class

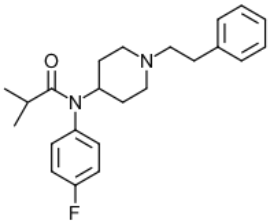
1. **Fentanyl**



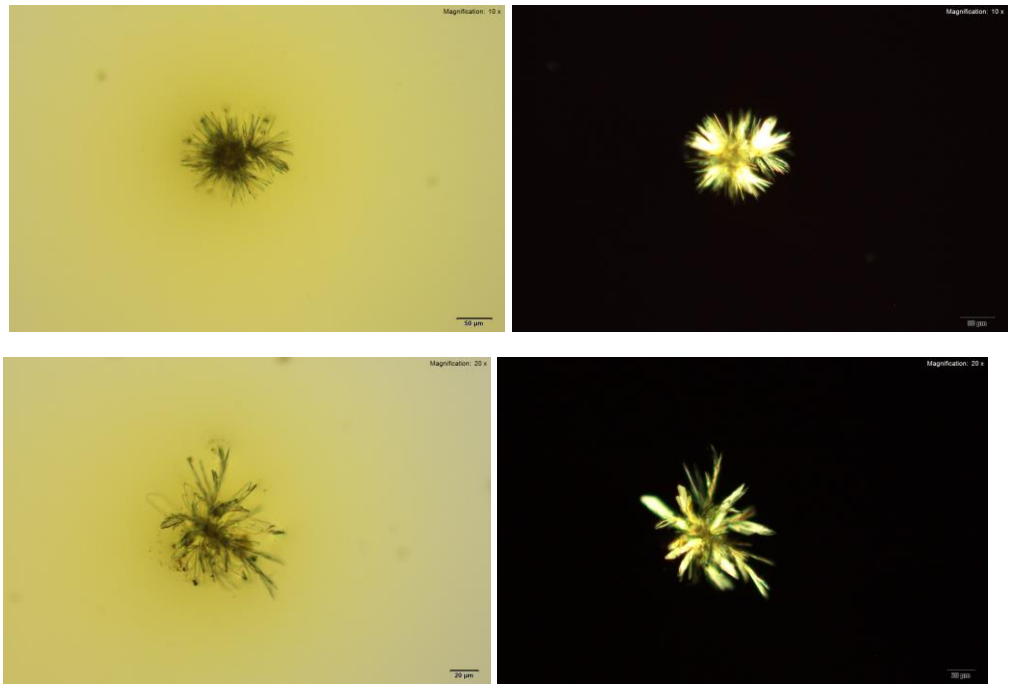
5% Aqueous $HAuCl_4$

Fentanyl forms dark branched comb or feathery clusters with the aqueous gold chloride. In acidic conditions, the crystal forms get smaller. In aqueous conditions, the crystals grow very long and highly branched. The formations are very bright under crossed polars and more details are visible. In light of the safety issues associated with fentanyl, handling this test with powders is not advised. However, these results are given to caution analysts who may encounter these formations as part of mixed street samples.

2. **FIBF**

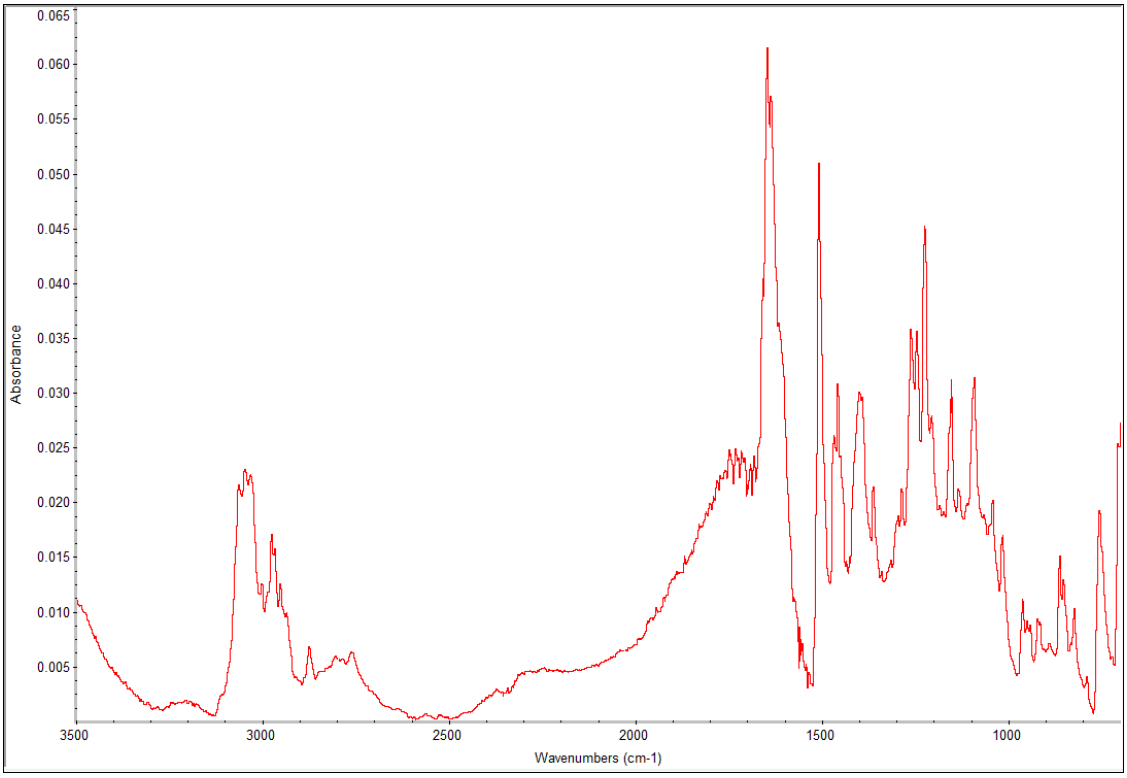


Platinic Chloride (H_2PtCl_6)

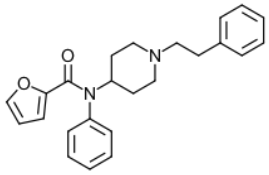


FIBF forms variations of clusters of colorless crystals with platinic chloride. The clusters are tight with short, thin needles in aqueous solutions. The clusters get looser and larger with 10% acetic acid. The individual crystals in the clusters formed are light and feathery blades. The crystals with 10% hydrochloric acid are tight clusters with very short needles that appear as dark dots under 100x magnification.

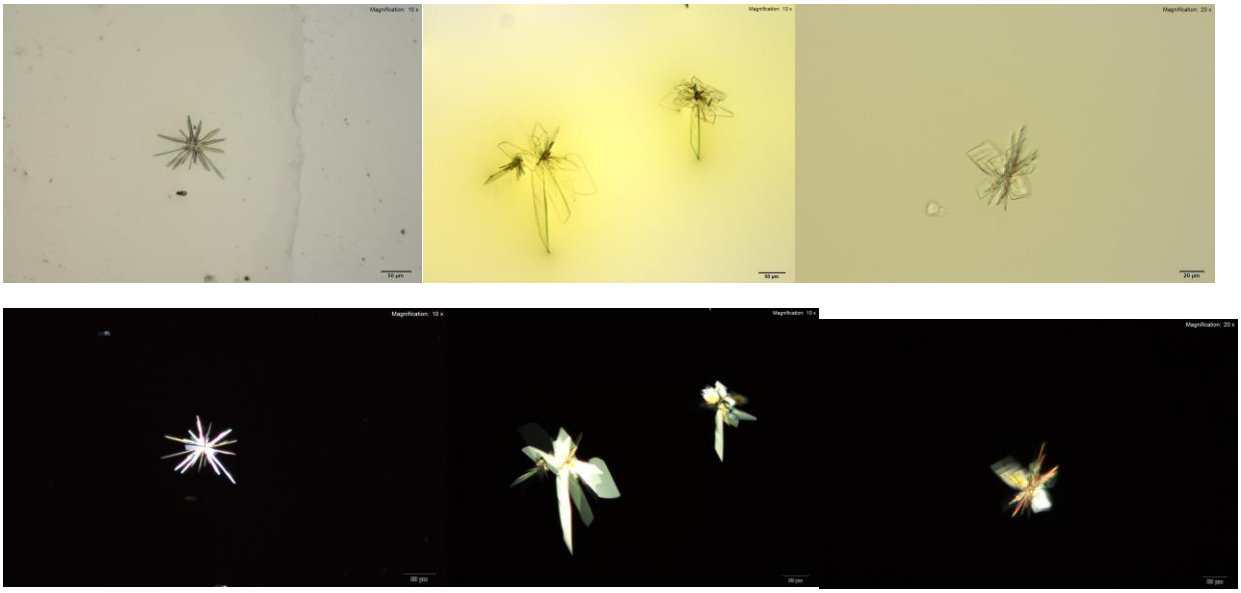
The larger clusters allow for a study of the crystals by IR microspectroscopy.


Absorbance
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

3. **Furanyl Fentanyl**

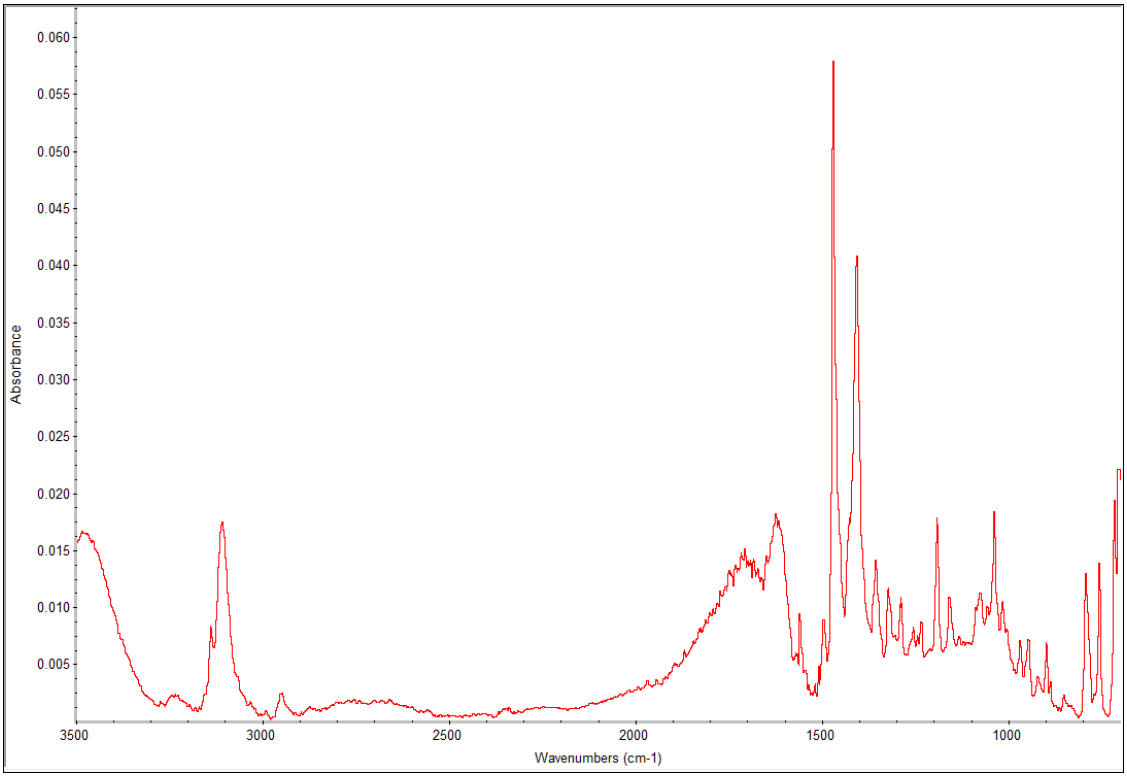


<u>Platinic Chloride (H_2PtCl_6)</u>



Furanyl fentanyl forms clusters of thin, colorless plates with the platinic chloride reagent. The crystal habit differs between the aqueous, 10% hydrochloric acid, and 10% acetic acid solutions. The largest and most reliable crystals are formed with 10% hydrochloric acid. In water, the individual crystals in the cluster are a mix of narrow rod like structures and light feathery plates. The clusters in 10% hydrochloric acid are larger with a mix of short and long plates. The clusters get tighter with short and broad plates in 10% acetic acid. All three forms of the crystals appear bright under crossed polars.

The acetic acid solution crystals are amenable for IR microspectroscopy but the quality of the spectra is not very good.



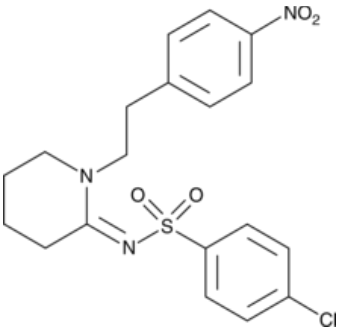
Platinic Bromide (H_2PtBr_6)

Platinic bromide gives characteristic sheaves of short red rods with furanyl fentanyl. The rods appear a bright yellow under crossed polars and form quickly and reliably.

Overall recommended test:

The platinic bromide reagent is recommended for the distinct sheaf formation of the crystals.

4. **W-18**



<u>Gold Bromide ($HAuBr_4$)</u>

W-18 was obtained as a solid and has poor solubility for all the solvents tested in this study. Even when partial dissolution was achieved, no characteristic crystal formation was observed for any of the reagents. With the gold bromide reagent upon standing, yellow feathery branches were observed. These structures are very faintly visible in crossed polars.