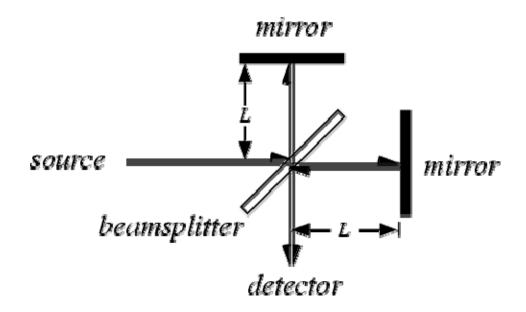
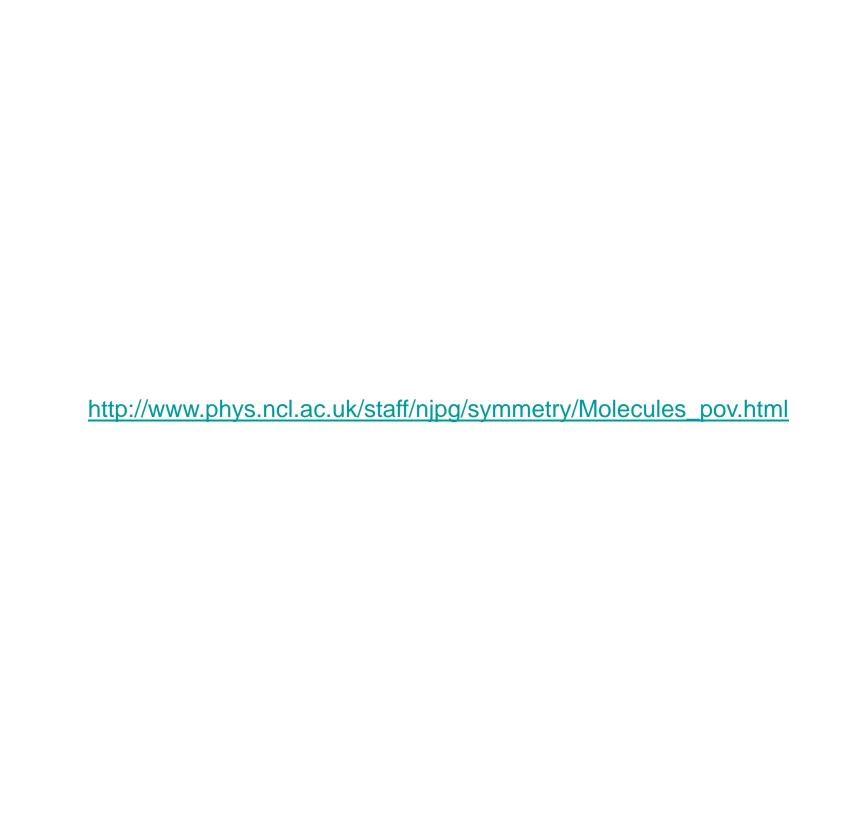
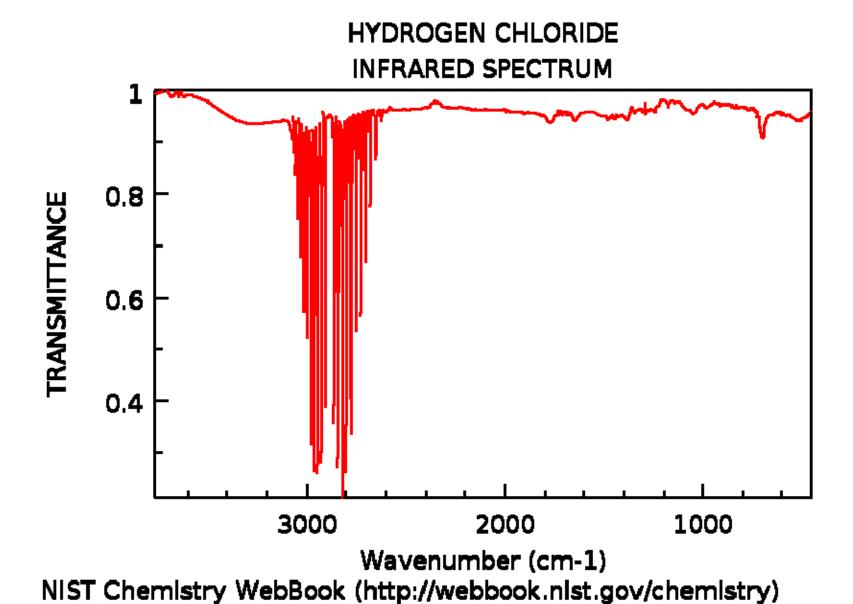


Interferometer







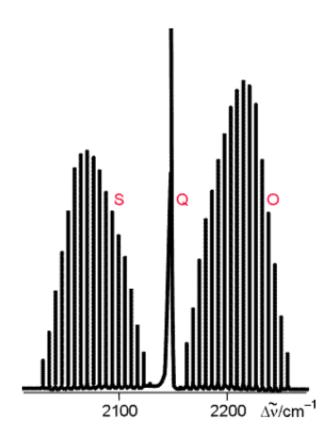
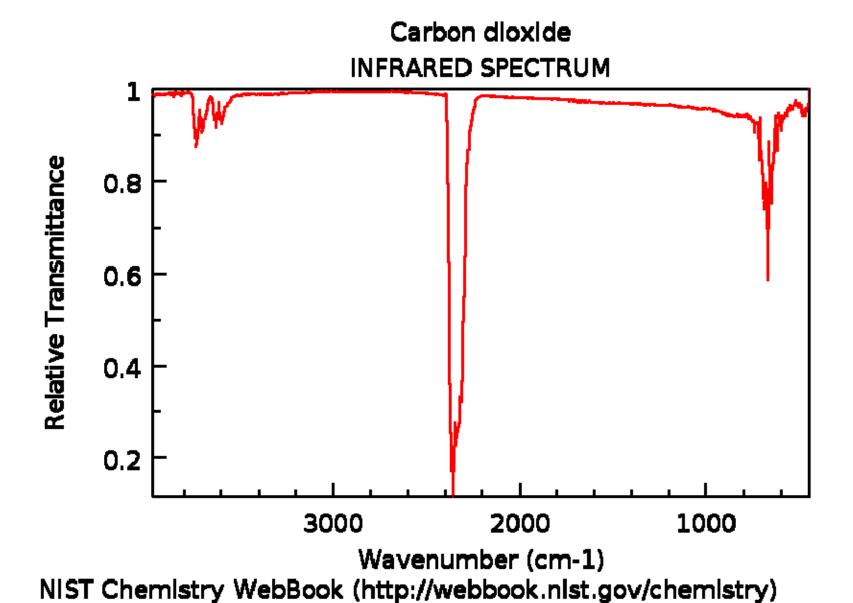
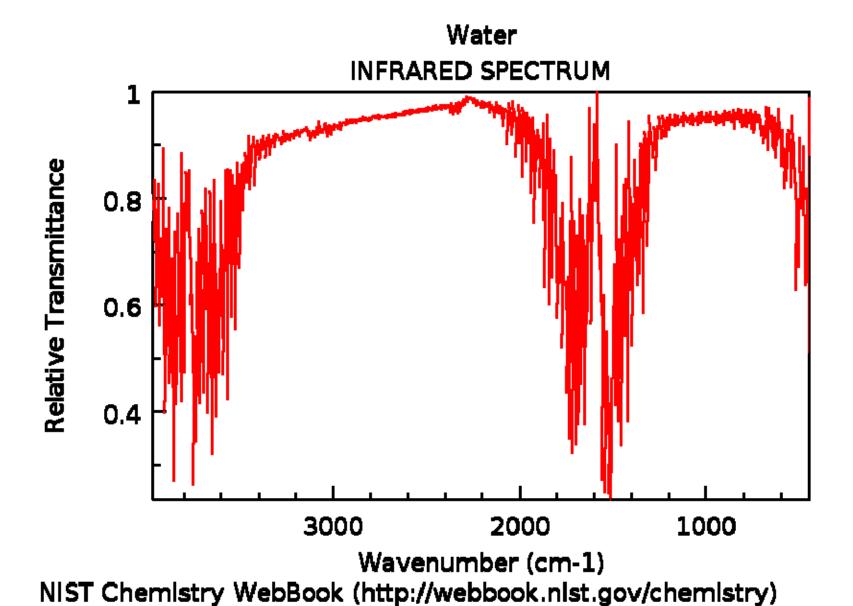


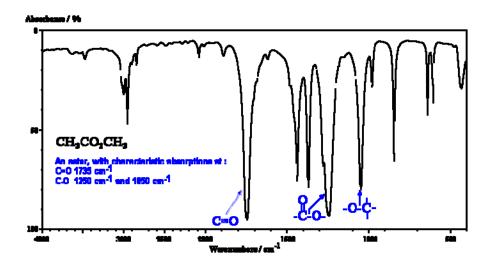
Table 6.3 Typical bond-stretching and angle-bending group vibration wavenumbers ω

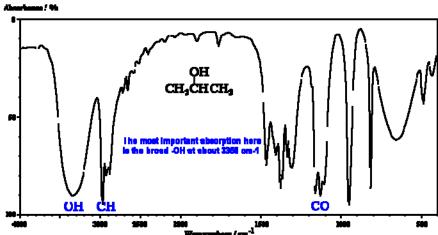
Bond-stre	etching	Bond-st	retching	Angle-bending					
Group	ω/cm ⁻¹	Group	ω/cm^{-1}	Group	ω/cm^{-1}				
≡с−н	3300	C≡N	2100	<u></u> C−H	700				
=c <h< td=""><td>3020</td><td>>c−ғ</td><td>1100</td><td>$= c <_{\rm H}^{\rm H}$</td><td>1100</td></h<>	3020	> c−ғ	1100	$= c <_{\rm H}^{\rm H}$	1100				
except: O=C H	2800	> c−cı	650	$-C \overset{H}{\overset{H}{_{\sim}}}$	1000				
> с−н	2960	⇒c– _{Br}	560	$\searrow \subset \circlearrowleft^H$	1450				
c≡c	2050	-> c⊢	500	c ≦c -c	300				
>c=c<	1650	—о-н	3600a						
>c-c\(-	900	>n−н	3350						
Şsi–si€	430	→ P=0	1295						
>c=o	1700	>s=o	1310						

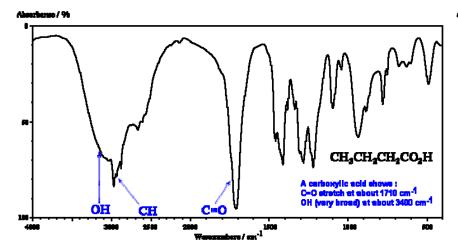
^a May be reduced in a condensed phase by hydrogen bonding.

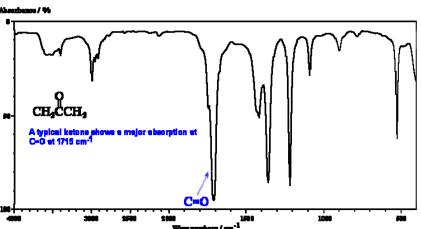


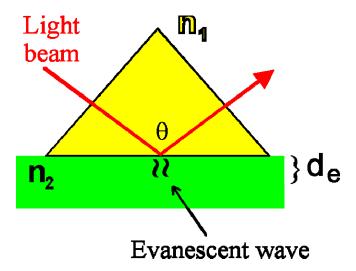


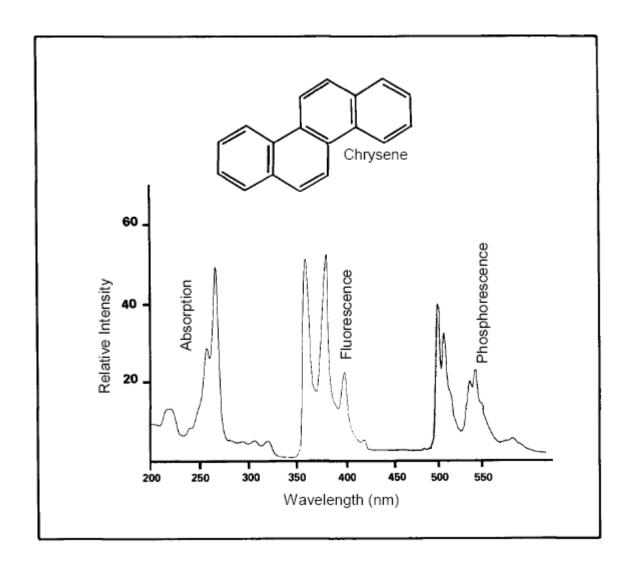




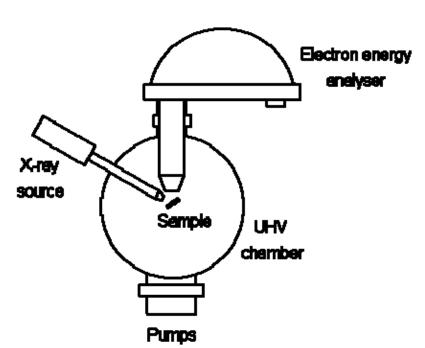


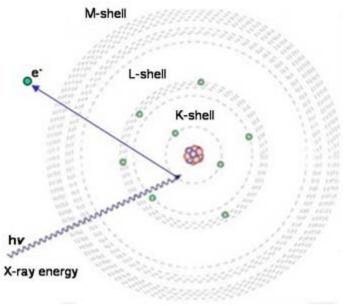




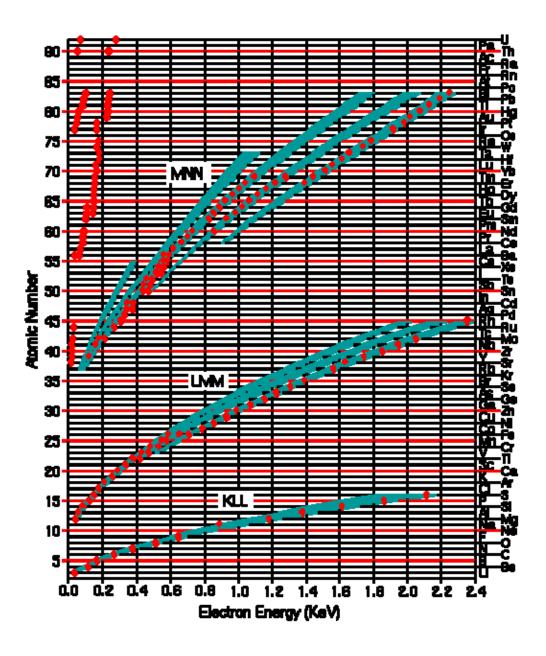


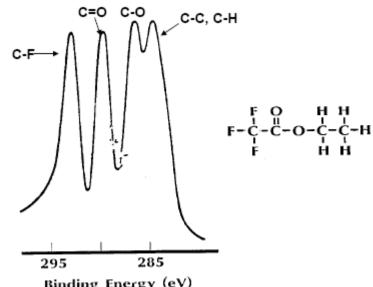
Photoelectron Spectroscopy





The first of the control of the first of the										35.0 eV rencing 18 2 He 1s Hee//Be Hee//																
	2							–		7									13		14	1	5	16	17	
3 Li 1s	4 Be 1s]					umber of E				-	nal for Eler n Oxide or			and of Fla	ment		Γ	5 B1s		C 1s	7 1		8 O 1s	9 F 1s	10 Ne 1s
Li° LiOH 54.9	Be° BeO	ł				E of Al" u	nder Native	Oxide 7	2.9 74.3	Al (2p	o3) BE of	Major Oxi	de Spec	ies in Pure	Oxide			ŀ	B° B2O3 187.5 194.0	284.5	Black 284.4	Kaptor 400.9	398.9	CuO Si 529.7 53		
(1.65) 285.0 531.8 (1.6)	(0.79) (1.73) 286.1 285.0 111.88 531.3		С	(1s) BE of	Al (2p3) FWH Hydrocarbons ble Reference Al (2p3) FW	Captured BE for lo	by Ion Etc n Etched, i	hed Al" 284 Pure Al" 72	1.7 285.0 82 531.1	C (1s O (1s	BE Defi	M of Major Ined to be Major Oxyg of Major C	at 285.0 en Spec	eV ies in Pure	Oxide				(0.87) (2.40) 285.2 285.0 187.8 532.5 (1.03) (2.22)	(0.42)	(1.04)	(1.31) 285.0	(1.10) 285.0 191.3 (1.03)	(0.98) (1.4 284.9 28 934.0 10 (1.42) (1.7	5.0 285.0 28 3.0 291.9 34	5.0 3.1
11 Na 1s	(0.69) (1.47) 12 Mg 2p	Energy res	olution setting	s for pure	ferenced to a oxide data gav	dventitious e FWHM	s hydrocart	ion with C (1s) or Ag (3d5) of i	BE at 285.0e	eV.	The FWH	HM and BE	values two SSI	presented XPS syste	in this tabl	e were all	l obtained l	y	13 Al 2p3		Si 2p3		2p3	16 S 2p	3 17 CI 2 ₁	3 18 Ar 2p3
Na° NaCl 1072.0	Mg° MgO 49.7 49.5	All non-cor C (1s) BEr	ductors were for "hydrocar	analyzed w rbons" on el	ith the Flood-0 lements were	Sun Mesh collected	Screen 0.5 from carbo	5-1.0 mm abow n captured by i	the specim on etched eli	ements.	monochro	solution li omatic Alu	minum)	K-ray source	es which I	have a the	eoretical	ŀ	AI° AI2O3 72.9 74.3	Si°	SiO2	P° 130.13	InP 128.8	S° Mos 164.0 162		
(1.40) 285.0	(0.58) (1.63) 286.5 285.0	Energy res	olution setting	s for ion et	ched elements	gave FV	VHM < 0.50	0 hours after io eV forAg (3d5) eV, and Au(4f7	of ion etche	d Ag*.	elements	can be us	sed as re	eliable sec	ondary en	ergy refer	ion etched ence value are <±0.15	eV/	(0.62) (1.41) 284.7 285.0	(0.57) 285.3	(1.14) 285.0	(0.67) 285.0	285.0	(0.72) (0.3 285.0	285.0 28	5.0 285.0
199.3 (1.19)	49.77 529.8 (0.60) (1.99)	3	4		5	(35) at 12	6	7		8		9		10	1		12		72.82 531.1 (0.41) (1.56)	99.35 (0.45)	532.5 (1.40)		444.7 (0.78)	229 (0.8		2.0
19 K 2p3	20 Ca 2p3	21 Sc 2p3	22 Ti 2		3 V 2p3		Cr 2p3	25 Mn 2p		Fe 2p3	27 C			Ni 2p3	29 C		30 Zn		31 Ga 3d5		Ge 3d5	33 A		34 Se 3d		
K° KI 293.2	Ca° CaO 346.5 347.1	Sc° Sc2O3 398.6 401.9	Ti° Ti	58.7 5	V2O5 12.2 517.3	_	Cr2O3 575.7	Mn° MnO 638.7 641	-	Fe2O3 709.8		779.5	Ni° 852.6	NiO 853.8	Cu° 932.7	932.5	1021.8	ZnO 1021.7	Ga° Ga2O3 18.7 20.7	Ge°	GeO2 33.2	As° 41,8	As2O3 45.1	Se° SeO: 54.8 59.	3 66	8 86.94
(1.11) 285.0	(7) (1.81) 284.6 285.0	(0.9) (1.27) 285.8 285.0	(0.90) (285.2 2	(1.09) (0 285.0 21	0.79) (1.32) 85.0 285.0	(1.05) 284.6	(1.20) 285.0	(1.00) (1.1 286.4 285	(0.90) 0 284.9	(1.32) 285.0	(0.99)	(1.39) 285.0	(1.14)	(1.42)	(1.22) 284.6	(1.10) 285.0	(1.10) 284.8	(1.50) 285.0	(0.70) (1.37) 285.0 285.0	(0.68) 285.0	(1.49) 285.0	(0.67) 284.5	(1.26) 285.0	(0.76) (1.05 284.2 285.	0 (0.5	.0 285.0
619.2 (1.30)	346.5 531.5 (1.07) (1.57)	398.46 530.0 (0.69) (1.33)		530.0 51 (1.18) (0	2.22 530.2 (1.33)	574.37 (0.89)	530.1 (1.24)	638.74 529 (0.89) (1.0		532.9 (1.05)	778.26 (0.85)	530.1 (1.00)		5 529.4 (1.03)	932.68 (0.92)	530.5 (1.01)	1021.76 (0.97)	530.5 (1.11)	18.5 531.3 (0.60) (1.51)	29.28 (0.64)		41.69 (0.67)	532.0 (1.41)	54.90 532. (0.78)	6 29	
37 Rb 3d5	38 Sr 3d5	39 Y 3d5	40 Zr	3d5 4	Nb 3d5	42 1	No 3d5	43 Tc 3d	5 44 F	Ru 3d5	45 R	Rh 3d5	46 P	d 3d5	47 A	g 3d5	48 Cd	3d5	49 In 3d5	50	Sn 3d5	51 S	b 3d5	52 Te 3d	53 I 3d	54 Xe 3d5
Rb° RbOAc	Sr° SrCO3	Y° Y2O3 155.9 156.6			b° Nb2O5 02.1 207.4		MoO3	Tc°	100	RuO2		Rh2O3	Pd° 335.1	PdO		Ag2O		CdO	In° In2O3	Sn° 484.9	SnO2 487.3	_	Sb2O5	Te° TeO 572.8 576		Xe+/Be Xe+/6 9.2 669.6
(1.40) 285.0	(1.63) 285.0	(0.80) (1.25) 286.0 285.0	(0.90) (1.18) (0	.78) (1.14) 85.0 285.0	227.8 (0.66) 285.4	233.1 (1.05) 285.0	CORCUNE	280.0 (0.67)		(0.73)	308.9 (0.80) 285.0	(0.86)	(0.97)	368.2 (0.64) 284.7	367.5 (1.00) 285.0	(0.90)	404.0 (1.38) 285.0	443.8 444.3 (1.08) (1.26) 284.9 285.0	(0.81)		528.2 (1.0) 284.6	529.8 (1.10) 285.0	(1.12) (1.2 284.2 285	7) (1.	9.2 30) (1.13) 5.0 285.0
530.9 (1.6)	531.5 (1.9)	155.92 531.0 (0.62) (1.30)	178,80 5	30.3 20	2.35 530.4 (57) (1.36)	227.94	531.0	R800-	280.11	529.7	307.21	530.5	335.10	530.7	368.28 (0.62)	529.4	405.04	528.6 (1.28)	443.87 529.9 (0.71) (1.19)	485.01	531.1	528.26 (0.80)		572.97 530 (0.83) (1.3	.7 29	3.2
55 Cs 3d5	56 Ba 3d5	57 La 3d5	72 Hf	4f7 7	3 Ta 4f7	74	W 4f7	75 Re 4f	7 76 0	Os 4f7	77 I	lr 4f7	78	Pt 4f7	79 A	u 4f7	80 Hg	4f7	81 TI 4f7	82	Pb 4f7	83	Bi 4f7	84 Po 4f7	85 At 41	7 86 Rn 4f7
Cs° CsCl 724.6	Ba° BaOAc	La° La2O3	Hf° H	10		-	WO3	Re° Re20		OsO4		lrO2		PtO2	_	Au2O3	-	HgO	TI° TI2O3	Pb°	PbO	_	Bi2O3			
(2.08) 285.0	780.0 (1.80) 285.0	834.7 (3.0) 285.0	(0.63) (285.7 2	1.26) (0 285.0 28	1.8 26.8 .80) (1.12) 35.0 285.0	285.3	(1.01) 285.0	40.3 46. (0.67) (1.6- 285.3 285.	(a) (b)		284.4	62.0 (0.98) 285.0	71.0 (0.96) 284.3	(1.16) 285.0	84.1 (0.83) 284.1	88.1 (1.12) 285.0	285.0	(1.06) 285.0)	117.8 117.4 (0.97) (1.01) 285.1 285.0	136.9 (0.67) 284.9	(1.10) 285.0	157.0 (0.73) 284.6	158.8 (1.11) 285.0	2 adjoactive	and the control of th	odicactive
199.2	531.4 (1.83)	529.2 (1.6)			1.78 531.0 .56) (1.46)		530.6 (1.27)	40.30 532 (0.54) (1.5				530.2 (0.97)	71.15 (0.88)		83.98 (0.68)	531.6 (1.13)			117.77 528.9 (0.66) (1.10)	136.95 (0.63)	528.9 (1.07)	157.05 (0.62)	529.6 (1.58)	Ψ.	- Qu	dry.
87 Fr 417	XPS International LLC: www.xpsdata.com B. Vincent Crist																									
and the last	-active	detive																							Last Update	
Radio	Q. addito	Rada	3 I	58 Ce 3 Ce° Ce(Pr 3d5 Pr2O5	60 N		Pm 4d5 Pm2O3	62 Sr Sm° S	m 4d5 m2O3	63 Eu	u 4d5	64 G	d 4d5 3d2O3	65 T	b 4d5 Tb3O7	66 Dy	- 1	Ho 4d5		r 4d5 Er2O3		m 4d5 70 Tm2O3 Yb ⁴	- 1	Lu 4f7
			"	88	2.1 (931.98	933.1	(980.86)	FIII			(134.9)		135.6	_	(1186.8)	145.9	(149.9)	152.4 (156.1) 159.8	161.3	167.7	168.5	175.3	176.3 1.6	7 184.9 7.	0 8.4
				(2. 28 52	5.0	(4.4) 285.0 528.2	118.0	0	advoactor		(10) 285.0 531.7	284.3 128.18	(3.7) 285.0 529.2	281,4 140,33	(5.4) 285.0 529.0	146.02	(7) 285.0 529.5		(7) 285.0 529.2 159.58	((6.0) 285.0 529.3	167.25	(3.8) 285.0 529.2	285.3 175.37		- (3.4) - 5.6 285.0 28 39) 529.4 7	
			L	(2.0	00) (2.17)	(1.4)	(1.80)	· · · · · ·		(2.57)	(2.4)	(1.08)	(1.4)	(1.02)	(1.7)	(1.33)	(1.6)	(1.88)	(1.7) (1.50)	(1.7)	(1.93)	(1.9)	(1.92)	(1.7) (0.8	31) (2.0) (0.	(1.8)
				90 Th 4	************	Pa 4f7	92 U		3 Np	94	Pu	95	Am	96	Cm	97	Bk	98	Cf 99	Es	100	Fm	101	Md 1	02 No	03 Lr
Th¹ ThO2 U¹ U203									0																	
					9,000	OHCH!		ai	dicate tra-	2300	activa	Cation	dia	0.96	active.	Q actio	ALCO .	Gadioni	crast	CHChi	Q366	ach.	2000	RCH.	and scale California	Radioach.
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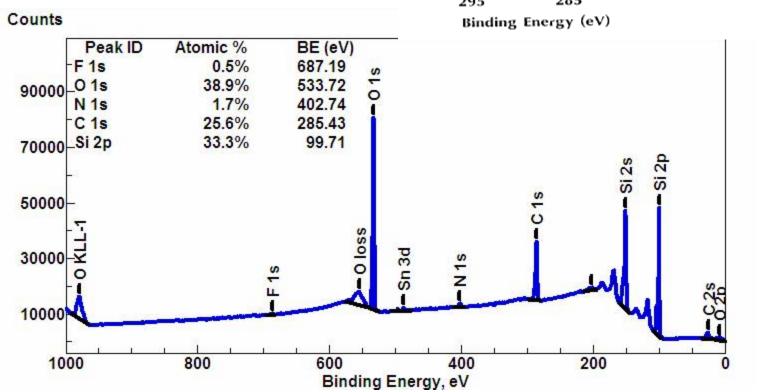


Table of Characteristic IR Absorptions

frequency, cm ⁻¹	bond	functional group
3640–3610 (s, sh)	O-H stretch, free hydroxyl	alcohols, phenols
3500-3200 (s,b)	O-H stretch, H-bonded	alcohols, phenols
3400-3250 (m)	N-H stretch	1°, 2° amines, amides
3300-2500 (m)	O-H stretch	carboxylic acids
3330–3270 (n, s)	-C≡C-H: C-H stretch	alkynes (terminal)
3100-3000 (s)	C-H stretch	aromatics
3100-3000 (m)	=C-H stretch	alkenes
3000-2850 (m)	C-H stretch	alkanes
2830-2695 (m)	H-C=O: C-H stretch	aldehydes
2260–2210 (v)	C≡N stretch	nitriles
2260–2100 (w)	-C≡C- stretch	alkynes
1760–1665 (s)	C=O stretch	carbonyls (general)
1760–1690 (s)	C=O stretch	carboxylic acids
1750–1735 (s)	C=O stretch	esters, saturated aliphatic
1740–1720 (s)	C=O stretch	aldehydes, saturated aliphatic
1730–1715 (s)	C=O stretch	α , β -unsaturated esters
1715 (s)	C=O stretch	ketones, saturated aliphatic
1710–1665 (s)	C=O stretch	α , β -unsaturated aldehydes, ketones
1680–1640 (m)	-C=C- stretch	alkenes
1650–1580 (m)	N-H bend	1° amines
1600–1585 (m)	C–C stretch (in–ring)	aromatics
1550–1475 (s)	N-O asymmetric stretch	nitro compounds
1500-1400 (m)	C-C stretch (in-ring)	aromatics
1470–1450 (m)	C–H bend	alkanes
1370–1350 (m)	C-H rock	alkanes
1360–1290 (m)	N-O symmetric stretch	nitro compounds
1335–1250 (s)	C–N stretch	aromatic amines
1320–1000 (s)	C–O stretch	alcohols, carboxylic acids, esters, ethers
1300–1150 (m)	$C-H$ wag $(-CH_2X)$	alkyl halides
1250–1020 (m)	C-N stretch	aliphatic amines
1000–650 (s)	=C-H bend	alkenes
950–910 (m)	O–H bend	carboxylic acids
910–665 (s, b)	N–H wag	1°, 2° amines
900–675 (s)	С–Н "оор"	aromatics
850-550 (m)	C-Cl stretch	alkyl halides
725–720 (m)	C–H rock	alkanes
700–610 (b, s)	-C≡C-H: C-H bend	alkynes
690–515 (m)	C-Br stretch	alkyl halides

m=medium, w=weak, s=strong, n=narrow, b=broad, sh=sharp



Micro-Crystal Identification Tests for Morphine, Heroin, Dilaudid, and Cocaine

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MICRO-CRYSTAL IDENTIFICATION TESTS FOR MORPHINE, HEROIN, DILAUDID, AND COCAINE

Charles C. Fulion† and John B. Dalton‡

In the identification of small amounts of suspected drugs the most valuable tests are of two kinds: color tests on the spot-plate, and crystal tests under the microscope. The former are especially useful for compounds of phenolic character, such as adrenalin, arbutin, aspirin, and the opium alkaloids. The micro-crystal tests are particularly useful for amines, such as all alkaloids, and amides, such as phenacetin and acetanilid. This method of identification by recognition of characteristic crystals under the microscope was begun by Wormley (1), Lyons (2), Behrens (3), and others, and developed in more recent years for the alkaloids especially by Grutterink (4), Stephenson (5), and Amelink (6).

A number of the more recently developed tests, including some which were previously unpublished, will be described in the course of this paper. The photomicrographs¹ which accompany the text show the crystals of the four alkaloids, morphine, heroin, dilaudid, and cocaine, resulting from several of these tests. Some of the micro-crystals have been previously described (11, 12, 13, 14, 15), but until now no photographs of them have appeared in any of the literature.

The crystal tests for a particular alkaloid require selected reagents which

will most readily give highly characteristic crystals with the alkaloid in question (7, 8, 9, 10), for the usual result with a reagent and an alkaloid taken at random is an amorphous precipitate that does not crystallize at all. Generally the chosen tests are such that the crystals can be definitely recognized by mere inspection under a low power microscope (50 to 100 X). However, since many of the crystals are highly pleochroic with polarized light, or highly birefringent and beautifully illuminated with crossed nicols, it is best to use a polarizing microscope whenever available.

Either of two methods for making a crystalline test are generally employed. Method A involves dissolving the alkaloidal salt in water and adding the reagent, while with Method B the reagent is added directly to the solid alkaloid. A more detailed discussion of both procedures follows.

With Method A about 0.2 mg. or less of the alkaloidal salt is dissolved in one drop (about 0.04 cc.) of water on the microscope slide. (If the free alkaloid has been obtained by extraction or otherwise it is dissolved in dilute acid and a drop put on the slide.) One drop of a selected reagent is then added by letting it fall from a 1 cc. pipette. The precipitating compound may be in

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¹ The photomicrographs which accompany this article were prepared by John B. Dalton.

aqueous solution or in concentrated or very strong acid.

With Method B a drop of the precipitating reagent (ordinarily in strong acid) is added directly to about 0.1 mg. or less of the solid, powdered alkaloid or its salt and a cover-glass is placed over the material immediately. The alkaloid dissolves and is precipitated at varying concentrations. In this method the test-drop can be a concentrated acid (hydrochloric or phosphoric), or acid of a concentration at which the crystals form best and which might be difficult to obtain by mixing two solutions as in Method A. Formulas are given in this paper for making up directly the reagents used for Method B, but in practice they are usually obtained by suitable dilutions (to three times the volume) of the more concentrated reagents kept for Method A.

With both methods in a successful test a precipitate appears, either at once or after a few minutes' standing. Usually this precipitate is amorphous at first but crystallizes in a short time, although in some cases it is crystalline from the start. If the test is unsuccessful because of amine impurities the crystals may be distorted so as to be unrecognizable, but the more common unsuccessful result is an amorphous precipitate which fails to crystallize.

In the case of a mixture of alkaloids or amines some kind of separation—by extraction methods, for instance—is often necessary before micro-crystal tests can be applied. However, inert adulterants or dilutents, such as powdered sugar or lactose, usually do not interfere at all, except of course that

more of the material must be used to make the test.

A number of the reagents mentioned below can be used for many other alkaloids besides morphine, heroin, dilaudid, and cocaine. Thus, Stephenson's work on 51 alkaloids (5) shows that gold chloride gives especially good tests for 10 of them, platinum chloride for 6, and Wagner's reagent for 6. The newer reagents presented herein have not been so thoroughly studied. bromide in half-concentrated hydrobromic acid is certainly quite useful, yet perhaps is not as satisfactory for general use as gold bromide in concentrated hydrochloric acid, which may be the best of all identifying reagents for alkaloids.

Tests for Morphine

The best-known tests for morphine are those made by Method A with Marme's reagent (5, 14) and Wagner's reagent (16, 5, 14). The latter test can be varied, using Method B, to identify less than a gamma (a millionth of a gram) of morphine (15). In addition to these better known tests are several which are quite valuable.

Gold Bromide in Half-Concentrated Hydrobromic Acid. A form of this test in which gold bromide is used in concentrated HCl has been previously described (14). If the reagent is applied to the aqueous solution of morphine (Method A), threads in rosettes form gradually from the amorphous precipitate. When added to an HCl solution of morphine, or applied directly to the solid alkaloid or its salt (Method B) the reagent gives, upon standing, salmon-colored or brownish-yellow-orange

plates. These are often feathered, or form rosettes of blades or irregular plates, while with polarized light they show dichroism of pale yellow to dark red.

Now it has been found that in using Method B half-concentrated acid gives the most rapid and best crystallization. Half-concentrated HCl may be used, but HBr is better except for the deeper color of the solution. Two types of crystals are formed, needles and dichroic plates (Figure 1). As the test is quite sensitive, only a very little morphine should be used.

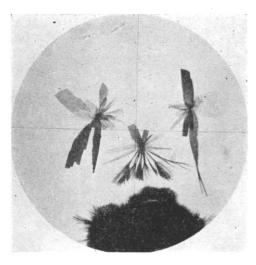


FIGURE 1

Morphine with $HAuBr_4$ in half-concentrated HBr. Method B. (Polarized light.)

The reagent is prepared according to the following formula: Gold chloride crystals (HAuCl₄3H₂O)—1 gram; HBr (40%)—30 cc; Water—30 cc.

This reagent also gives an exceptionally fine test for novocaine and may likewise be used for the identification of cocaine, nupercaine, heroin, cotarnine, creatinine, hyoscyamine, scopolamine, theophylline, and other alkaloids.

In general, these different crystals can be distinguished at a glance from those of morphine and from each other although dichroic plates with scopolamine show some resemblance to those with morphine.

Platinum Bromide in Hydrobromic Acid, and Bromo-Chlorides in Hydrobromic - Hydrochloric Acid.Method B, with various ratios of HBr to HCl, morphine forms a series of different platinum crystals, but these platinum reagents give no morphine crystals in plain water, requiring instead about 60% to 100% of concentrated acid. The different types of crystals were first studied with empirical mixtures, and some of them were found to correspond quite definitely to simple molecular ratios of HBr and HCl. There are some distinctions between the crystals for each of the six possible compounds from H,PtBrCl₅ to H,PtBr₆. H,PtBrCl₅ gives the most rapid crystallization, H₂PtBr₆ the slowest.

The reagents, their formulas, and a description of the crystals are given below which, to the writers' knowledge, have never previously been described. In all instances these reagents should stand for a few days after being mixed in order to bring them to final equilibrium. The first three (1a, 1b, 2, and 3) are much more satisfactory than the last three.

1a. $H_2PtBrCl_5$ in HCl. Platinum chloride crystals (H_2PtCl_6 $6H_2O$)—1 gram, HBr (40%)—2 cc., HCl (conc.)—46 cc., Water—12 cc. The resulting crystals are light yellow, rectangular plates, which are very highly birefrin-



FIGURE 2 $^{\circ}$ Morphine with $H_2PtBrCl_5$ in HCl. Method B. (Crossed nicols.)

gent and form gradually on standing with little or no amorphous precipitation. Figure 2 shows these crystals photographed with crossed nicols.

1b. $H_2PtBrCl_5$ in 1 HBr:5 HCl. $H_2PtCl_6.6H_2O-1$ gram, HBr (40%)—12.5 cc., HCl (conc.)—35.5 cc., Water-12 cc. The crystals are similar to the preceding but deeper yellow and not so perfectly formed, and there is an amorphous precipitate before crystallization. The test, however, is a little more sensitive than with 1a.

- 2. $H_2PtBr_2Cl_4$ in 1 HBr:2HCl. H_2PtCl_6 $6H_2O-1$ gram, HBr (40%)-27.5 cc., HCl (conc.)—29.5 cc., Water—3 cc. The crystals are orange-yellow and have pointed rather than square-cut ends—that is, they have become hexagons which are more or less elongated.
- 3. H₂PtBr₃Cl₃ in 1 HBr:1 HCl. H₂PtCl₆·6H₂O—1 gram, HBr (40%)— 35.5 cc., HCl (conc.)—18.5 cc., Water—6 cc. The crystals are orange plates,

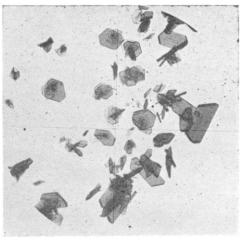


FIGURE 3

Morphine with $H_2PtBr_3Cl_3$ in 1 HBr : 1 HCl.

Method B.

usually hexagonal but sometimes diamond or coffin-shaped. (Figure 3.)

- 4. $H_2PtBr_4Cl_2$ in 2 HBr:1 HCl. H_2PtCl_6 $6H_2O-1$ gram, HBr (40%)- 42.5 cc., HCl (conc.)—11.5 cc., Water—6 cc. This gives crystals which are orange-red, thick, and hexagonal, coffin-shaped, or diamond-shaped plates, smaller than with the preceding reagent. A few rosettes or sheaves of needles (morphine with H_2PtBr_6) may also form.
- 5. H_2PtBr_5Cl in 5 HBr:1 HCl. $H_2PtCl_6\cdot 6H_2O-1$ gram, HBr (40%)—52 cc., HC1 (conc.)—5 cc., Water-3 cc. The characteristic crystals are quite small diamond-shaped plates varying in color from orange to red. Usually a considerable part of the precipitate crystallizes in the morphine- H_2PtBr_6 needles instead.
- 6. H_2PtBr_6 in HBr. $H_2PtCl_6 \cdot 6H_2O$ —1 gram, HBr (40%)—48 cc., Water—12 cc. The crystals are small rosettes of dark red needles, nearly opaque to



Figure 4 Heroin with ${\rm HgI_2}$ in 10% HCl. Method B.

transmitted light, but highly reflecting when illuminated with light coming obliquely from above.

Tests for Heroin

In the past the favorite test for heroin has been the one made with platinum chloride (16, 5). However, heroin in the illegal traffic is nowadays so grossly adulterated that this test often fails. The adulteration is chiefly with milk sugar, powdered sugar, mannitol, or other inert material, but novocaine, morphine, or other alkaloids, are sometimes present in small proportion, hence the need for more sensitive heroin tests and, if possible, some less affected by impurities.

Other old tests are with sodium carbonate, sodium phosphate, mercuric chloride, and picric acid (5). The crystals of free heroin, obtained with sodium carbonate or tri-sodium phosphate, are characteristic, but the test is even less sensitive than with platinum chloride. Mercuric chloride is more

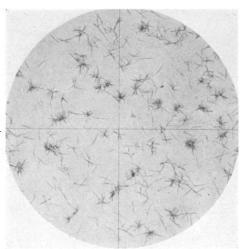


FIGURE 5
Heroin with HAuBr $_4$ in diluted H $_2$ SO $_4$. Method A. (Extremely dilute solution.)
sensitive but less characteristic while the picric acid test, modified to the extent of using sodium picrate (13), is very sensitive.

The best tests now known for heroin are made with mercuric iodide in hydrochloric acid (13), gold bromide in sulfuric acid (8), and gold chloride in sulfuric acid (8). Below are descriptions and the first published photographs of these crystals (Figures 4, 5, and 6). Gold bromide in concentrated hydrochloric acid, and gold chloride in concentrated hydrochloric acid, can also be used (13), but do not form crystals as readily as the sulfuric acid reagents, nor are they as sensitive.

Mercuric Iodide in 10% Hydrochloric Acid. HCl (conc.)—27 cc., Water—73 cc., HgI₂—to saturation. This reagent was originally used by Method A, but it has been found to give a more sensitive test by Method B. As the crystals are colorless, care should be taken that

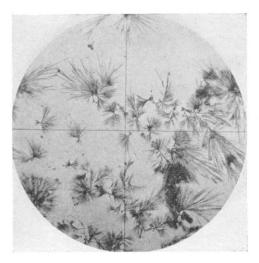
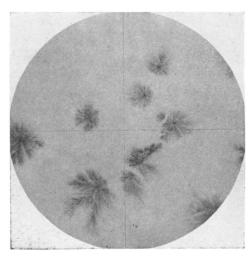


FIGURE 6

Heroin with HAuCl₄ in diluted H₂SO₄. Method A. they are not confused with crystals of undissolved material. They are slender blades and branching threads (Figure 4), and are only feebly illuminated when viewed with crossed nicols, thus appearing as pale gray "ghosts".

Gold Bromide in Diluted Sulfuric Acid. Gold chloride crystals (HAuCl₄·3H₂O)—1 gram, HBr (40%)—1.5 cc., Diluted H₂SO₄ (H₂SO₄—2 vol., Water —3 vol.)—to make 20 cc. By using Method A this reagent gives the most sensitive crystal test known for heroin, and the best results can be obtained with a minute amount of the sample. The crystals so formed are fine needles, mostly scattered. (Figure 5.)

Gold Chloride in Diluted Sulfuric Acid. Gold chloride crytals (HAuCl₄· 3H₂O)—1 gram, Diluted H₂SO₄ (H₂SO₄ —1 vol., Water—1 vol.)—20 cc. Again Method A is used. This reagent, however, is much less sensitive than the preceding, but still superior to older



 $\begin{array}{c} \text{Figure 7} \\ \text{Dilaudid with } \text{H_2PtB}_6 \text{ in diluted } \text{H_2SO}_4. \\ \text{Method A.} \end{array}$

tests. The resulting crystals are rosettes of needles and are illustrated in Figure 6.

Tests for Dilaudid

Dilaudid (dihydromorphinone hydrochloride) is a synthetic derivative of morphine now used in medicine, and may also be used in drug addiction. The established test is with sodium nitroprusside, adding a small solid crystal of the reagent to a drop of aqueous dilaudid solution (6). In addition the following new tests are of value.

Platinum Bromide in Diluted Sulfuric Acid. Platinic chloride crystals (H₂PtCl₆·6H₂O)—1 gram, HBr (40%) —1.7 cc., Diluted H₂SO₄ (H₂O—2 vol., H₂SO₄—3 vol.)—to make 20 cc.

Using Method A one drop of the reagent is applied to a drop of aqueous dilaudid solution giving orange-yellow mossy rosettes of very vague structure. Whether definitely crystals or not,

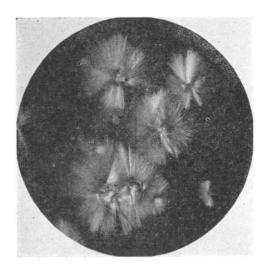


FIGURE 8
Dilaudid with H_2PtBr_6 in $HBr-H_2SO_4$ solution.
Method B. (Reflected light).

these are quite characteristic in appearance. (Figure 7.) Aqueous platinic bromide reagent gives a similar result, but with a heavier amorphous precipitate, which crystallizes more slowly.

A second technique following Method A consists of dissolving the dilaudid in a drop of diluted sulfuric acid (1 part H_2O and 1 part H_2SO_4) instead of in water and then adding a drop of the reagent. Crystals form as fine needles in sheaves and rosettes. They are nearly opaque to transmitted light, but with light falling on them obliquely from above they are seen to be highly reflecting, with a red-gold sheen.

Platinum Bromide in Hydrobromic-Sulphuric Acid Solution. Platinic chloride crystals (H₂PtCl₆·6H₂O)—1 gram, HBr (40%)—10 cc., Diluted H₂SO₄ (H₂O—1 vol. to H₂SO₄—1 vol.)—20 cc. Add a drop of this reagent to a little solid dilaudid powder and apply a cover glass. The crystals are the fine needles in sheaves and rosettes just described



Figure 9 Cocaine with ${\rm HAuCl_4}$ in diluted Acetic acid. Method B. (Crossed nicols).

above, and are shown in Figure 8. This Method B test is much more sensitive than the preceding method.

TEST FOR COCAINE

The best Method A tests for cocaine, with platinum chloride (16, 5) and gold chloride (5, 6), have been known for a long time (2, 3). Gold chloride in concentrated hydrochloric acid will give better results than aqueous gold chloride—quicker and more complete crystallization with less interference from most impurities. To supplement these tests two new Method B tests are presented herewith.

Gold Chloride in Acetic Acid. Gold chloride crystals (HAuCl₄ 3H₂O)—1 gram, glacial acetic acid—40 cc., Water—20 cc. When a drop of this reagent is added to a little solid cocaine or its salt the characteristic crystals form immediately. These are pale yellow plates, very transparent and intensely birefringent, and Figure 9 shows them photographed with crossed nicols. Un-

fortunately the photograph can give no idea of the brilliance and beauty of the interference colors.

Gold Bromide in Acetic Acid. Gold chloride crystals—1 gram, HBr (40%)—1.5 cc., Diluted acetic acid (glacial acetic acid—2 vol. to H₂O—1 vol.)—to make 60 cc. If a drop of the reagent is added to a little solid cocaine or its salt the crystals form immediately. They are strongly birefringent and also very highly dichroic with polarized light, showing pale straw color or light yellow when oriented in one direction, and

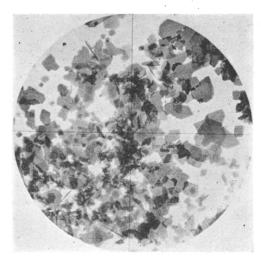


FIGURE 10

Cocaine with HAuBr₄ in diluted Acetic acid.

Method B. (Ordinary light).

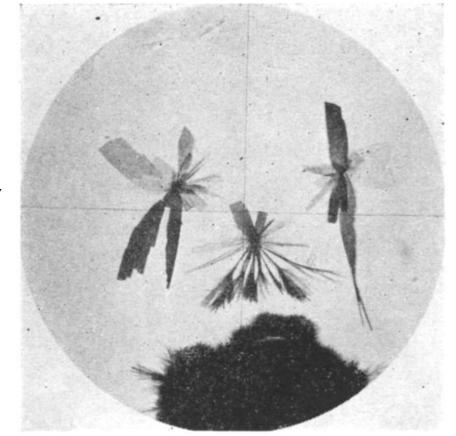
deep orange or even red at right angles to this. By ordinary light they are light salmon where the light comes through a single crystal but orange to red where the crystals overlap with differing orientation. Figure 10, showing the crystals, was taken with ordinary light.

BIBLIOGRAPHY

- 1. Wormley, T. G., Microchemistry of Poisons (1869), 2nd ed. (1885).
- Lyons, A. B., "Notes on the Alkaloids of Coca Leaves," Amer. J. Pharm., 30, (October 1885).
- Behrens, H., Anleitung zur Mikrochemischen Analyse, Vol. III (1896).
- Grutterink, Alide, Beiträge zur Mikrochemischen Analyse Einiger Alkaloide und Drogen mit besonderer Berucksichtigung der Methoden von H. Behrens (1910).
- Stephenson, Charles H., Some Microchemical Tests for Alkaloids (including Chemical Tests for the Alkaloids used, by C. E. Parker), (1921).
- Amelink, F., "Schema zur Mikrochemischen Identifikation von Alkaloiden," Amsterdam (1934).
- Fulton, Charles C., "The Precipitating Agents for Alkaloids," Amer. J. Pharm. 104 (4): 244-271 (April, 1932).
- 8. Fulton, Charles C., "New Precipitating Agents for Alkaloids and Amines," Amer. J. Pharm. 112 (2 and 4): 51-64, 134-154 (Feb. and Apr., 1940).
- Fulton, Charles C., "The Identification of Alkaloids by Precipitation: I. A Natural Classification of the Alkaloids Based on Precipitation," J. Assoc. Off. Agri. Chemists, 13 (4): 481 (1930).
- Fulton, Charles C., "Alkaloids and Their Reagents," Amer. J. Pharm. 111 (5): 184-192 (May, 1939).
- Fulton, Charles C., "The Identification of Atropine with Wagner's Reagent," J. Assoc. Off. Agri. Chemists, 12 (3): 312 (1929).
- Fulton, Charles C., "The Identification of Cocaine and Novocaine," Amer. J. Pharm. 105 (7 and 8): 326-339, 374-380 (July and Aug., 1933).
- Williams, G. D., and Fulton, C. C., "The Microscopic Identification of Heroin," Amer. J. Pharm. 105 (9): 435-439 (September, 1933).
- Fulton, Charles C., "The Principal Chemical Tests for Morphine," Amer. J. Pharm. 109 (5): 219-240 (May, 1937).
- Fulton, Charles C., "Crystal Tests for Minute Amounts of Morphine," J. Lab. and Clin. Medicine, 23 (6): 622-625 (March, 1938).
- Putt, E. B., "Microchemical Tests for the Identification of Some of the Alkaloids," J. Ind. Eng. Chem. 4: 508 (1912).

MICRO-CRYSTAL IDENTIFICATION TESTS FOR MORPHINE, HEROIN, DILAUDID, AND COCAINE

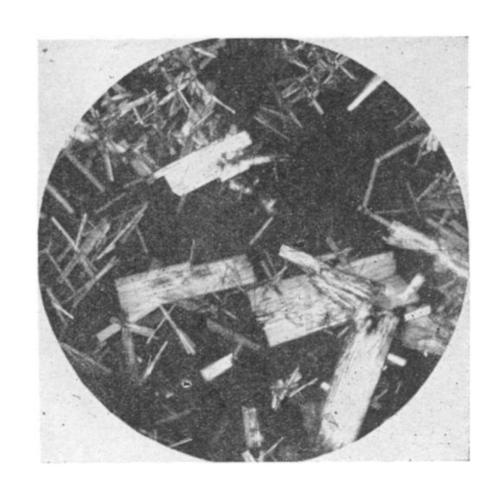
Charles C. Fulion† and John B. Dalton‡



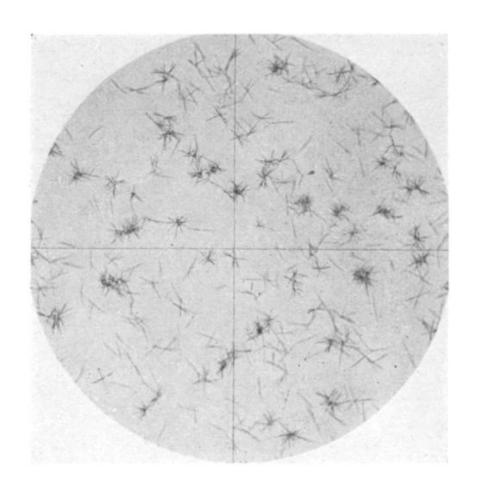
Micro-Crystal Identification Tests for Morphine, Heroin, Dilaudid, and Cocaine

Charles C. Fulton; John B. Dalton *Journal of Criminal Law and Criminology* (1931-1951), Vol. 32, No. 3. (Sep. - Oct., 1941), pp. 358-365.

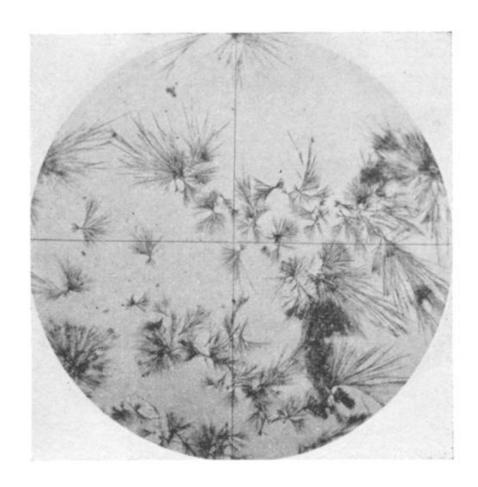
Morphine with HAuBr₄ in half-concentrated HBr. Method B. (Polarized light.)



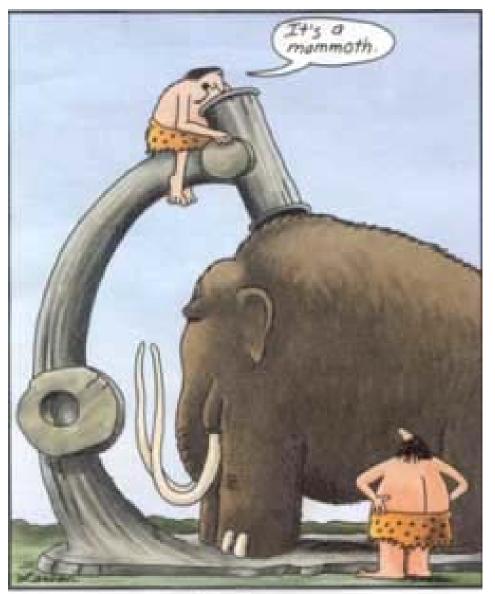
Morphine with H₂PtBrCl₅ in HCl. Method B. (Crossed nicols.)



Heroin with $HAuBr_4$ in diluted H_2SO_4 . Method A. (Extremely dilute solution.)



Dilaudid with H_2PtBr_6 in diluted H_2SO_4 . Method A.



JUST KIDDING

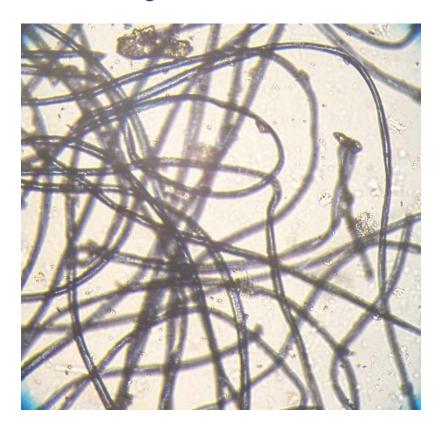


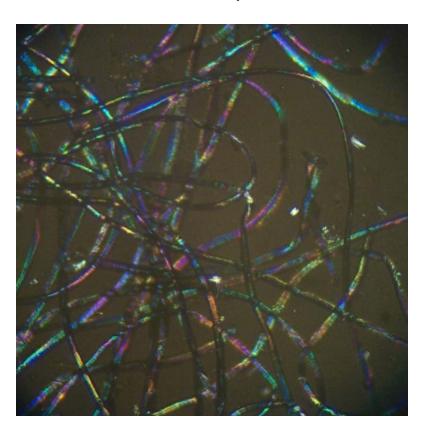
Early microscopes

Photomicrographs of polyester fibers

Regular transmission

crossed polars

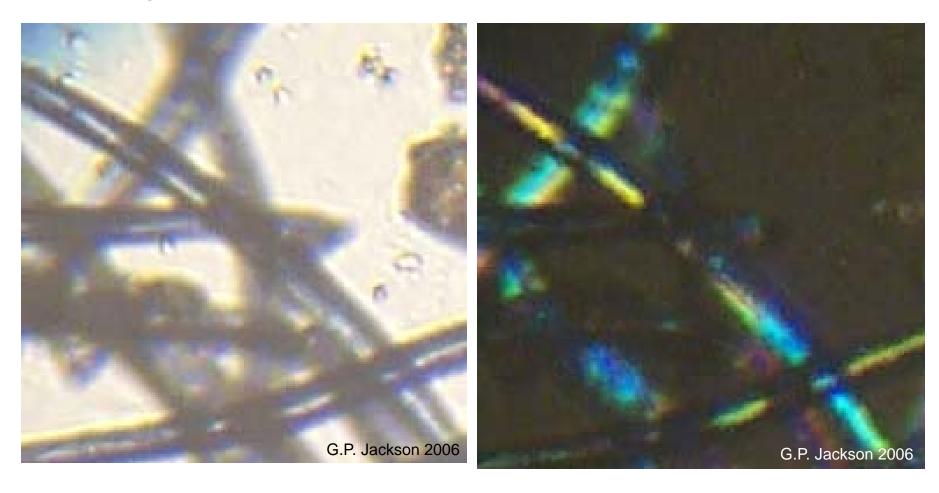




Photomicrographs of polyester fibers

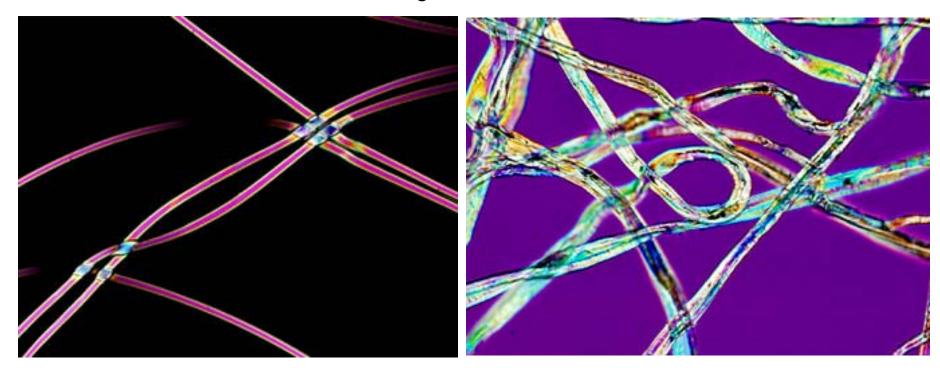
Regular transmission

crossed polars



Polarized light microscopy

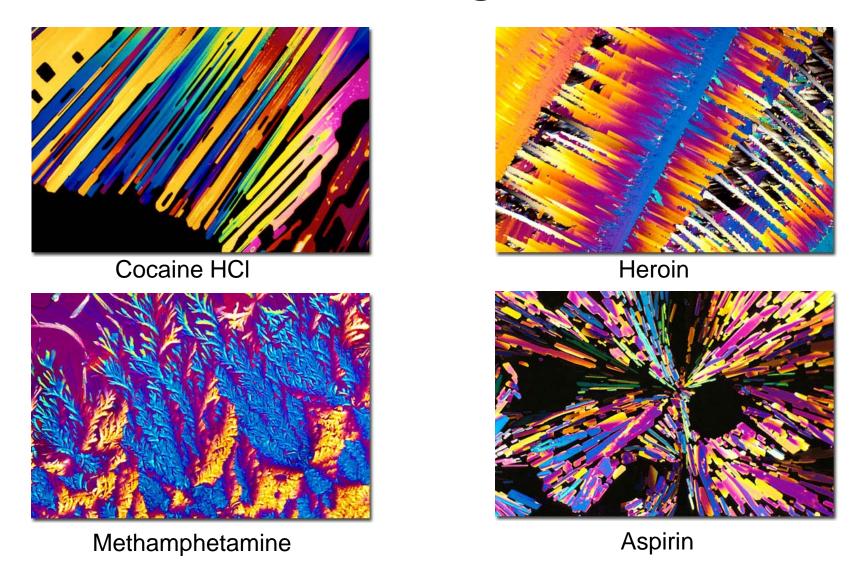
Birefringent materials



Synthetic nylon

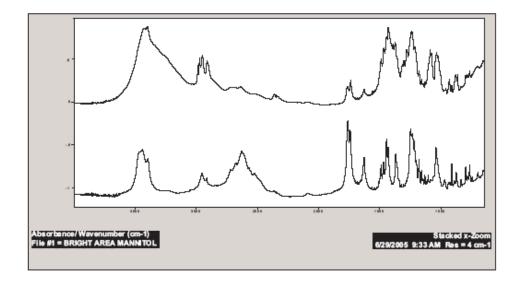
Natural cotton

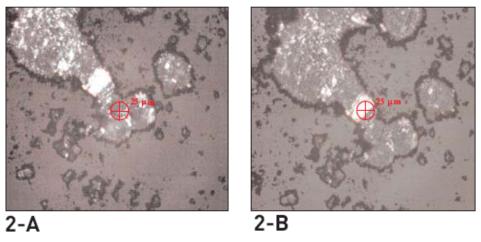
PLM images



FTIR microscopy

- E.g. of suspected heroin seizure
- Dark areas give heroin IR signal
- Light area gives mannitol signal
 - Common sugar





FTIR microscopy



Figure 3.
Micrograph of crystals
formed when AuCl3 is
added to cocaine.
This is a common
crystalline test for
cocaine.

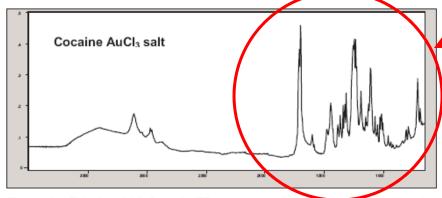


Figure 4. This is mid-infrared ATR spectrum of the crystal formed by the reaction of gold chloride with cocaine.

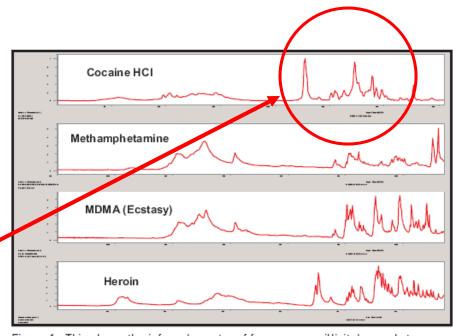
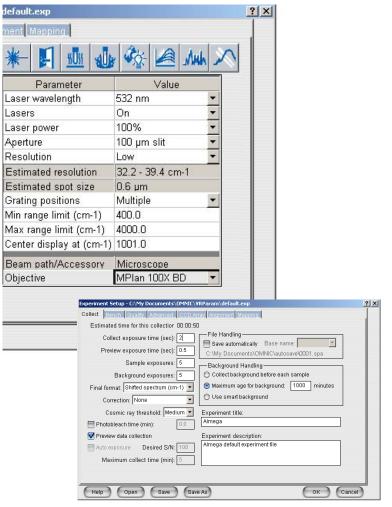


Figure 1. This shows the infrared spectra of four common illicit drug substances.

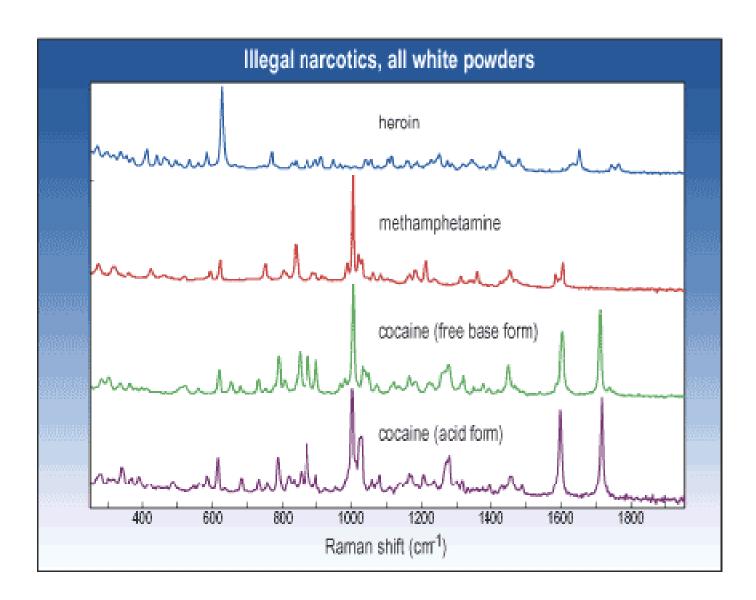
Raman Microscopy

Thermo Nicolet Almega XR



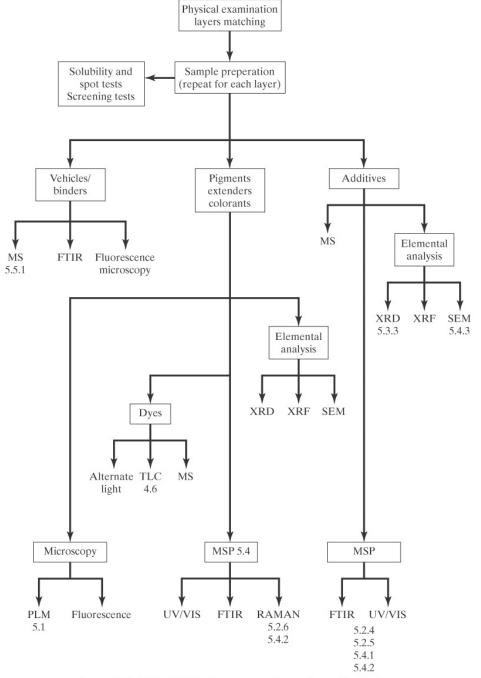


http://www.thermoscientific.com/ecomm/servlet/productscatalog_11152_L 11024_91245_-1_4 8



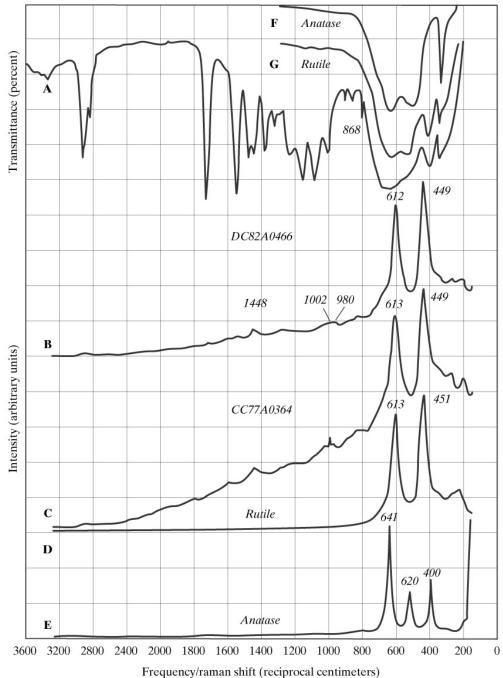
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Paint and ink analysis



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IR and Raman for paints

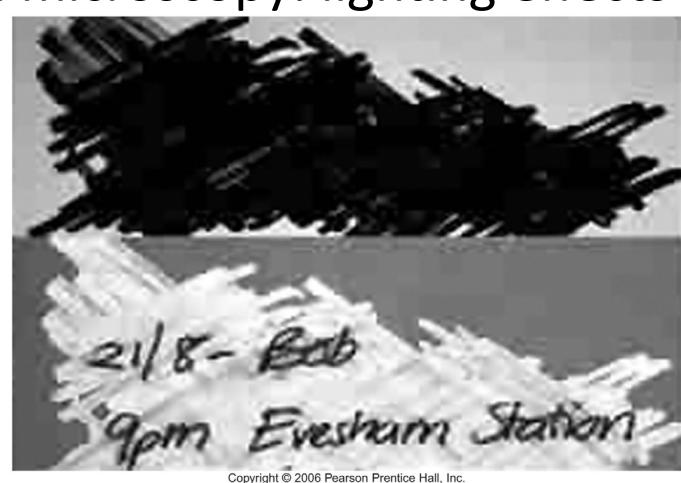


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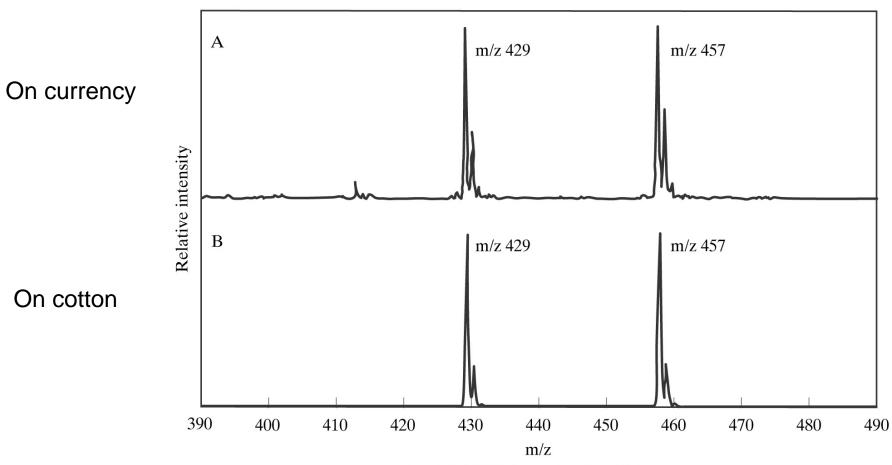
Optical microscopy: lighting effects

Near IR lighting

Mid IR lighting

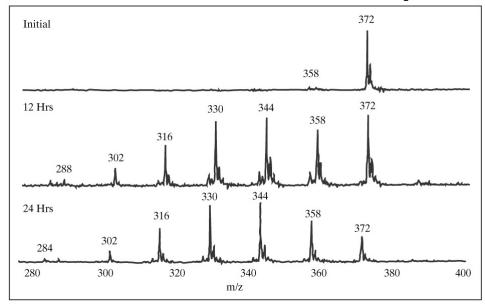


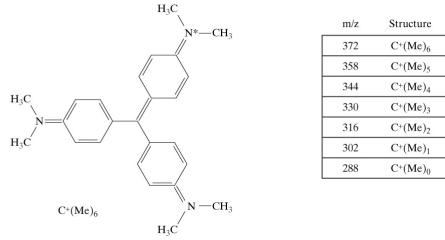
LDI-MS of inks



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LDI-MS of inks after UV exposure

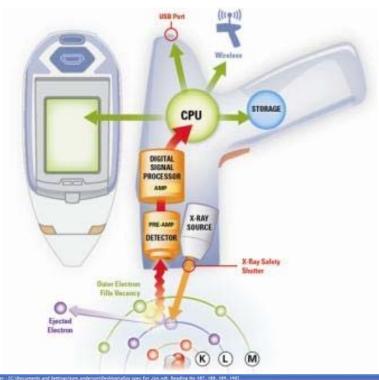


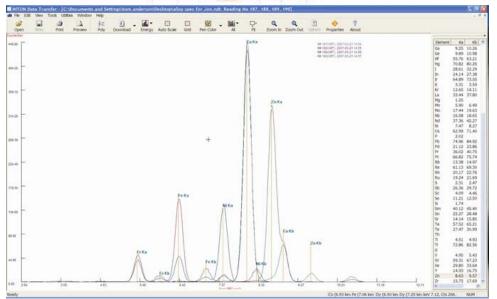


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Analytik Now Offering Handheld FTIR Spectrometers from Agilent









Art and Artefacts with the Thermo Scientific Niton XRF Analyser

Lignin