

**NOTEBOOK NO.** 123  
**ISSUED TO** Yanan Didsatha-amparj  
**ON** August 4th **2008**  
**DEPARTMENT** \_\_\_\_\_  
**RETURNED** Aug 14 **2008**

SCIENTIFIC NOTEBOOK COMPANY  
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10. Using automatic headspace sample injection to measure  
furan in corn oil Pood

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11. LOD and LOQ of the method

19.

14/7/08  
Procedures

- I. Prepare a new d4-furan stock solution
  1. Place a 40 ml vial on the analytical balance with four decimal points and tare balance to 0.0000 .
  2. Pipette 20 ml of water into the vial
  3. Weigh water (W1:19.91915)
  4. Take d4-furan out from the fridge and let it sit at room temperature
  5. Pipette 2 drops into the vial of water
  6. Weigh water + d4-furan (W2: 19.9317)
  7. Calculation  $W_2 - W_1 / 20\text{ml} = (19.9317 - 19.91915) / 20\text{ml} = 0.64 \text{ mg/ml}$
- II. Prepare 1.6  $\mu\text{g/ml}$  of d4-furan working solution
  1. Pipette 20 ml of water into 40 ml vial
  2. Use micropipette to transfer 50  $\mu\text{l}$  of 0.64  $\mu\text{g/ml}$  of d4-furan into the water vial
  3. The concentration is  $(0.64 \cdot 50) / 1000 \cdot 20.05\text{ml} = 1.6 \mu\text{g/ml}$
- III. Prepare 6.3  $\mu\text{g/ml}$  working furan solution from 1.27  $\mu\text{g/ml}$  stock furan solution
  1. Pipette 20 ml of water into a 40 ml vial
  2. By using micropipette, take 100  $\mu\text{l}$  of 1.27  $\mu\text{g/ml}$  stock furan and place into the vial
  3. The concentration is  $(1.27 \cdot 100) / 1000 / 20.1 = 6.3 \mu\text{g/ml}$
- IV. Prepare 9.45  $\mu\text{g/ml}$  working furan solution from 1.27  $\mu\text{g/ml}$  stock furan solution
  1. Pipette 20 ml of water into 40 ml vial
  2. By using micropipette, take 150  $\mu\text{l}$  of 1.27  $\mu\text{g/ml}$  stock furan and place into the vial
  3. The concentration is  $(1.27 \cdot 150) / 1000 / 20.15 = 9.45 \mu\text{g/ml}$
- V. Repeat measurement of Jewel Medium June Pea, Can C, same lot (AYB6217)
  1. The batch of pea that was prepared on 10/7/08 was put in a water bath to defrost
  2. 10g of sample was transfer into a 20 ml vial
  3. Incubate in ice for 5 mins
  4. Prepare 0.63  $\mu\text{g/ml}$  by dilute 1/10 of furan 6.3  $\mu\text{g/ml}$ : take 1000  $\mu\text{l}$  of 6.3  $\mu\text{g/ml}$  into 9 ml of water ( Prepare in 20 ml vial)

There for 4 standard additions are:

X0: 10 g of sample + 25  $\mu\text{l}$  of IS ( 1.6  $\mu\text{g/ml}$  of d4-furan)  
 X1: 10 g of sample + 25  $\mu\text{l}$  of IS + 25  $\mu\text{l}$  0.63  $\mu\text{g/ml}$  of spiked furan  
 X2: 10 g of sample + 25  $\mu\text{l}$  IS + 50  $\mu\text{l}$  spiked 0.63  $\mu\text{g/ml}$  furan  
 X3: 10 g of sample + 25  $\mu\text{l}$  IS + 100  $\mu\text{l}$  spiked 0.63  $\mu\text{g/ml}$  furan

- Repeat analysis of pea solid of can C lot A 3 times (data fine: peac05-08,,2peax0-x3,3peax0-x3)

Witnessed &amp; Understood by me,

*Yvonne B.*

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+ 7/11/08

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- VI. Prepare and measure furan in applesauce baby food
1. Prepare a batch of baby applesauce by putting 50g of applesauce into 150 ml water. (Dil factor = 4)
  2. Transfer 10 g of applesauce into 20 ml vial
  3. Incubate in an icebath for 5 mins
  4. Fortified d4-furan and 0.63 µg/ml furan
- X0: 10 g of sample + 25 µl of IS ( 1.6 µg/ml of d4-furan)  
 X1: 10 g of sample + 25 µl of IS + 25 µl 0.63 µg/ml of spiked furan  
 X2: 10 g of sample + 25 µl IS + 50 µl spiked 0.63 µg/ml furan  
 X3: 10 g of sample + 25 µl IS + 100µl spiked 0.63 µg/ml furan

## V. Set up a sequence on Chemstation and the combi/pal autosampler

Results

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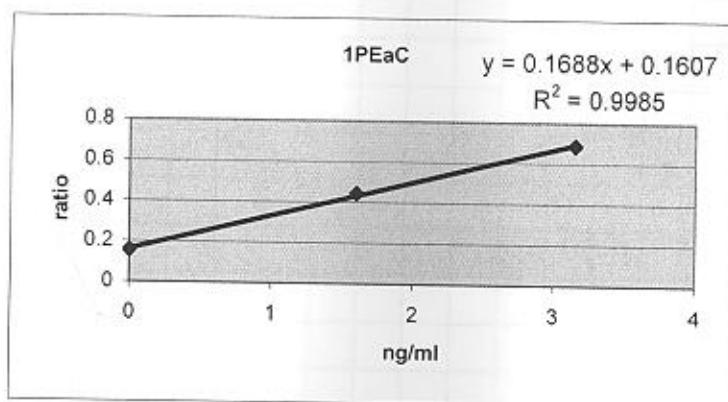
14/7/08

AYB6217 2nd can (canC)

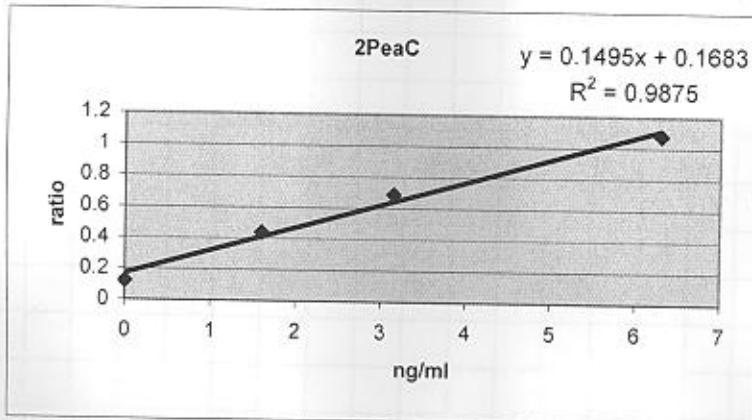
d4-furan 1.6 ug/ml

furan 0.63ug/ml 0,25,50,100ul

level	File name	conc spike	68	72	ratio	39	37	42	44
0	PeaC05	0	164301	1E+06	0.154908437	161867	-	1358731	280020
0.5x	PeaC06	1.6	386602	873647	0.442515112	552532	-	1133824	227802
1x	PeaC07	3.15	594529	866122	0.686426393	816214	96661	1140879	226716
2x	PeaC08	6.3	1E+06	601695	1.795100508	1E+06	179788	740474	156868



level	File name	conc spike	68	72	ratio	39	37	42	44
0	2Peax0	0	123165	1E+06	0.122879673	121335	-	1275973	262393
0.5x	2Peax1	1.6	401326	919237	0.436585995	567366	-	1194137	242887
1x	2Peax2	3.15	548177	798450	0.686551443	755906	93953	1050455	209149
2x	2Peax3	6.3	905860	839732	1.078748934	1E+06	151402	1119825	223583



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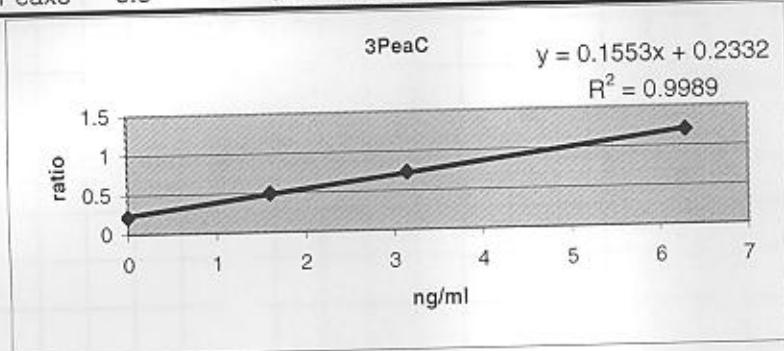
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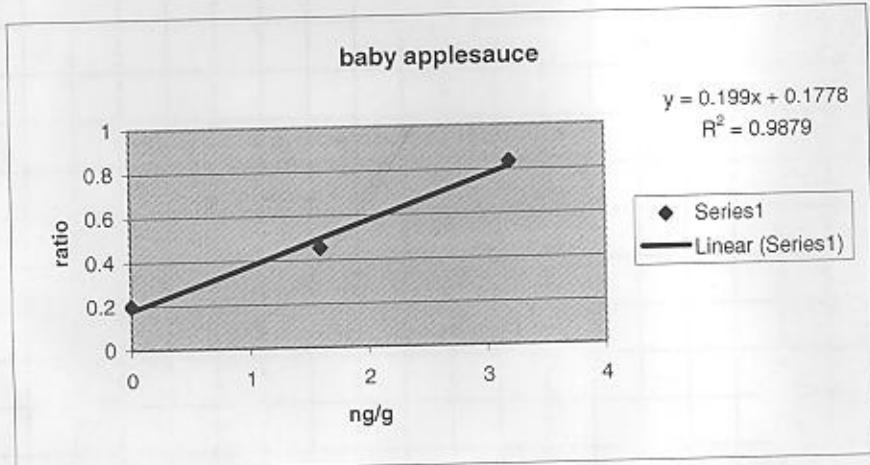
level	File name	conc spike	68	72	ratio	39	37	42	44
0	3Peax0	0	164987	754709	0.218610087	154698	-	969217	196827
0.5x	3Peax1	1.6	375974	763143	0.492665202	554260	-	994505	201800
1x	3Peax2	3.15	532312	723977	0.735260927	727615	87987	957098	191345
2x	3Peax3	6.3	926179	770329	1.202316153	1E+06	212097	1055812	202980



6 ng/ml

Applesauce

level	File name	conc spike	68	72	ratio	39	37	42	44
0	Appx0	0	149065	752136	0.198188891	190220	-	988937	198157
0.5x	APPX1	1.6	345023	757346	0.455568525	450034	-	1012516	198797
1x	APPx2	3.2	582872	698021	0.835035049	775556	121667	943906	179462
2x	APPX3	6.3	750841	746656	1.005604991	986704	158716	1044240	195744



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15\_07\_08

## Procedures

- I. Prepare spiked solution 0.63 µg/ml furan: 1/10 dilution of 6.3µg/ml by micropipette 1000 µl of furan into 10 ml of water (20ml vial)
- II. Prepare a new batch of Jewel June medium pea, lot A (AYB6217), can C, from the refrigerated canned that had been opened once on 7/9/08: 100g of can C into 300 ml of water in 500 ml glass bottle. Homogenized/keep cold
- III. Prepare a new batch of carrot lot A can A (Jewel sliced carrot:AGCA267; opened 7/14/08): taking 100 g of carrot into 300 ml of water in 500 ml glass bottle. Homogenized/keep cold.
- IV. Prepare two batches of carrot lot B can A (Jewel sliced carrot:NGBA317); opened 7/15/08; taking 100 g of carrot into 300 ml of water in a 500 glass bottle. Homogenized/keep cold.
- V. Furan analysis of 3 samples (sequence set at day time): 1. sliced carrot lot A can A 2. Applesauce lot A can A 3. Pea Lot A can C

**a. For sliced carrot lot A can A (data file carra1-4):**

1. Take 10 g of sample into 20 ml vial
2. Incubate in ice for 5 mins
3. Fortified 1.6 µg/ml d4-furan and 0.63 µg/ml furan  
X0 : 10 g + 25 µl 1.6µg/ml d4-furan  
X1 : 10 g + 25 µl 1.6 µg/ml d4 furan + 25 µl 0.63 spiked furan  
X2: 10 g + 25 µl IS + 50 µl of 0.63 spiked furan  
X3: 10 g + 25 µl IS + 100 µl of 0.63 spiked furan

**b. For applesauce (data file peac1-3,applx3):**

- 1 Take 10 g of sample into 20 ml vial
- 2 Incubate in ice for 5 mins
- 3 Fortified 1.6 µg/ml d4-furan and 0.63 µg/ml furan  
X0 : 10 g + 25 µl 1.6µg/ml d4-furan  
X1 : 10 g + 25 µl 1.6 µg/ml d4 furan + 25 µl 0.63 spiked furan  
X2: 10 g + 25 µl IS + 50 µl of 0.63 spiked furan  
X3: 10 g + 25 µl IS + 100 µl of 0.63 spiked furan

**c. For Pea(data file peac5-8).**

- 1 Take 10 g of sample into 20 ml vial
- 2 Incubate in ice for 5 mins
- 3 Fortified 1.6 µg/ml d4-furan and 0.63 µg/ml furan  
X0 : 10 g + 25 µl 1.6µg/ml d4-furan  
X1 : 10 g + 25 µl 1.6 µg/ml d4 furan + 25 µl 0.63 spiked furan  
X2: 10 g + 25 µl IS + 50 µl of 0.63 spiked furan  
X3: 10 g + 25 µl IS + 100 µl of 0.63 spiked furan

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VI. Prepare a sample and sequence to run overnight

**a. For liquid pea lot A can C, run duplicate analysis of liquid pea (data file****LPEAC1 -8 )**

1. Pipette 2ml of pea liquor into a vial having 8ml vial (2/10 tot; dil factor=5);

8 vials (2 set)

2. Incubate in ice for 5 mins

3. Spiked 15  $\mu$ l of 1.67  $\mu$ g/ml d4-furan and 0.63  $\mu$ g/ml furan

X0: 10 ml sample + 15  $\mu$ l ISX1: 10 ml sample + 15 IS + 15  $\mu$ l spikedX2: 10 ml sample + 15 IS + 30  $\mu$ l spikedX3: 10 ml sample + 60  $\mu$ l spiked**b. Prepare 1 set of solid carrot lot A can A**

1. From a batch of dilute sample prepared in the day time, transferred 10 g of sample into a 20g headspace vial

2. Incubate in ice for 5 mins

3. spiked 15  $\mu$ l of 1.67  $\mu$ g/ml d4-furan and 0.63  $\mu$ g/ml furan

X0: 10 g sample + 15  $\mu$ l ISX1: 10 g sample + 15  $\mu$ l IS + 15  $\mu$ l spikedX2: 10 g sample + 15  $\mu$ l IS + 30  $\mu$ l spikedX3: 10 g sample + 15 IS + 60  $\mu$ l spiked**c. Prepare 1 set of liquid carrot lot A can A**

1. Pipette 8 ml of water into 20 ml headspace vial

2. From a batch of liq carrot prepared in daytime, pipette 2 ml of sample in to each vial. Vortex.

3. Incubate in ice

4. spiked 15  $\mu$ l of 1.67  $\mu$ g/ml d4-furan and 0.63  $\mu$ g/ml furan

X0: 10 g sample + 15  $\mu$ l ISX1: 10 g sample + 15  $\mu$ l IS + 15  $\mu$ l spikedX2: 10 g sample + 15  $\mu$ l IS + 30  $\mu$ l spikedX3: 10 g sample + 15 IS + 60  $\mu$ l spiked

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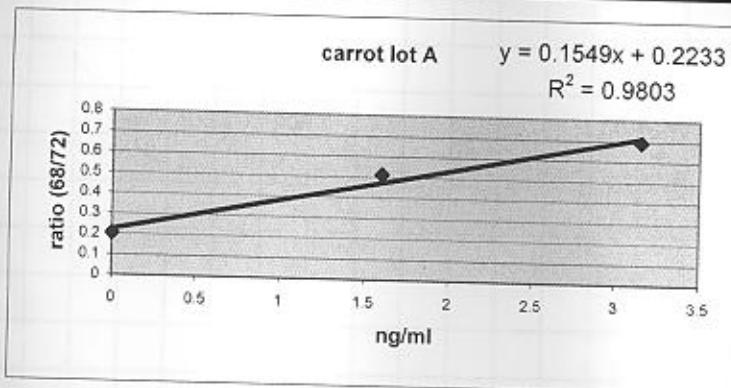
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15/7/07 Day-time

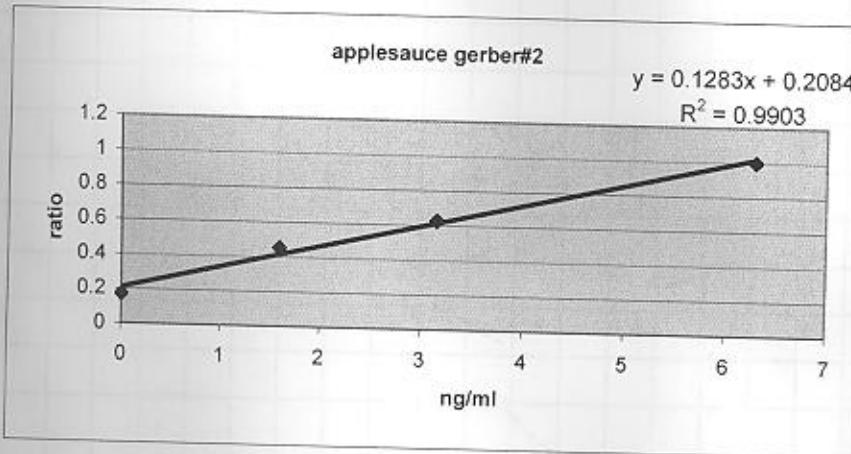
Spiked fur 0.25, 50, 100ul spiked of 0.63 ug/ml      d4-furan 1.6 ug/ml  
carrot lot A #1

level	File name	conc	spil	68	72	ratio	39	37	42	44
0	carra1.d	0		171979	844501	0.203645703	228911	34939	1E+06	227219
0.5x	carra2.d	1.6		372481	728951	0.51098222	494968	81094	989207	194552
1x	carra3.d	3.15		475746	688564	0.690924881	632098	99940	936469	180056
2x	carra4.d	6.3		422890	562748	0.751473128	557005	91183	759760	148797



Amount 5.7 ng/ml

applesau	file name	conc	spil	68	72	ratio	39	37	42	44
0	peac1	0		113410	662084	0.171292464	138439	22681	849998	174217
0.5x	pea2	1.6		299765	661369	0.453249245	396530	60422	872681	173776
1x	peac3	3.15		382366	609367	0.627480648	506820	76418	826699	160252
2x	applx3	6.3		422335	422839	0.998808057	560488	91082	591412	113072



Amount 6.4 ng/ml

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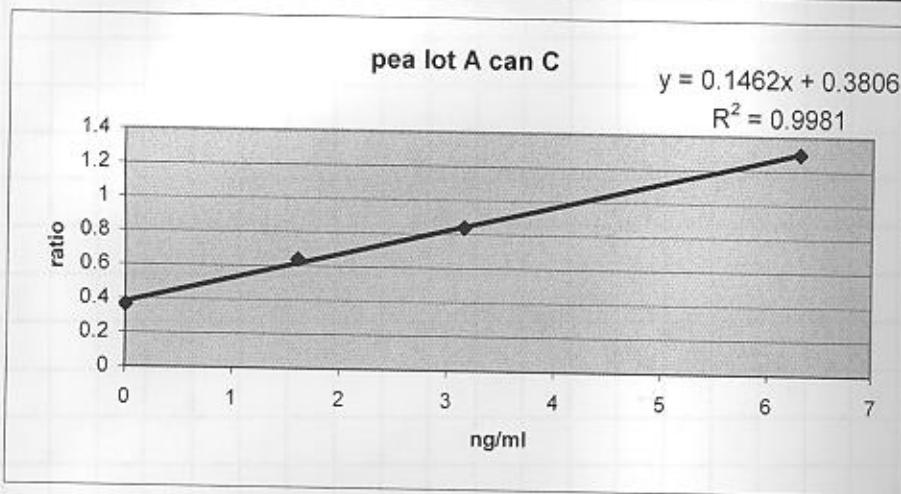
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Pea samp	file name	conc	spil	68	72	ratio	39	37	42	44
0	peac5.D	0		169548	464965	0.364646801	226703	37812	618801	124022
0.5x	peac6.D	1.6		270483	423489	0.638701359	358416	59394	575431	111088
1x	PEAC7.D	3.15		364358	435491	0.836660229	470361	79783	593949	115220
2x	PEAC8.D	6.3		577459	445059	1.297488648	759737	125130	626290	117931



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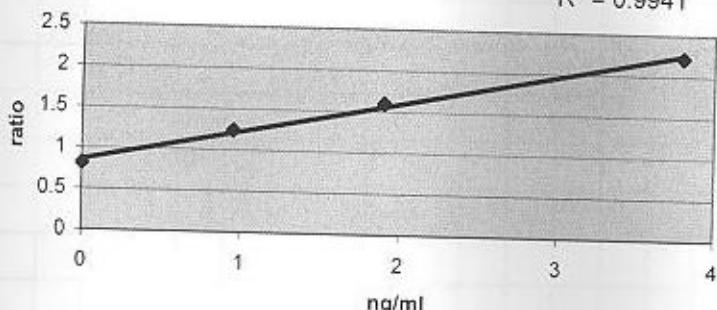
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15\_7\_08 night time

spiked furan 0,15,30,60 ug/ml of 0.63 ug/ml furan, 1.67 ug/ml d4-furan

Pea liquo file name conc spiked (	68	72	ratio	39	37	42	44
0 Lpeac1 0	254673	314279	0.810340494	337729	56681	424178	80497
0.5x Lpeac2 0.95	375233	302909	1.238764778	487369	83855	384573	80652
1x Lpeac3 1.9	486235	304630	1.596149427	636703	106466	381607	81793
2x Lpeac4 3.8	667240	301174	2.215463486	871466	143449	368604	78055

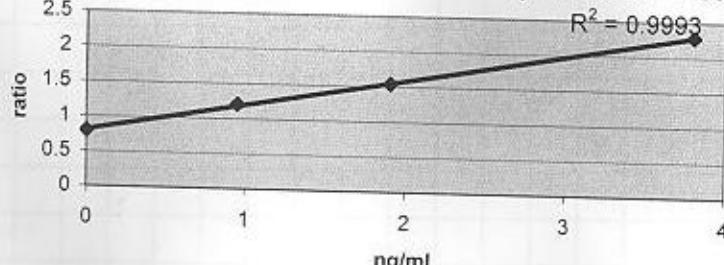
liq pea canC lot A       $y = 0.3653x + 0.8578$   
 $R^2 = 0.9941$



Amount 11.7 ng/ml

Pea liquo file name conc spiked (	68	72	ratio	39	37	42	44
0 Lpeac5 0	245218	308620	0.794562893	325472	52954	422369	81452
0.5x Lpeac6 0.95	339730	282810	1.201265868	446507	67517	357671	74618
1x Lpeac7 1.9	417452	271419	1.538035289	545644	91012	347153	71844
2x Lpeac8 3.8	640646	280902	2.280674399	835252	138423	351251	74407

liq pea canc lot A2       $y = 0.3879x + 0.8087$   
 $R^2 = 0.9993$



Amount 10.4 ng/ml

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16/7/08

## Procedures

1. Bring a batch of solid carrot lot B, solid carrot lot A, liquid carrot lot B out of a refrigerator and defrost in a water bath

2. Prepare test portions for a run overnight

**a) solid carrot lot B (File name ca2bx0-3,car2bx0-3,carb01-04)**

1. Bring out 1.67 µg/ml d4-furan and 6.3 µg/ml furan out and let it sit at room temperature

2. Dilute 6.3 µg/ml furan by taking 1000µl of 6.3 µg/ml into 9 ml water

3. Transfer 10 g of solid carrot lot B into a headspace vial ( triplicate)

4. Incubate in ice for 5 mins

5. Spike 1.67 µg/ml d4-furan and 0.63 µg/ml furan

X0 : 10 g + 15 µl d4-furan

X1: 10 g + 15 ul d4-furan + 15 ul spiked furan

X2: 10 g + 15 µl d4 furan + 30 µl spiked furan

X3: 10 g + 15 µl furan + 60 µl spiked furan

**b) solid carrot lot A (File name carax0-3)**

1. Transfer 10 g of solid carrot lot A into 20 headspace vial

2. Incubate in ice for 5 mins

3. Spike 1.67 µg/ml d4-furan and 0.63 µg/ml furan

X0 : 10 g + 15 µl d4-furan

X1: 10 g + 15 ul d4-furan + 15 ul spiked furan

X2: 10 g + 15 µl d4 furan + 30 µl spiked furan

X3: 10 g + 15 µl furan + 60 µl spiked furan

**c) liquid carrot lot B (File name carlb1-4)**

1. Pipette 8 ml of water into a headspace vial

2. Transfer 2 ml of liquid carrot into the vial

3. Vortex

4. Spike 1.67 µg/ml d4-furan and 0.63 µg/ml furan

X0 : 10 g + 15 µl d4-furan

X1: 10 g + 15 ul d4-furan + 15 ul spiked furan

X2: 10 g + 15 µl d4 furan + 30 µl spiked furan

X3: 10 g + 15 µl furan + 60 µl spiked furan

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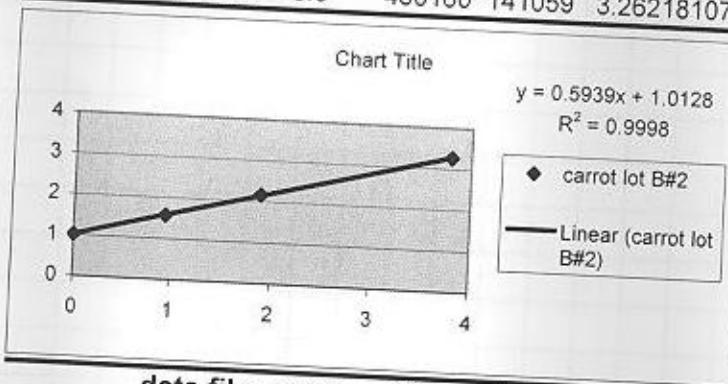
carrot lot B

IS d4-furan 1.67 g/ml

spiked furan 0.63 ug/ml

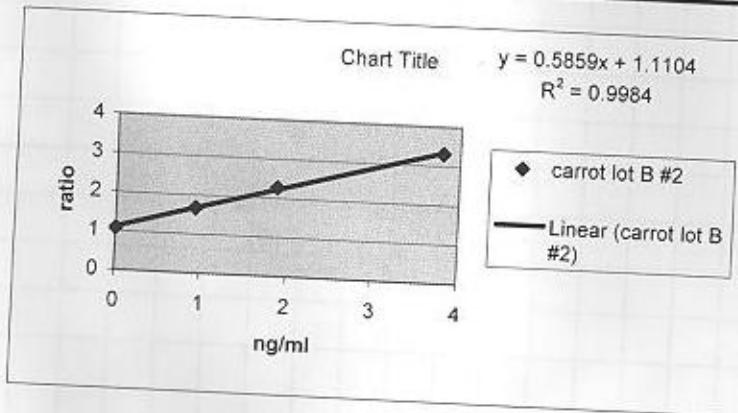
0,15,30, ng/ml

	data file	conc	68	72	ratio	39	37	42	44
0	CA2Bx0	0	224100	222185	1.008618944	290007	45818	274718	57286
0.5x	CA2Bx1	0.945	344178	219561	1.567573476	441591	70769	268806	56399
1x	CA2Bx2	1.89	341162	158460	2.15298498	438359	69411	198491	43280
2x	CA2Bx3	3.8	460160	141059	3.262181073	591816	93914	169552	36787



Amount 6.8 ng/ml

	data file	conc	68	72	ratio	39	37	42	44
0	CAR2Bx0	0	172603	159864	1.079686484	220777	34715	200003	42974
0.5x	CAR2Bx2	0.945	286023	171236	1.670343853	342195	55022	146856	31570
1x	CAR2Bx1	1.89	271396	119595	2.269292194	355346	58120	207541	44558
2x	CAR2Bx3	3.8	392488	118596	3.309453944	497567	80711	147259	32788



Amount 7.6 ng/ml

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7/16/08

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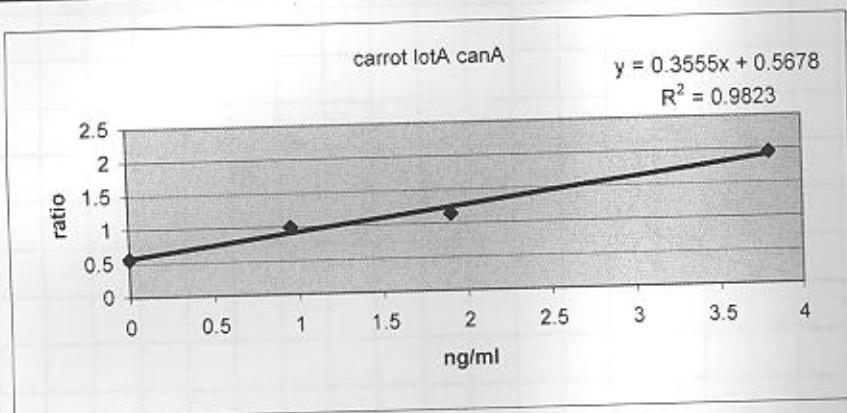
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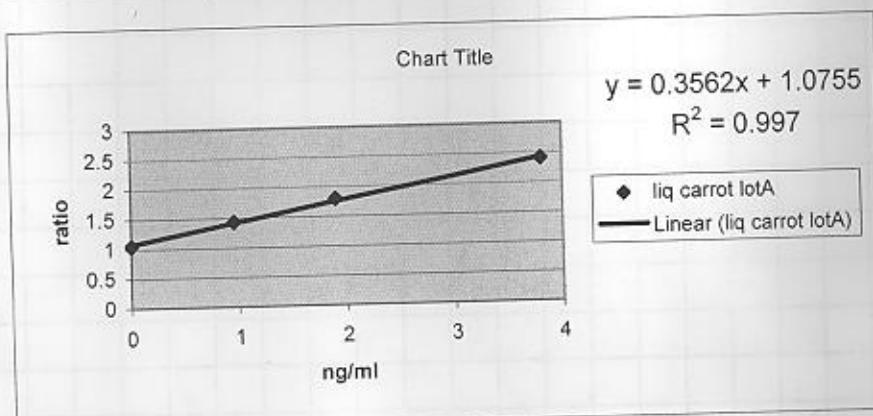
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Carrot Lot A can A conc spiked (		68	72	ratio	39	37	42	44
0	Cara201.d	0	197904	353566	0.559737079	259004	40605	472591
0.5x	cara202	0.95	313632	318429	0.984935417	400182	65853	439376
1x	cara203	1.9	309402	271316	1.140375061	404419	65049	378303
2x	cara204	3.8	500010	256356	1.950451716	652129	104813	319978



Amount 6.4 ng/ml

Liq carrot lotA can/ conc spiked (		68	72	ratio	39	37	42	44
0	carl01	0	294450	281630	1.045520719	384317	61216	389981
0.5x	carl02	0.95	386284	270380	1.42867076	502079	82894	345806
1x	carl03	1.9	347266	194024	1.789809508	452335	73255	239776
2x	carl04	3.8	653511	271580	2.406329627	876220	145123	21171



Amount 15 ng/ml

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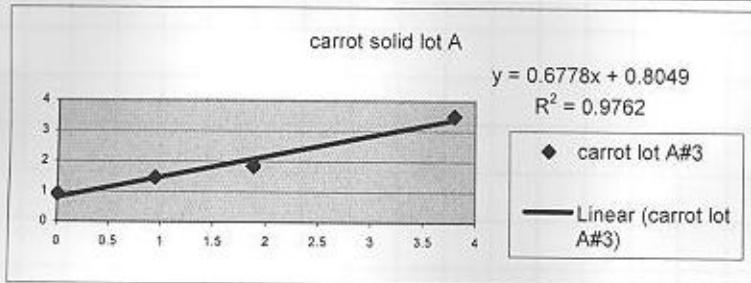
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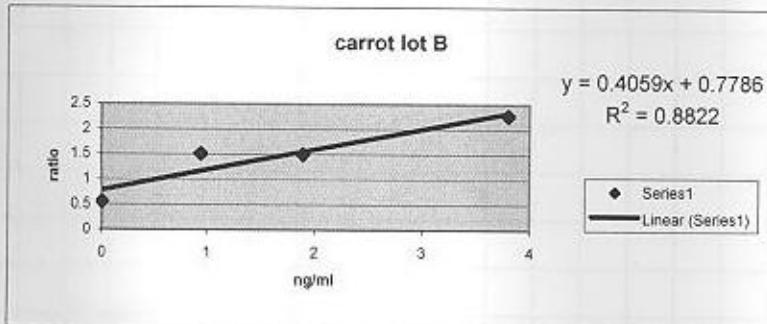
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	data file	conc	68	72	ratio	39	37	42	44
0	CARAX0	0	172626	188782	0.914419807	215642	35222	253839	47746
0.5x	CARAx1	0.945	258261	176137	1.466250703	322047	53127	214201	43660
1x	CARAx2	1.89	291607	158751	1.836882917	374643	58521	194363	42219
2x	CARAx3	3.8	494625	141352	3.499243025	497147	80596	169621	36744

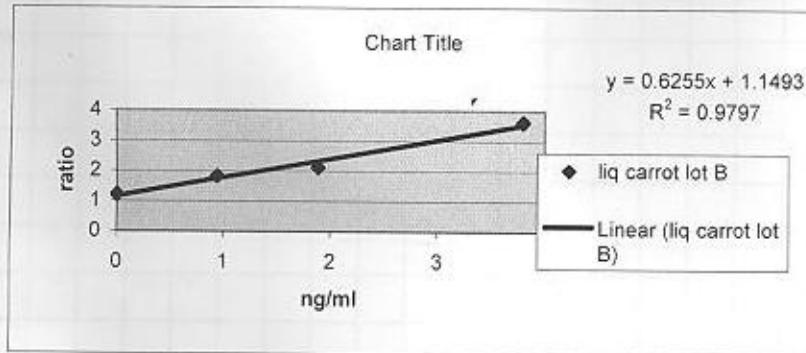


4.75 ng/ml

	data file	conc	68	72	ratio	39	37	42	44
0	carb01	0	198891	361304	0.550481035		43778	475046	94663
0.5x	carb02	0.945	321670	214165	1.501972778		64490	261582	54308
1x	carb03	1.89	413555	277227	1.491755854	532352	86720	339316	73115
2x	carb04	3.8	595375	263050	2.263352975	759107	126539	321578	67907



	data file	conc	68	72	ratio	39	37	42	44
0	carlb1	0	191111	158582	1.205124163	241788	36404	216823	40448
0.5x	carlb2	0.945	313952	173238	1.812258281	401523	61551	207699	43792
1x	carlb3	1.89	395592	187198	2.113227705	459490	75466	226034	49282
2x	carlb4	3.8	635852	175793	3.617049598	805246	134330	213570	45667



Amount 7.3 ng/ml

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7/17/08

## Procedures

- I. Prepare 0.63 µg/ml of furan solution by taking 1000 µl of 6.3 µg/ml furan working solution
  1. Take 1000 µl of 6.3 µg/ml furan into a 20 ml vial containing 9 ml of water
  
- II. Measure furan in solid carrot lot A
  1. Brought a batch of solid carrot out and put it on a water bath to let it defrost
  2. Transfer 10 g of sample into a 20 ml headspace vial
  3. Incubate in ice for 5 minutes
  4. Fortified 25µl of 1.67 µg/ml of d4-furan and 0,20,40 and 80µl of 0.63 µg/ml furan

X0 : 10 g sample + 25 µl of IS  
 X1 : 10 g sample + 25 µl of IS + 20 µl spiked furan  
 X2 : 10 g sample + 25 µl of IS + 40 µl spiked furan  
 X3 : 10 g sample + 25 µl of IS + 80 µl spiked furan
  
- III. Measure furan in liquid carrot lot A
  1. Brought a batch of liquid carrot lot A
  2. Defrost in a water bath
  3. Take 8 ml of water into a 20 ml vial and 2 ml of liquid carrot into the vial
  4. Mix using vortex
  5. Fortified d4-furan (1.67 µg/ml) and furan (0.63 µg/ml )

X0 : 10 g sample + 25 µl of IS  
 X1 : 10 g sample + 25 µl of IS + 20 µl spiked furan  
 X2 : 10 g sample + 25 µl of IS + 40 µl spiked furan  
 X3 : 10 g sample + 25 µl of IS + 80 µl spiked furan
  
- IV. Measure furan in Corn (Del monte, Whole Kernel corn, no salt added, lot 724714) solid and liquor.
  1. Brought out a refrigerated corn and let it defrost in a water bath
  2. Open the can and quickly separated the solid and the liquid
  3. For solid, take 100 g into 500 ml bottle (cold) and homogenize
    - a.) Solid corn
      - Transfer 10 g of homogenized solid portion of corn into 20 ml headspace vial. Incubate in ice. Fortified 25 µl d4-furan (1.67 µg/ml) and Spike 0,20,40, and 80 µl (0.63 µg/ml )
  
    - b.) Liquid corn
      - Transfer 10 ml of corn liquor into a 20 ml headspace vial. Incubate in ice. Fortified 25 µl d4-furan (1.67 µg/ml) and Spike 0,20,40, and 80 µl (0.63 µg/ml )

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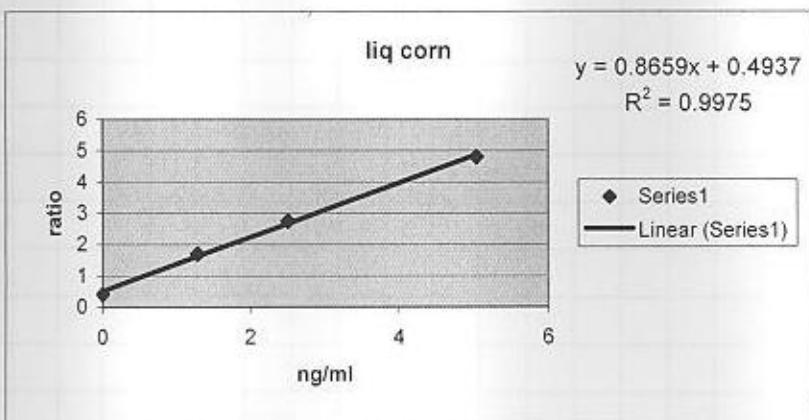
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7/17/2008

IS d4-furan 1.67 g/ml

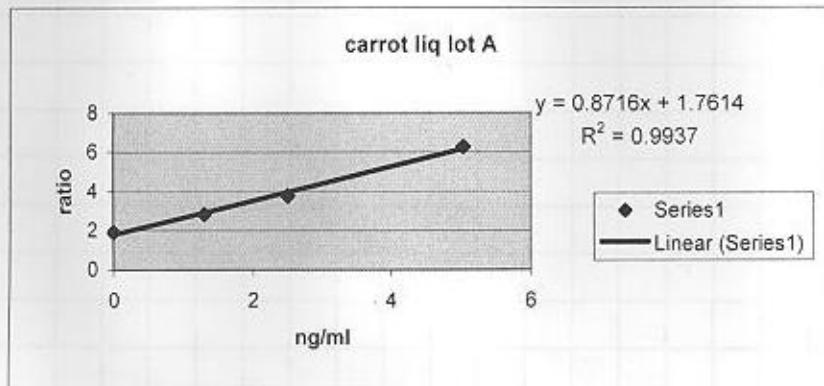
spiked furan 0.63 ug/ml 0.20,40,80 ul added

	data file	conc	68	72	ratio	39	37	42	44
0	1lcorn1.d	0	40828	102887	0.3968237	49983	7652	129791	25787
0.5x	1lcorn2.d	1.3	154781	91507	1.691466227	195015	31971	110578	22718
1x	1lcorn3.d	2.5	260171	94794	2.74459354	329929	54791	115377	24564
2x	1lcorn4.d	5.04	402726	83959	4.796698388	512948	82030	99585	22125



Amount 2.85 ng/ml

	data file	conc	68	72	ratio	39	37	42	44
0	2lcarx0	0	298342	156953	1.900836556	378060	61388	189629	44764
0.5x	2lcarx1	1.3	483673	171088	2.827042224	615431	101733	203482	43748
1x	2lcarx2	2.5	623266	165626	3.763092751	783436	129062	197168	37009
2x	2lcarx3	5.04	888969	142011	6.259860152	1E+06	181868	167030	29636



Amount 10.1 ng/ml

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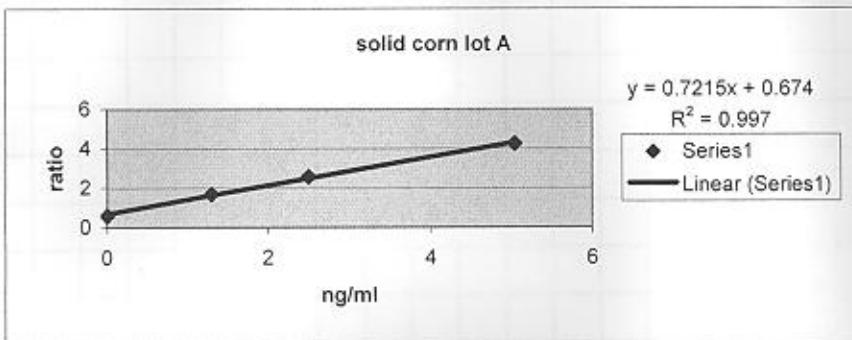
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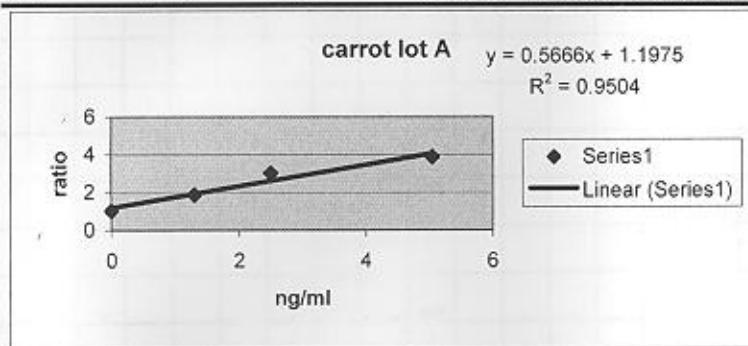
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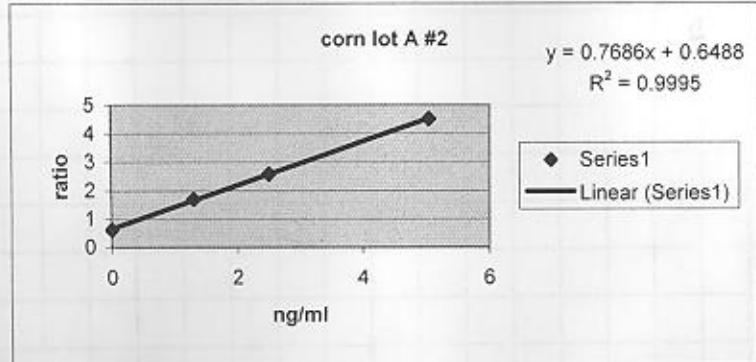
	data file	conc	68	72	ratio	39	37	42	44
0	2scorn1.d	0	69612	118294	0.588466025	87463	14792	153925	29636
0.5x	2scorn2.d	1.3	178667	107282	1.665395873	226447	36946	130859	28047
1x	2scorn3.d	2.5	284518	110753	2.568941699	360628	58565	134291	28475
2x	2scorn4.d	5.04	481918	113349	4.251629922	607172	100687	138466	29550



	data file	conc	68	72	ratio	39	37	42	44
0	3ax0	0	181182	174612	1.03762628	229717	37681	212148	43050
0.5x	3ax1	1.3	330075	176306	1.872171112	422172	69192	215461	46099
1x	3ax2	2.5	477774	158060	3.022738201	602327	98411	192080	41972
2x	3ax3	5.04	487346	126035	3.866751299	614130	98501	154353	34386



	data file	conc	68	72	ratio	39	37	42	44
0	scorn1	0	60076	98287	0.611230376	76025	11693	131287	26397
0.5x	scorn2	1.3	165426	97592	1.695077465	215369	35586	121105	26302
1x	scorn3	2.5	306329	118926	2.575795032	391088	64311	146652	32264
2x	scorn4	5.04	440093	97627	4.507902527	565103	91949	118147	24997



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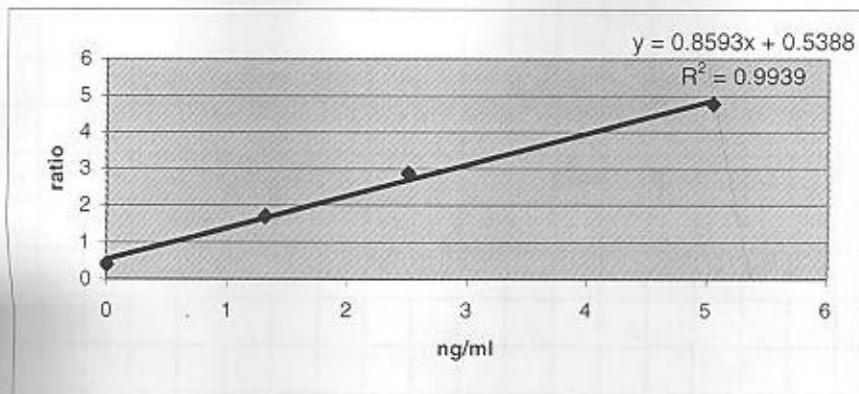
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18/7/08

d4-furan 0.67 ug/ml  
furan 0.63 ng/ml 0.20,40,80 ul

	data file	spike conc	68	72	ratio	39	37	42	44
0	2lcorn1	0	41470	100216	0.413806179	51833	nd	124039	nd
0.5x	2lcorn2	1.3	181760	107058	1.697771302	226693	nd	146705	nd
1x	2lcorn3	2.5	286190	99601	2.873364725	363096	59991	96982	nd
2x	2lcorn4	5.04	510323	107070	4.766255721	645148	104236	95411	nd



3.15ng/ml

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<i>Yuan Li</i>	7/18/08	Recorded by:	)

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Book No. \_\_\_\_\_

TITLE LOD and LOQ

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7/28/08

## Procedure.

## 1) Prepare furan stock solution

1.1) Take balance after put 40 ml vial on t

1.2) Pipette 20 ml of water

1.3) Weigh the water = 19.90346 g (H<sub>2</sub>O)

1.4) Bring out furan and add 4 drops

1.5) Weigh water + furan = 19.95725 (H<sub>2</sub>)1.6) Furan stock solution =  $\frac{19.95725 - 19.90346}{20 \text{ ml}} = 0.00269 \text{ g/ml}$ 

$$= 2.7 \text{ mg/ml}$$

## 2) Prepare furan working solution

2.1) Use micropipette to pipette 50 µl of 2.7 mg/ml into 40 ml vial containing 20 ml water

$$\text{The concentration of working sol}^{1/2} = \left( \frac{50 \times 2.7}{1000} \right) \times \frac{1}{20.05} = 6.7 \mu\text{g/ml}$$

2.2) Dilute 2.1 sol<sup>1/2</sup> by pipette 200 µl of 6.7 µg/ml into a 40 ml vial containing 20 ml of water

$$\text{The concentration is } \left( \frac{200 \times 6.7}{1000} \right) \times \frac{1}{20.2} = 66.3 \mu\text{g/ml}$$

2.3) Prepare 0.3 and 0.5 ng by putting 92 µl of 66.3 µg/ml into 20 ml prepelle and 152 µl of 66.3 µg/ml into 20 ml water to achieved 0.3 &amp; 0.5 µg/ml respectively.

2.4) Prepare 0.2 ng/ml by prepare 33 ng/ml furan (10 µl of 6.7 µg/ml into 20 ml) and put 60 µl of 33 ng/ml into 10 ml of water

3) Met run furan M GC/MS and analyze S/N ratio (Triplicate)  
Results.

To Page No. \_\_\_\_\_

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Gene Rij

Date

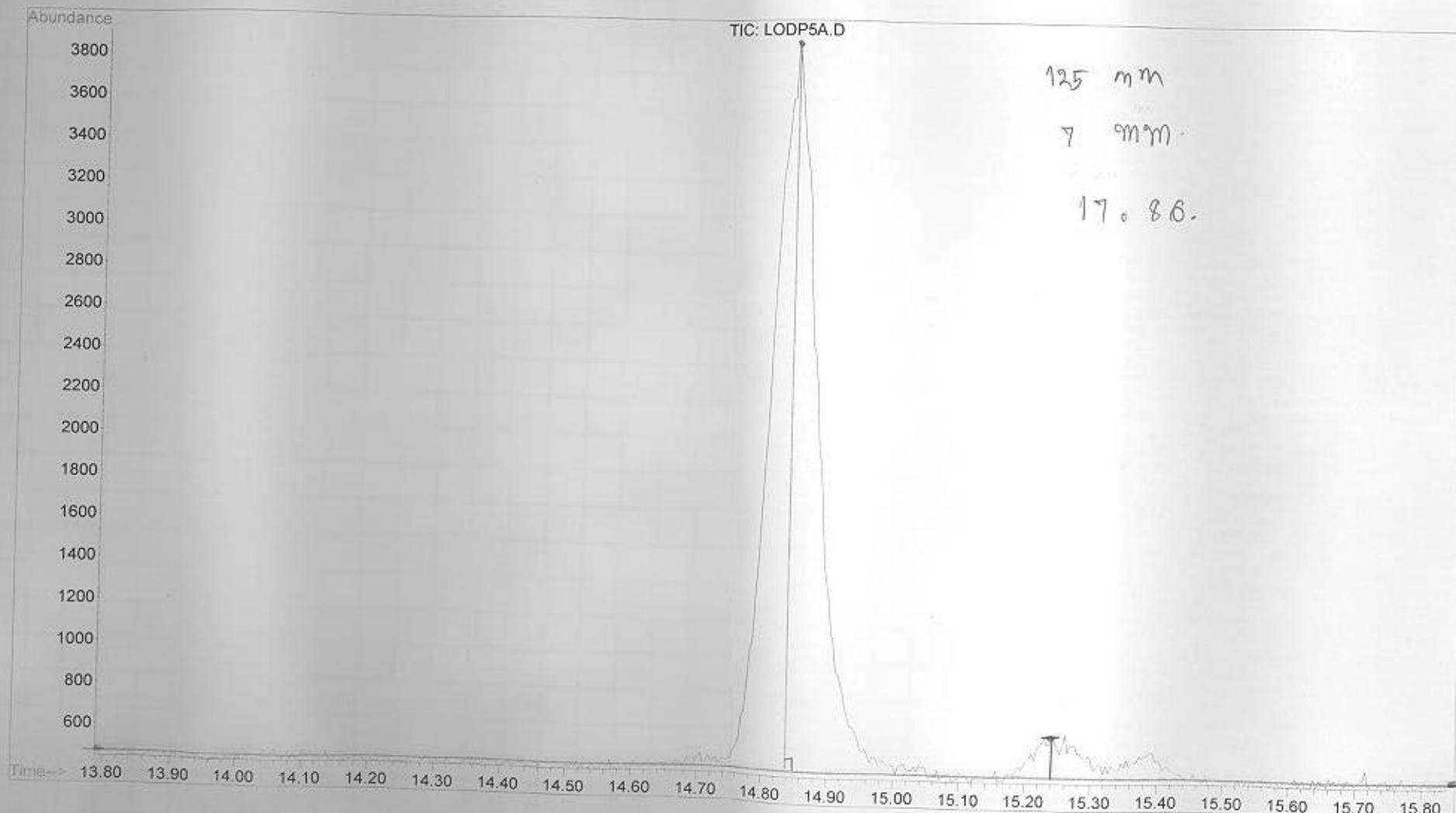
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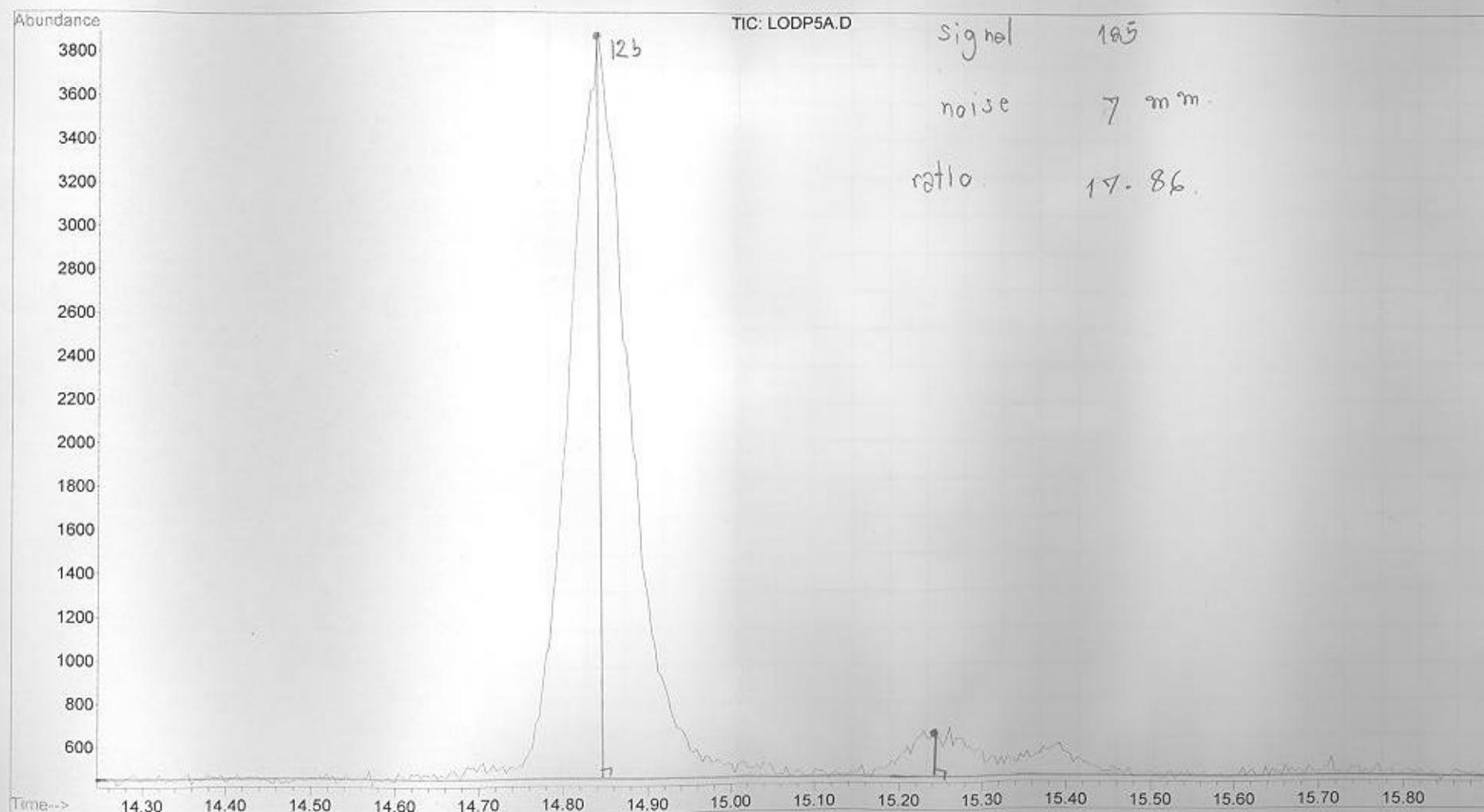
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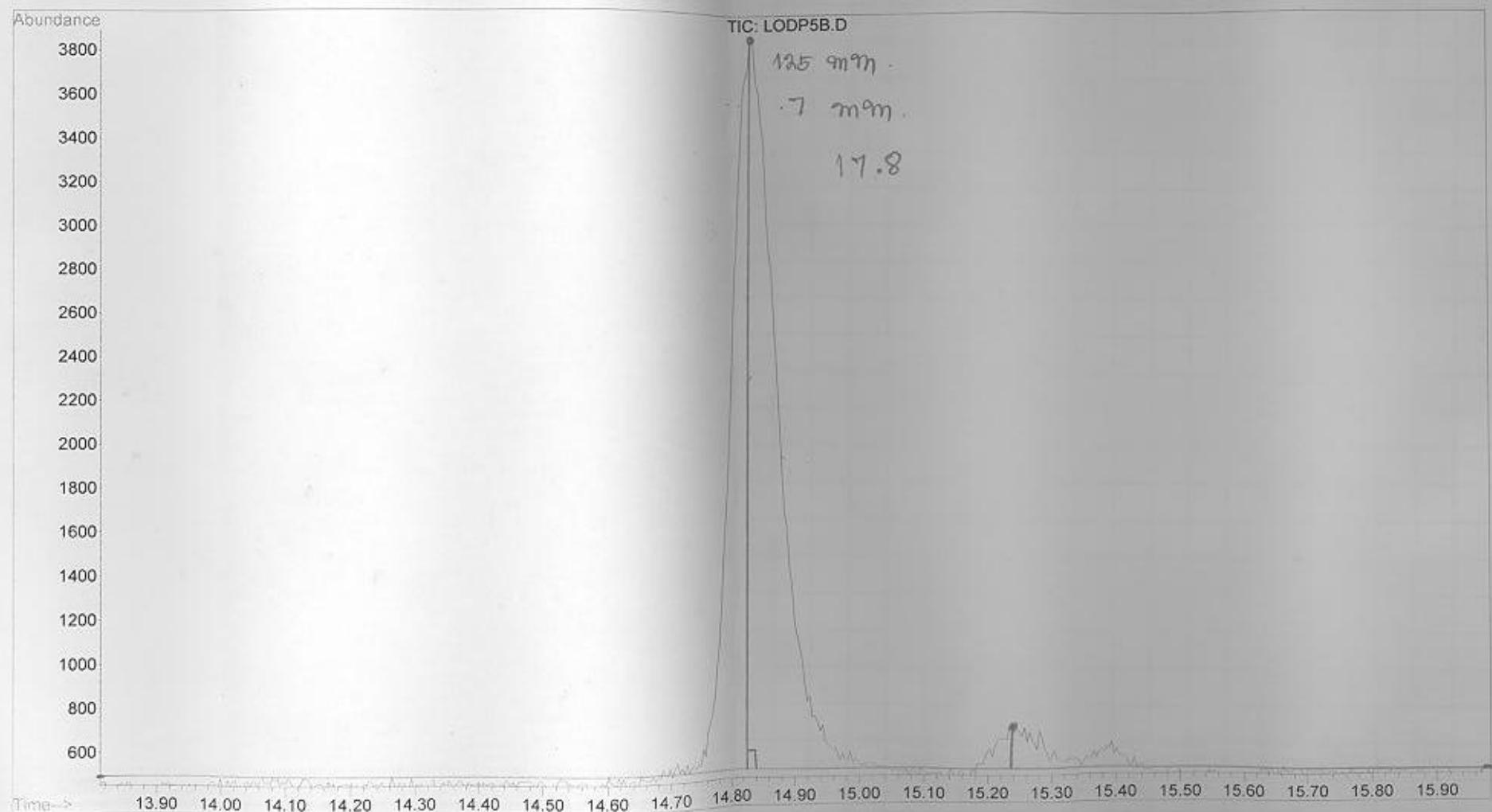
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Instrument : GC/MS Ins  
Sample Name: 0.5ng/ml  
Misc Info :  
Vial Number: 7



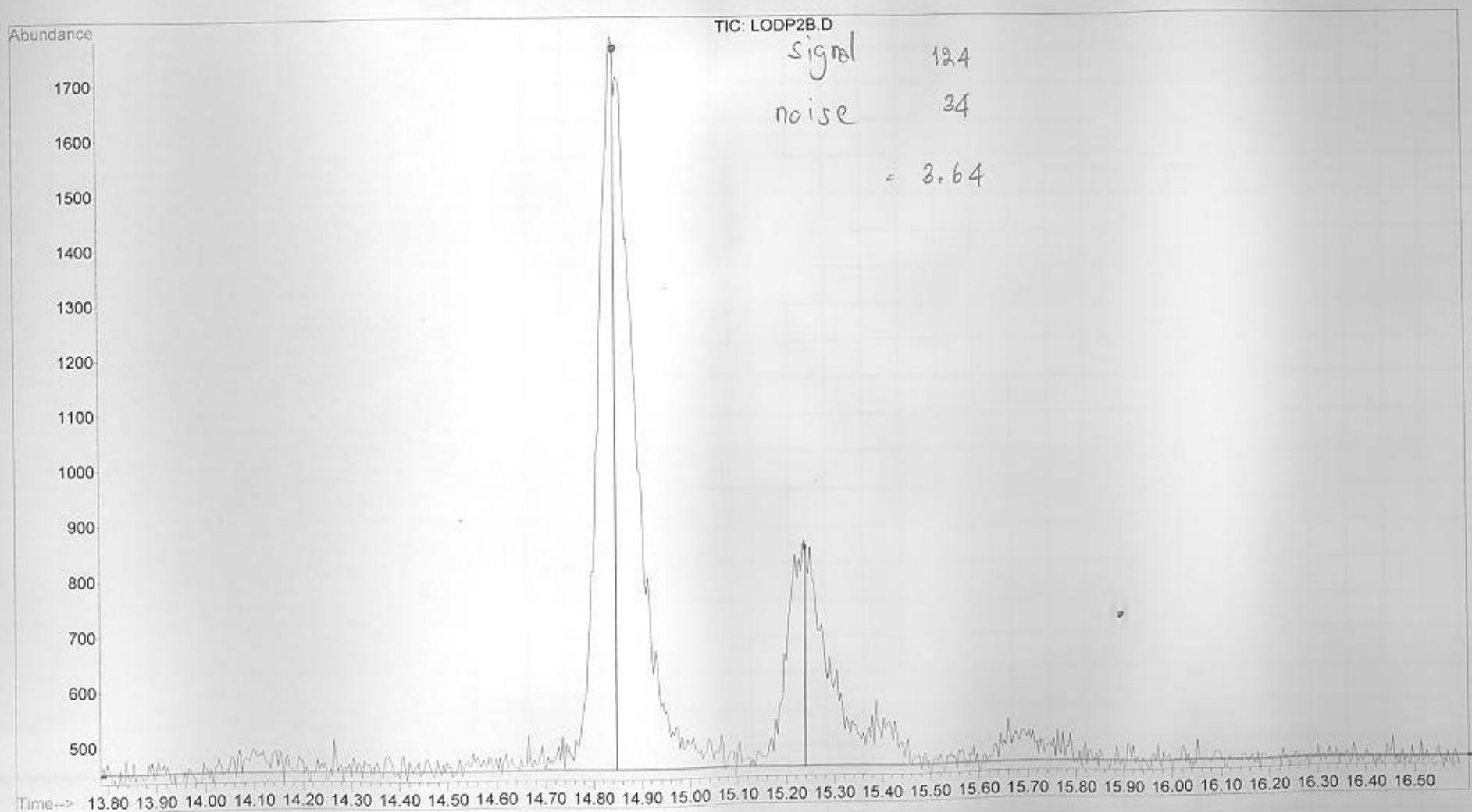
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Misc Info :  
Vial Number: 7



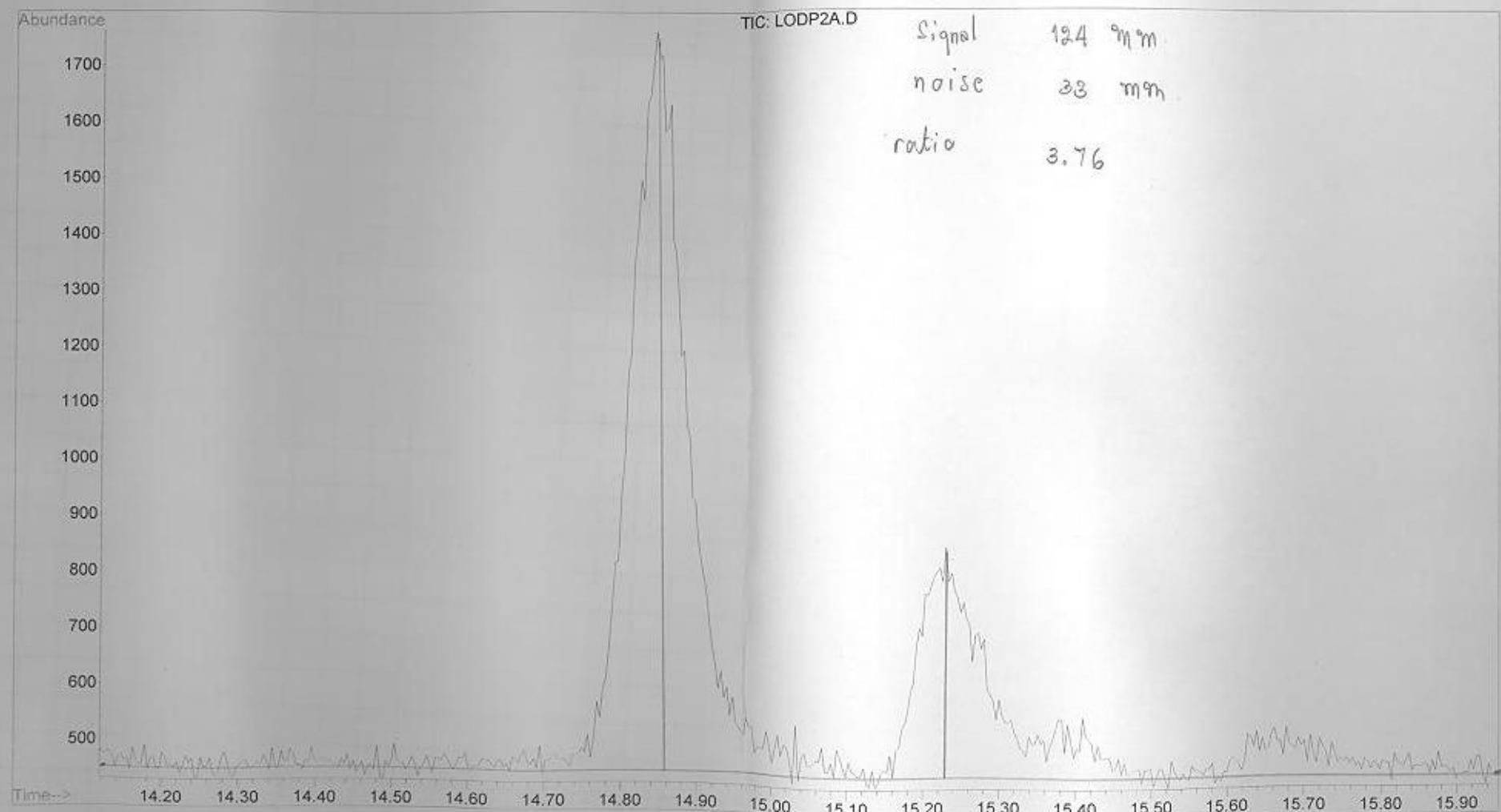
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Sample Name: 0.5ng/ml  
Misc Info :  
Vial Number: 8



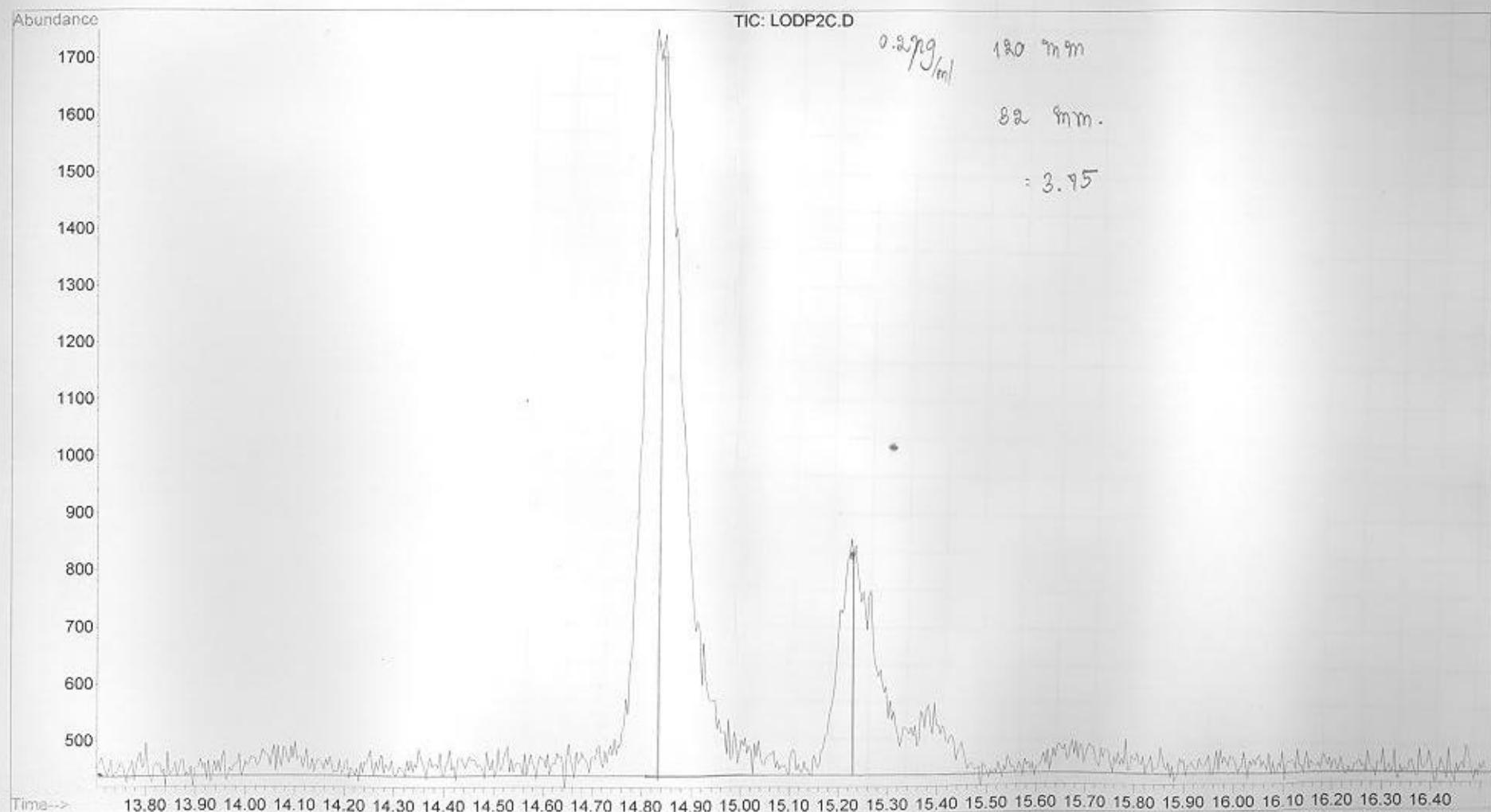
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Instrument : GC/MS Ins  
Sample Name: 0.2ng/ml  
Misc Info :  
Vial Number: 5



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Operator :  
Acquired : 28 Jul 2008 17:51 using AcqMethod FURAN  
Instrument : GC/MS Ins  
Sample Name: 0.2ng/ml  
Misc Info :  
Vial Number: 4



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Operator :  
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Instrument : GC/MS Ins  
Sample Name: 0.2ng/ml  
Misc Info :  
Vial Number: 6

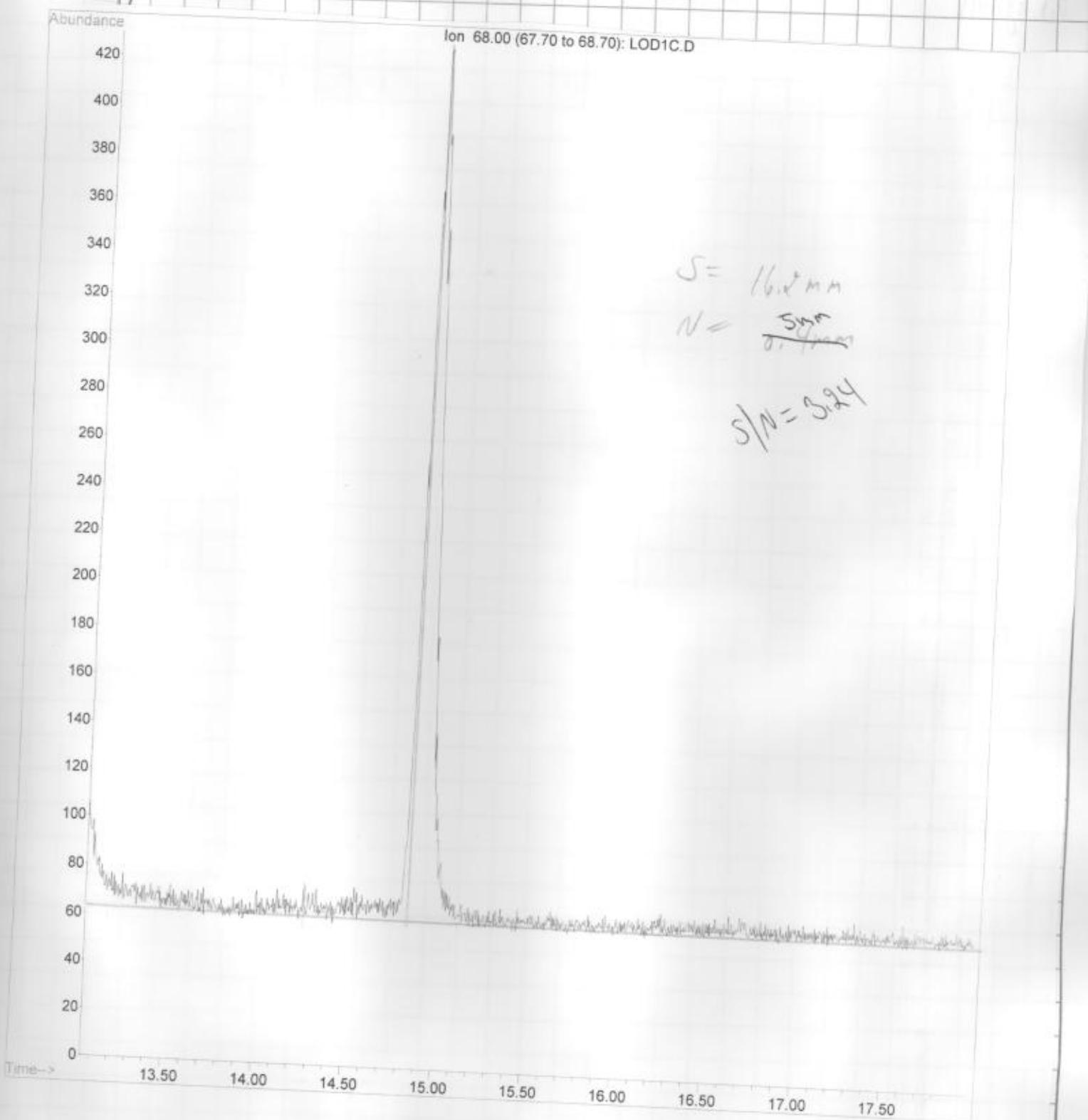


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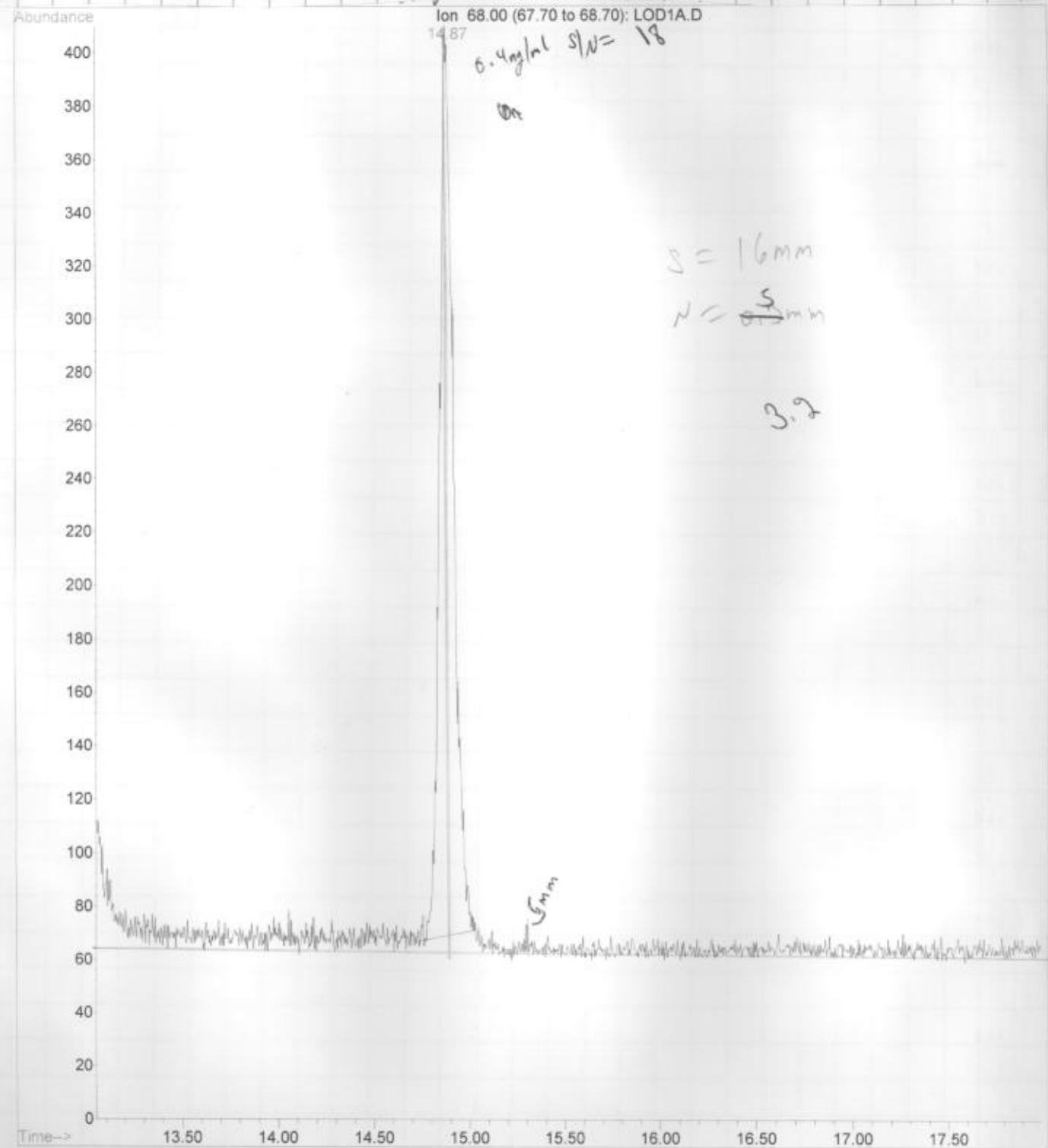
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0.1 ng/ml



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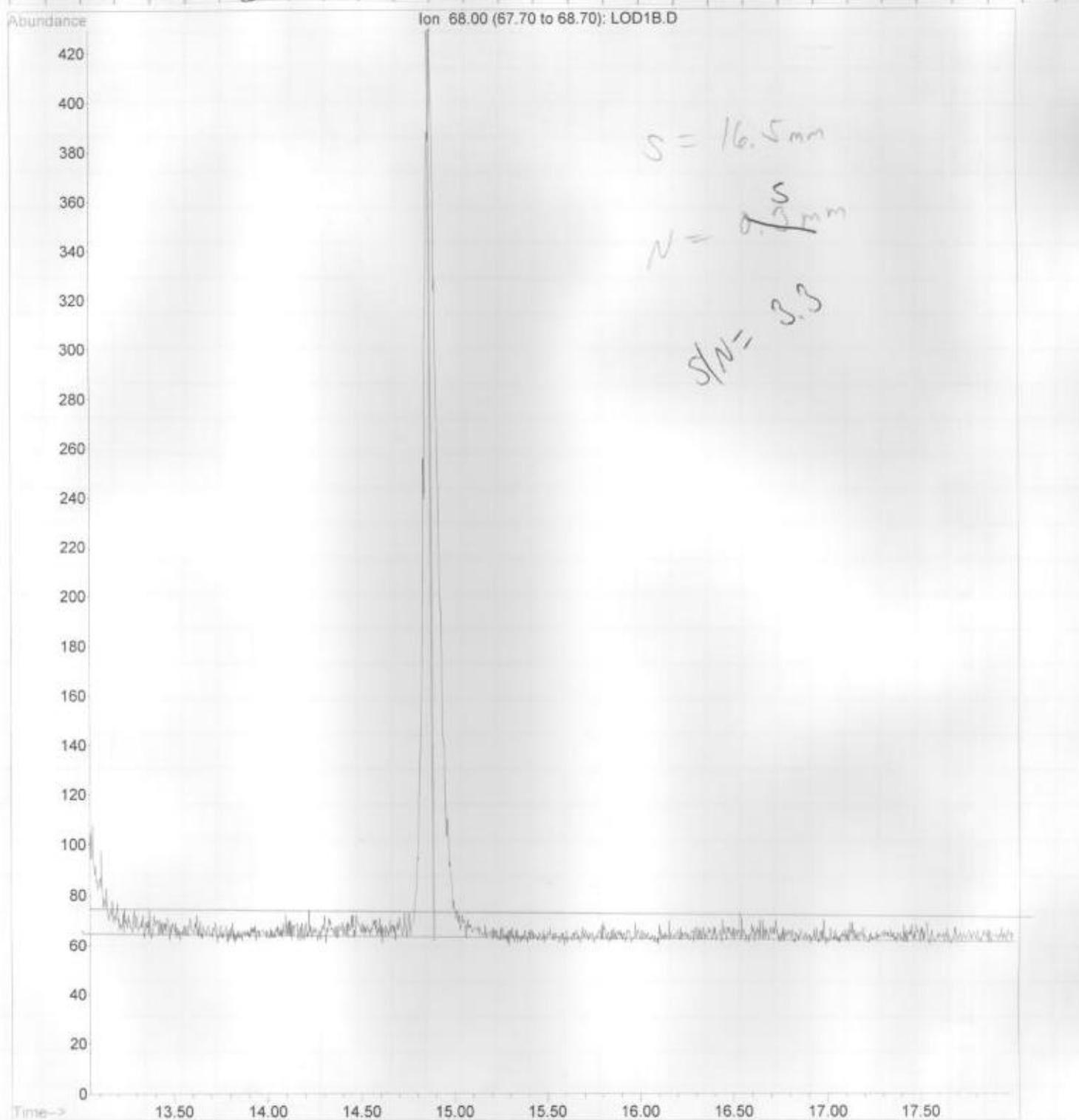
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0.1 ng/ml



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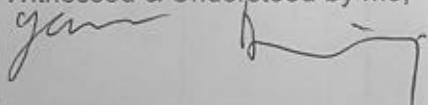
Procedures.

- 1) Bring out 19.8 µg/ml Furam working solution, prepared by Padma on 7/31/08
- 2) Take 85 µl of ① into 10 ml of water → 0.05 µg/ml
- 3) Take 200 µl of ② into 10 ml of water → 0.1 µg/ml
- 4) Furam analysis. (Triplicate)

Results.

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0.05 nglml

Abundance

TIC: LODP05A.D

1000

950

900

850

800

750

700

650

600

550

500

450

400

Time--&gt;

13.50

14.00

14.50

15.00

15.50

16.00

16.50

17.00

17.50

S 17.2 nm

N 54 nm.

3. 18.

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<i>John D. J.</i>		8/6/08			Recorded by:		

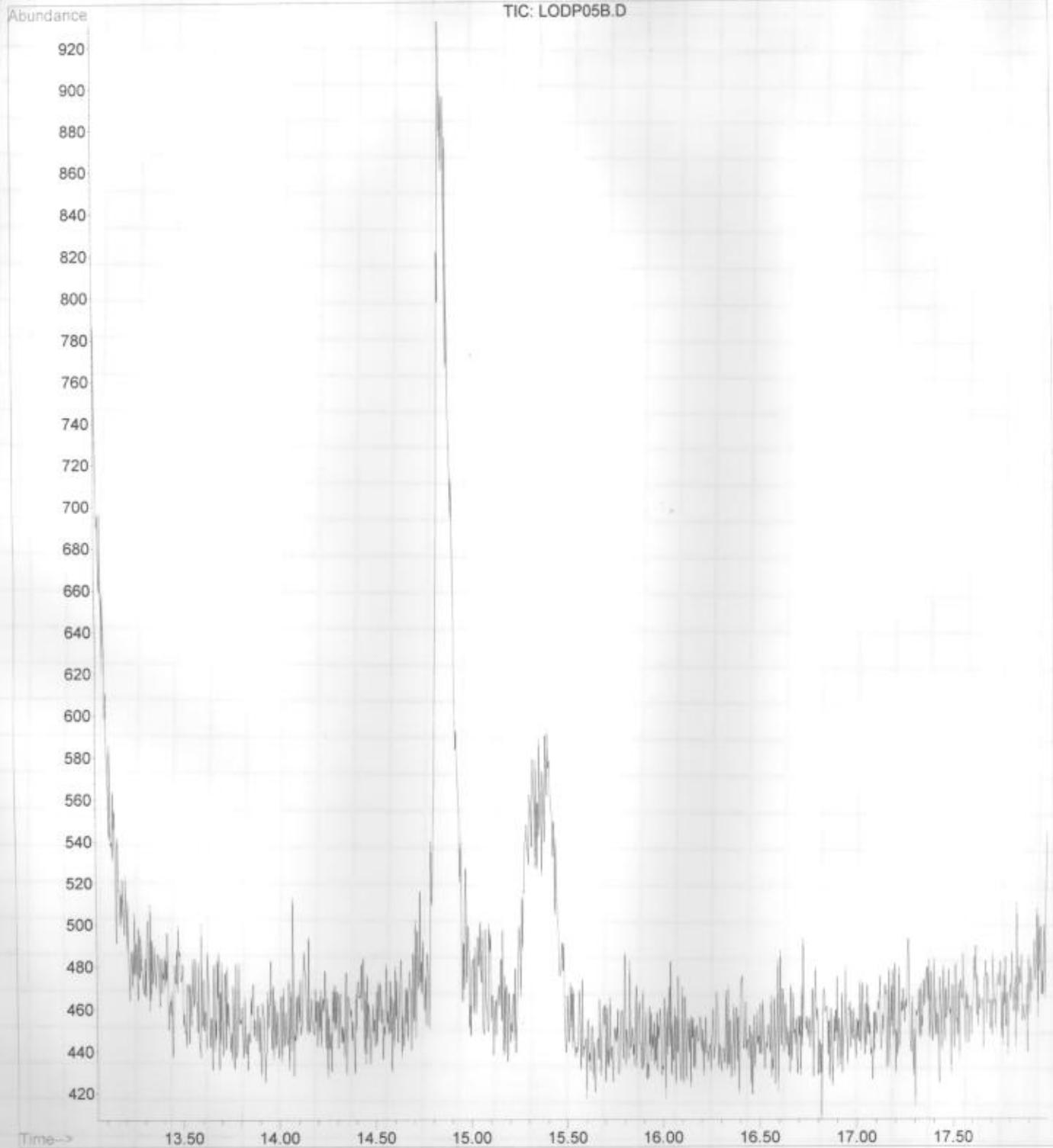
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Book No. \_\_\_\_\_

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From Page No. \_\_\_\_\_  
0.09 ng/ml

TIC: LODP05B.D



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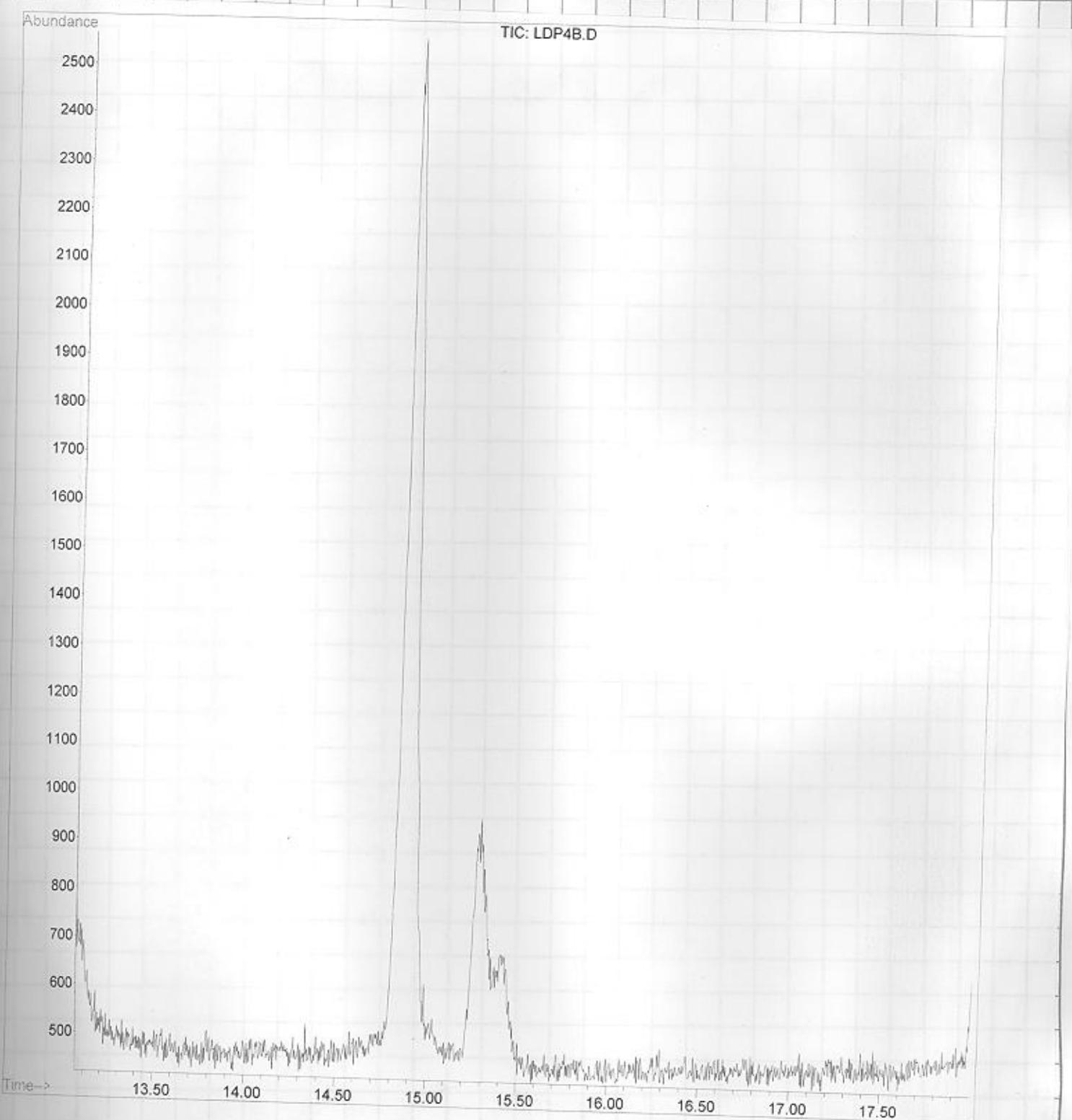
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0.4 ng /ml



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From Page No. \_\_\_\_\_ 0.4 ng (M)

Abundance

TIC: LDP4A.D

2500  
2400  
2300  
2200  
2100  
2000  
1900  
1800  
1700  
1600  
1500  
1400  
1300  
1200  
1100  
1000  
900  
800  
700  
600  
500

Time--&gt; 13.50 14.00 14.50 15.00 15.50 16.00 16.50 17.00 17.50

S 186 mm

N 38 mm -

5

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*Yaron*

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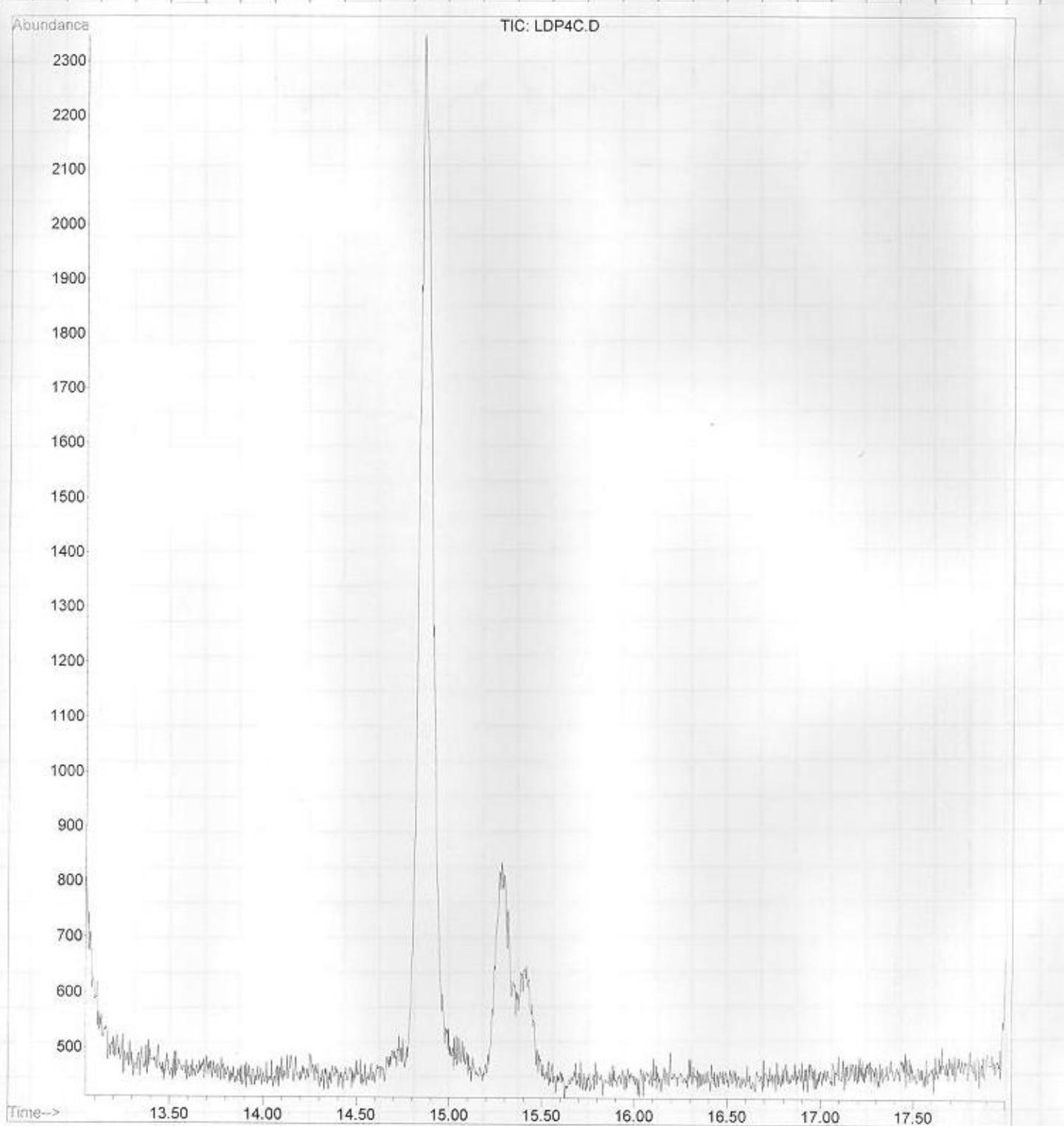
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0.4 ng/ml



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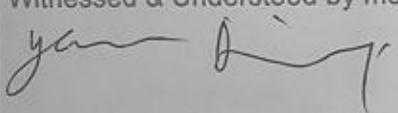
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**Conclusion**

- Based on triplicate analysis of 0.05, 0.1, 0.2, 0.4, and 0.5 ng/ml  
The LOD that gives  $S/N = 3$  is 0.1 ng/ml  
and The LOQ that gives  $S/N = 10$  is 0.5 ng/ml

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