



A Rapid iMethod™ Application for Screening Pesticides V.2.2 for Cliquant® Software



A Rapid iMethod™ Application for Screening Pesticides V.2.2 for Cliquid® Software

Tools for routine testing labs for easy implementation of pesticide screening in food, water, and other consumer products

Overview

With more than 800 pesticides currently in use there is growing concern by government agencies about the effect of pesticide exposure on human health. As a result, the residue level of pesticides in food, water and other consumer products are highly regulated. While analytical techniques like GC or GC/MS have traditionally been used for pesticide analysis, LC/MS/MS has quickly become the technique of choice due to the ability to analyze a more diverse set of pesticides, faster with better sensitivity and less sample preparation.

The following description outlines the instrument requirements and expected results obtainable from the AB SCIEX iMethod™ Application for Pesticide Screening and Quantitation using an AB SCIEX 3200 QTRAP® system or API 3200™ system. This iMethod™ application consists of a pre-configured method to screen for 534 pesticides; a positive polarity method and a negative polarity method to screen for the analytes in EU MRL list; and individual quantitation methods for carbamate, phenyl urea, triazine, organophosphorus, and acidic pesticides.

All methods have also been verified for use on AB SCIEX 4000 QTRAP®, API 4000™, QTRAP® 5500 and AB SCIEX Triple Quad™ 5500 systems.

The iMethod™ Application also includes the *iDQuant™* Pesticide Standard Kit containing 204 compounds distributed in 10 vials for easy preparation of standards. See Tables 2 – 11 for a comprehensive list. More in-depth sample preparation, and instrument parameter information is included as part of the standard operating procedure provided with the method, as are the required analytical columns. Solvents, standards and any supplies required for sample preparation are not included. Please note that the use of QTRAP® system technology is recommended for use with the screening method provided and that the associated library is not included and may be purchased separately.

Experimental details

The pesticide screening method included in this iMethod™ application is for the routine screening of 534 pesticides from food samples using the QuEChERS extraction and cleanup technique. The method uses external calibration standards and matrix spike recoveries to correct for sample and instrument variability and is based upon the use of a 3200 QTRAP® LC/MS/MS system.

A suggested method for the extraction of pesticides from vegetable, nut and citrus plant materials is also provided and is based upon the use of widely available QuEChERS tubes. Separation is achieved using a simple methanol / water gradient with ammonium formate with a Phenomenex Synergi 2.5 µm Fusion 100 Å, 50 x 2 mm HPLC column that is included with this iMethod™ application. Table 1 provides representative recoveries, signal-to-noise ratios, and estimated detection limits for 130 selected pesticides spiked at 25 ng/mL in a cucumber matrix using QuEChERS sample processing technique.

Quantitation Methods

The following information outlines the list of standard compounds in each of the quantitation methods for each compound class listed above. The methodology presented below was developed to quantify each pesticide when run by compound class, at < 10 ppb in order to meet regulatory requirements. The 603-compound MRM catalogue provided can also be used to create new customized tests according to any combinations required.

Example sample preparation procedures are provided for fruits and vegetables, based upon a simple sample homogenization, centrifugation, extraction and dilution. These procedures may require additional optimization based upon the actual composition and consistency of the fruit or vegetable under investigation. Deuterated and/or C13-labeled internal standards of known concentrations are added during sample preparation to monitor sample recovery.

Figure 1: Chromatogram of a 303 pesticides mix on an API 3200™ LC/MS/MS system (10 ng/mL)

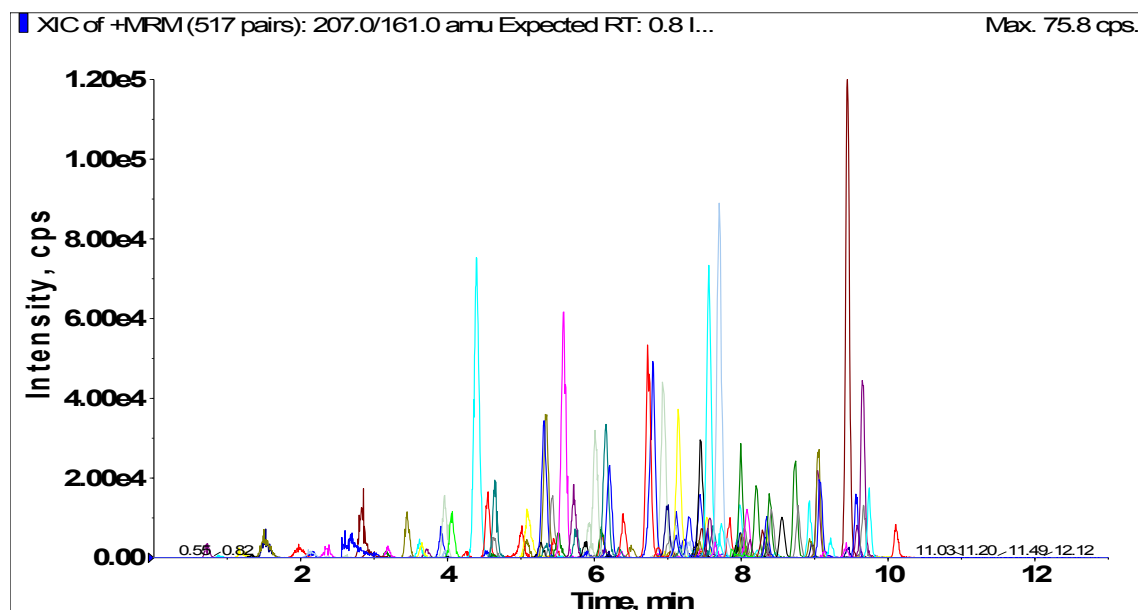


Table 1.

Representative recoveries, signal-to-noise ratios and estimated detection limits for 130 selected pesticides spiked at 25 ng/mL in a cucumber matrix using QuEChERS sample processing, as run on the 3200 QTRAP® LC/MS/MS system

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Aldicarb-sulfoxide	97.0%	26	4.2%	9.6
Aldoxycarb	96.4%	33.2	10.0%	7.5
Alloxydim	60.8%	32.2	7.5%	7.8
Aminocarb	84.2%	84	2.6%	3.0
Atrazine	65.1%	34	2.4%	7.4
Atrazine-2-hydroxy	79.9%	77.8	2.9%	3.2
Atrazine-desethyl-2-hydroxy	104.1%	38.8	1.9%	6.4
Benalaxyl	94.9%	39.5	18.1%	6.3
Bendiocarb	120.2%	33.1	16.2%	7.6
Benzoximate	119.9%	29.3	10.0%	8.5
Boscalid	15.3%	14.3	18.7%	17.5
Bromacil	99.0%	15.9	8.4%	15.7
Bupirimate	106.2%	43.8	9.0%	5.7
Butocarboxim-sulfoxid	102.2%	37.7	2.3%	6.6
Buturon	98.6%	20.4	4.1%	12.3
Carbaryl	98.4%	44.7	1.5%	5.6
Carbetamide	94.3%	58.8	4.9%	4.3
Chloridazon	111.1%	35.5	1.7%	7.0
Chlorotoluron	149.2%	38.1	2.4%	6.6
Chloroxuron	69.5%	24.7	11.0%	10.1
Cinosulfuron	88.6%	28.8	18.9%	8.7
Clomazone	107.2%	42.3	1.8%	5.9
Cloquintocet-mexyl	140.8%	95.8	10.1%	2.6
Coumaphos	120.8%	24.4	3.6%	10.2
Cyanazine	115.6%	22.1	20.1%	11.3
Cyproconazole	48.6%	24.6	7.1%	10.2
Cyprodinil	120.2%	11.3	7.6%	22.1

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Demeton-S-methyl-sulfone	90.0%	32.8	1.9%	7.6
Diazinon	100.5%	81.9	7.2%	3.1
Diclobutrazol	88.3%	26.3	7.6%	9.5
Dicrotophos	112.7%	58.1	19.1%	4.3
Diethofencarb	28.5%	16.2	8.0%	15.4
Difenoxyuron	104.0%	51	5.3%	5.0
Dimefuron	92.5%	20	6.6%	12.6
Dimethachlor	88.8%	13	11.8%	19.5
Dimethenamide	100.5%	39	2.7%	6.5
Dimetilan	106.0%	101	2.5%	2.5
Dodemorph	67.0%	107	3.0%	2.3
Ethidimuron	139.0%	28	6.5%	8.9
Ethiofencarb-sulfon	113.0%	59	14.7%	4.3
Ethiofencarb-sulfoxid	99.1%	119	3.6%	2.1
Ethion	113.9%	17	7.2%	14.8
Ethirimol	99.6%	42	4.9%	6.0
Ethoprophos	112.9%	16	14.1%	15.2
Etrimfos	93.3%	64	4.6%	3.9
Fenbuconazole	93.0%	21	2.2%	11.9
Fenfuram	98.0%	46	7.7%	5.5
Fenhexamid	47.9%	15	15.1%	17.0
Fenoxaprop-P-ethyl	113.9%	32	7.2%	7.8
Fenpropimorph	130.8%	152	4.2%	1.6
Fenpyroximate	161.6%	15	8.4%	16.8
Fenuron	102.8%	47	5.5%	5.3
Flamprop-M-isopropyl	104.1%	55	4.1%	4.5
Flamprop-M-methyl	90.2%	20	3.7%	12.4
Flazasulfuron	100.8%	74	7.5%	3.4
Fluazifop-butyl	144.5%	50	3.6%	5.0
Flufenacet	89.7%	22	12.8%	11.5
Fluometuron	120.9%	77	2.3%	3.2
Fluridone	68.4%	30	11.8%	8.4
Flurtamone	89.7%	20	5.2%	12.3
Fuberidazole	102.7%	125	6.2%	2.0
Haloxifop-P-methyl	106.4%	15	3.2%	16.9
Hexaconazole	100.9%	23	2.1%	11.0
Hexazinone	106.3%	202	15.8%	1.2
Imazalil	117.7%	34	18.1%	7.3
Isazofos	74.9%	13	8.0%	19.1
Isoprothiolane	62.1%	77	1.2%	3.2
Lenacil	111.3%	43	6.1%	5.8
Malaoxon	44.7%	50	9.5%	5.0
Malathion	94.7%	30	5.9%	8.3
Mepanipyrim	79.5%	3	7.9%	80.6
Metconazole	118.2%	19	7.3%	13.4
Methamidophos	90.9%	33	6.5%	7.6
Methfuroxam	66.2%	96	1.1%	2.6
Methidathion	47.9%	35	4.2%	7.2
Metobromuron	116.1%	19	4.0%	12.9
Metolachlor	82.7%	22	2.6%	11.4
Metosulam	99.7%	51	2.7%	4.9
Monuron	100.3%	22	2.8%	11.6
Napropamide	103.7%	31	11.5%	8.1
Oxasulfuron	87.8%	78	6.6%	3.2

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Oxycarboxin	98.5%	110	5.3%	2.3
Paclobutrazol	105.4%	48	9.2%	5.2
Phosalone	102.9%	13	9.0%	18.9
Phosphamidon	95.2%	104	6.5%	2.4
Picoxystrobin	133.1%	104	3.8%	2.4
Pirimiphos-ethyl	129.7%	57	4.1%	4.4
Pirimiphos-methyl	105.1%	63	3.1%	4.0
Prometon	100.5%	105	6.0%	2.4
Prometryne	49.5%	14	19.5%	18.4
Propamocarb	106.5%	20	1.9%	12.8
Propazin-2-hydroxy	81.5%	19	3.6%	12.9
Propazine	72.8%	11	10.7%	22.5
Prosulfocarb	114.9%	145	6.1%	1.7
Pymetrozine	101.4%	115	2.6%	2.2
Pyraclostrobin	141.1%	24	9.6%	10.5
Pyrazophos	108.9%	55	4.8%	4.5
Pyridaben	102.8%	18	4.1%	14.1
Pyridaphenthion	88.6%	10	6.3%	25.3
Pyrimethanil	40.1%	18	5.8%	13.6
Pyriproxyfen	153.8%	51	4.3%	4.9
Quinalphos	124.9%	17	7.3%	14.8
Quinmerac	104.9%	116	1.3%	2.2
Sebuthylazine	56.9%	55	3.6%	4.6
Sebuthylazine-desethyl	88.6%	39	7.1%	6.5
Sethoxydim	82.5%	28	10.1%	8.9
Siduron	59.7%	14	5.3%	17.5
Simazine-2-hydroxy	105.5%	99	10.6%	2.5
Simetryn	95.7%	96	6.6%	2.6
Spiroxamine	97.1%	230	6.0%	1.1
Sulfometuron-methyl	98.2%	103	5.1%	2.4
Tebuconazol	87.3%	45	9.6%	5.5
Tebuconpyrad	125.2%	13	10.6%	18.8
Tebutam	87.2%	22	7.3%	11.4
Tebuthiuron	97.8%	61	14.4%	4.1
Terbufos	44.3%	4	20.3%	65.8
Terbuthylazine	31.7%	18	3.3%	14.1
Terbuthylazine-desethyl	88.7%	39	8.1%	6.5
Terbutryn	31.9%	119	6.0%	2.1
Tetrachlorvinphos	101.3%	20	8.7%	12.5
Tetraconazole	82.1%	32	16.3%	7.8
Thifensulfuron-methyl	100.8%	74	4.2%	3.4
Triasulfuron	80.5%	35	7.5%	7.2
Triazophos	31.9%	19	9.9%	13.2
Tricyclazole	93.8%	72	1.9%	3.5
Trietazine	75.4%	12	13.6%	21.0
Trifloxystrobin	113.9%	83	5.8%	3.0
Triflumizole	122.4%	25	11.9%	10.1
Triticonazole	117.1%	24	5.8%	10.6
Uniconazole	66.5%	24	24.0%	10.6
Vamidothion	113.1%	36	13.9%	7.0

* Signal-to-noise obtained at three standard deviations on a 3200 QTRAP® System.

** Estimated detection limit based on estimated signal at 10 x the Signal-to-noise ratio.

Table 2. Content iDQuant™ Pesticide Standard A1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
3-Hydroxycarbofuran	16655-82-6	Formetanate HCL	23422-53-9
Acephate	30560-19-1	Fuberidazole	3878-19-1
Aldicarb sulfone	1646-88-4	Methamidophos	10265-92-6
Aldicarb sulfoxide	1646-87-3	Methomyl	16752-77-5
Aminocarb	2032-59-9	Mevinphos	7786-34-7
Butocarboxim	34681-10-2	Monocrotophos	6923-22-4
Butoxycarboxim	34681-23-7	Omethoate	1113-02-6
Carbendazim	10605-21-7	Oxamyl	23135-22-0
Cymoxanil	57966-95-7	Propamocarb	24579-73-5
Dicrotophos	141-66-2	Thiabendazole	148-79-8
Dimethoate	60-51-5	Tricyclazole	41814-78-2
Dioxacarb	6988-21-2	Formetanate HCL	23422-53-9

Table 3. Content iDQuant™ Pesticide Standard B1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Aldicarb	116-06-3	Isoprocab	2631-40-5
Bendiocarb	22781-23-3	Metalaxyl	57837-19-1
Carbaryl	63-25-2	Methiocarb	2032-65-7
Carbetamide	16118-49-3	Mexacarbate	315-18-4
Carbofuran	1563-66-2	Oxadixyl	77732-09-3
Carboxin	5234-68-4	Pirimicarb	23103-98-2
Clethodim	99129-21-2	Promecarb	2631-37-0
Desmedipham	13684-56-5	Propham	122-42-9
Diethofencarb	87130-20-9	Propoxur	114-26-1
Ethiofencarb	29973-13-5	Pyracarbolid	24691-76-7
Furalaxyl	57646-30-7	Thiofanox	39196-18-4
Iprovalicarb	140923-17-7	Thiophanate-methyl	23564-05-8

Table 4. Content iDQuant™ Pesticide Standard C1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Alanycarb	83130-01-2	Flufenacet	142459-58-3
Amitraz	33089-61-1	Furathiocarb	65907-30-4
Benalaxyl	71626-11-4	Indoxacarb	144171-61-9
Benfuracarb	82560-54-1	Mefenacet	73250-68-7
Benzoximate	29104-30-1	Mepronil	55814-41-0
Bifenazate	149877-41-8	Piperonyl butoxide	51-03-6
Cyazofamid	120116-88-3	Quinoxifen	124495-18-7
Fenamidone	161326-34-7	Spiroxamine	118134-30-8
Fenazaquin	120928-09-8	Thiobencarb	28249-77-6
Fenhexamid	126833-17-8	Zoxamide	156052-68-5
Fenoxycarb	79127-80-3		

Table 5. Content iDQuant™ Pesticide Standard D1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Acetamiprid	135410-20-7	Metribuzin	21087-64-9
Acibenzolar-S-methyl	135158-54-2	Myclobutanil	88671-89-0
Bromucanazol	116255-48-2	Nitenpyram	120738-89-8
Clothianidin	210880-92-5	Nuarimol	63284-71-9
Cyproconazole	113096-99-4	Paclobutrazol	76738-62-0
Epoxiconazole	135319-73-2	Pyrimethanil	53112-28-0
Etaconazole	60207-93-4	Thiacloprid	111988-49-9
Fenarimol	60168-88-9	Thiamethoxam	153719-23-4
Flutriafol	76674-21-0	Triadimenol	55219-65-3
Imazalil	35554-44-0	Triticonazole	131983-72-7
Imidacloprid	138261-41-3		

Table 6. Content iDQuant™ Pesticide Standard E1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Abamectin	71751-41-2	Flutolanil	66332-96-5
Bitertanol	55179-31-2	Hexaconazole	79983-71-4
Bupirimate	41483-43-6	Mepanipyrim	110235-47-7
Butafenacil	134605-64-4	Metconazole	125116-23-6
Clofentezine	74115-24-5	Methoxyfenozid	161050-58-4
Cyprodinil	121552-61-2	Penconazole	66246-88-6
Diclobutrazol	75736-33-3	Prochloraz	67747-09-5
Difenoconazole	119446-68-3	Propiconazole	60207-90-1
Diniconazol	83657-24-3	Rotenone	83-79-4
Ethofumesate	26225-79-6	Tebufozide	112410-23-8
Fenbuconazole	114369-43-6	Triflumizole	68694-11-1
Flusilazole	85509-19-9		

Table 7. Content iDQuant™ Pesticide Standard F1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Carfentrazone-ethyl	128639-02-1	Mesotrione	104206-82-8
Doramectin	117704-25-3	Moxidectin	113507-06-5
Emamectin-benzoate	155569-91-8	Propargite	2312-35-8
Eprinomectin	123997-26-2	Spinosad	168316-95-8
Fluquinconazole	136426-54-5	Spirodiclofen	148477-71-8
Hexythiazox	78587-05-0	Spiromesifen	283594-90-1
Hydramethylnon	67485-29-4	Tebuconazol	107534-96-3
Ipconazole	125225-28-7	Tetraconazole	112281-77-3
Ivermectin	70288-86-7		

Table 8. Content iDQuant™ Pesticide Standard G1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
Chlortoluron	15545-48-9	Monolinuron	1746-81-2
Cycluron	2163-69-1	Prometon	1610-18-0
Diuron	330-54-1	Pymetrozine	123312-89-0
Fenuron	101-42-8	Secbumeton	26259-45-0
Fluometuron	2164-17-2	Simetryn	1014-70-6
Forchlorfenuron	68157-60-8	Sulfentrazone	122836-35-5
Isoproturon	34123-59-6	Tebuthiuron	34014-18-1
Methabenzthiazuron	18691-97-9	Terbumeton	33693-04-8
Methoprotryne	841-06-5	Thidiazuron	51707-55-2
Metobromuron	3060-89-7	Triadimefon	43121-43-3

Table 9. Content iDQuant™ Pesticide Standard H1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
<i>Ametryn</i>	834-12-8	<i>Hexaflumuron</i>	86479-06-3
<i>Azoxystrobin</i>	131860-33-8	<i>Kresoxim-methyl</i>	143390-89-0
<i>Boscalid</i>	188425-85-6	<i>Linuron</i>	330-55-2
<i>Chloroxuron</i>	1982-47-4	<i>Neburon</i>	555-37-3
<i>Diflubenzuron</i>	35367-38-5	<i>Phenmedipham</i>	13684-63-4
<i>Dimethomorph</i>	110488-70-5	<i>Picoxystrobin</i>	117428-22-5
<i>Dimoxystrobin</i>	149961-52-4	<i>Prometryne</i>	7287-19-6
<i>Famoxadon</i>	131807-57-3	<i>Siduron</i>	1982-49-6
<i>Fipronil</i>	120068-37-3	<i>Terbutryne</i>	886-50-0
<i>Fludioxonil</i>	131341-86-1	<i>Triflumuron</i>	64628-44-0

Table 10. Content iDQuant™ Pesticide Standard J1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
<i>Buprofezin</i>	69327-76-0	<i>Lufenuron</i>	103055-07-8
<i>Chlorfluazuron</i>	71422-67-8	<i>Novaluron</i>	116714-46-6
<i>Ethiprole</i>	181587-01-9	<i>Prothioconazole</i>	178928-70-6
<i>Etoxazole</i>	153233-91-1	<i>Pyraclostrobin</i>	175013-18-0
<i>Fenpropimorph</i>	67564-91-4	<i>Pyridaben</i>	96489-71-3
<i>Fenpyroximate</i>	111812-58-9	<i>Pyriproxyfen</i>	95737-68-1
<i>Fluazinam</i>	79622-59-6	<i>Tebufenpyrad</i>	119168-77-3
<i>Flufenoxuron</i>	101463-69-8	<i>Teflubenzuron</i>	83121-18-0
<i>Fluoxastrobin</i>	361377-29-9	<i>Trifloxystrobin</i>	141517-21-7

Table 11. Content iDQuant™ Pesticide Standard K1

Pesticide Name	CAS Number	Pesticide Name	CAS Number
<i>Chlorantraniliprole</i>	500008-45-7	<i>Isocarboxiphos</i>	24353-61-5
<i>Cyromazine</i>	66215-27-8	<i>Mandipropamid</i>	374726-62-2
<i>Dinotefuran</i>	165252-70-0	<i>Metaflumizone</i>	139968-49-3
<i>Ethirimol</i>	23947-60-6	<i>Pencycuron</i>	66063-05-6
<i>Fenobucarb</i>	3766-81-2	<i>Spinetoram</i>	187166-40-1
<i>Flonicamid</i>	158062-67-0	<i>Spirotetramat</i>	203313-25-1
<i>Flubendimide</i>	272451-65-7	<i>Temephos</i>	3383-96-8
<i>Halofenozide</i>	112226-61-6	<i>Trichlorfon</i>	52-68-6

Table 12. List of triazine pesticides with retention times for each

Triazines	RT (Min)	Triazines	RT (Min)
<i>Atrazine</i>	8.5	<i>Prometon</i>	8.3
<i>Ametryn</i>	7.8	<i>Prometryn</i>	9.0
<i>Cyanazine</i>	6.7	<i>Propazine</i>	8.6
<i>Desisopropyl-atrazine</i>	5.4	<i>Sebutylazine</i>	8.4
<i>Desethyl-atrazine</i>	4.0	<i>Simazine</i>	6.9
<i>Hexazinon</i>	6.9	<i>Terbutylazine</i>	8.6
<i>Metazachlor</i>	7.8	<i>Terbutryn</i>	9.1
<i>Metolachlor</i>	9.2	<i>D5-atrazine (internal std)</i>	7.7
<i>Metribuzin</i>	7.4		

Figure 2: Chromatogram of a 300 pesticides mix spiked by API 3200™ LC/MS/MS system (100 ng/mL)

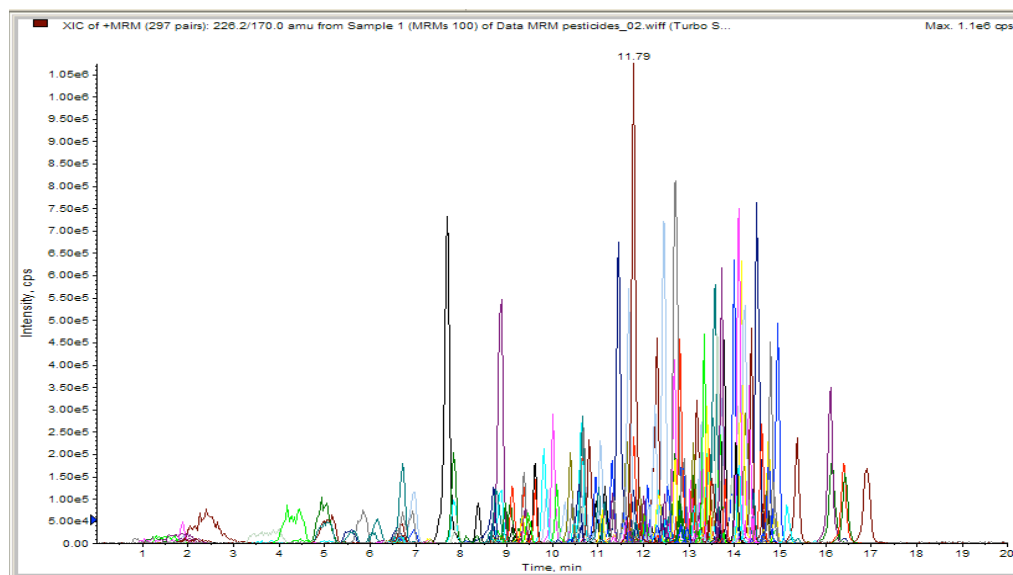
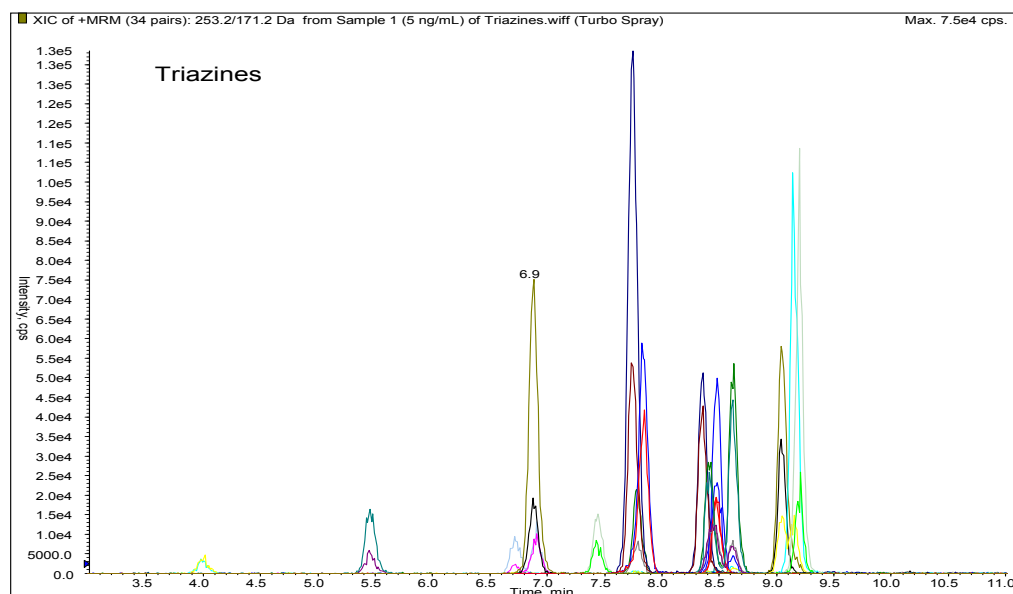


Figure 3: Chromatogram of triazine pesticides at 5 ng / mL



Get additional details on customizing a pesticide screen for your specific laboratory application by contacting AB SCIEX at support@absciex.com.

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