رقم البحث (44)

LC – MS/MS ANALYSIS FOR ZERANOL RESIDUES IN BEEF AND CATTLE LIVERS AND FOR SOME ANTIMICROBIALS RESIDUES IN CHICKEN

BY

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ABSTRACT

A total of 75 beef and chicken tissues samples, distributed as 20 imported frozen beef and 10 imported frozen cattle livers samples (represented several meat - exporting foreign countries), besides locally – manufactured meat products consisted of 10 samples each of beef luncheon and canned beef, plus 5 samples of pastirma (dried and cured beef) (represented many Egyptian meat plants) purchased from shops and groceries, in addition to 20 chicken breast samples taken form correspondent number of locally – reared birds after being slaughtered and dressed at different Egyptian poultry shops. Beef, its products and livers samples were investigated for zeranol (synthetic growth promoter having estrogenic activity) residues, whereas chicken breast samples analyzed for some, common antimicrobials residues. Qualitative and quantitative detection of all beef and chicken tissues residues were performed by using a validated liquid chromatography coupled with a tendem mass spectrometry (LC – MS / MS) technique.

Our findings declared absence of zeranol residues in imported frozen beef and cattle livers samples as well as in locally – manufactured beef luncheon, canned beef, and pastirma (dried & cured beef) samples that derived from the imported frozen beef. Also, neither chloramphenicol, penicillin, nor tetracycline residues could be detected in tested chicken breast samples. Whereas, the residues of florfenicol were recognized in 7 (35%), of sulphaquinoxaline in 3 (15%), and of tylosin in 15 (75%) of such chicken samples. The lowest quantities of the determined antimicrobials residues (florfenicol -sulphaquinoxaline - tylosin) were 0.032, 1.101, and 0.696 micrograms per each kilogram of chicken breast

samples (µg / kg = ppb), meanwhile, the highest levels for the same antimicrobials residues were 2.363, 3.090 and 8.160 µg/kg, respectively. By comparison, non of the quantified antimicrobials residues levels was exceeded the maximum residue limit (MRL) that recommended by several regulatory agencies as 100 µg/ kg for each antimicrobial residue.

Effects of chicken cooking by boiling water and roasting – on its antimicrobials residues, public health impact of surveyed chemical residues (zeranol & antimicrobials) besides the recommendations essential to ensure safety margin for consumers were also literature and discussed.

INTRODUCTION

Hormones are chemical messengers that are secreted into the blood to control various processes within the body including growth. They may be given to cattle for a number of therapeutic or other veterinary reasons and are also used in the US and some other countries to boost the growth rate of cattle reared for beef production. To date, six different hormones have been approved for such use in cattle in the US. They include three naturally occurring hormones as well as three synthetic substances that mimic the action of these hormones. **Zeranol** is a synthetic hormone that mimics the action of oestradiol. It is often given as the sole component of implants at doses up to about 72 mg.

Chicken is a flesh derived mainly from chicken broilers and is considered as the favorite food for Egyptians, due to its a relatively low price when compared with red meat. A large number of drugs are nowadays used to control or prevent infections or to promote growth in modern poultry production system. Antimicrobials including antibiotics and sulphonamides are the two of the oldest groups used in veterinary medicine. Antibiotics are widely used in poultry farms as dietary supplements. **(Afify, 2010)**.

Residual antibiotics in food constitute a risk to human health. Their presence in food can provoke allergic reactions in some hypersensitive individuals and may compromise the human immune system. Even more important, the presence of subtherapeutic doses of the above drugs in foodstuffs for long periods has led to the problem of drug-resistant pathogenic bacterial strains. To ensure the safety of food for consumers, the World Health Organization (WHO) and the Food and Agriculture Organization (FAO) have proposed standards of residual antibiotics to animal food since early 1969, and the US Food and Drug Administration (FDA), the European Union (EU), and the State Food and Drug Administration (SFDA) in People's Republic of China have set maximum residue limits

(MRLs) for antibiotics in food. The extremely low part per billion (ppb or μg/kg) levels at which an antimicrobial residue need to be analyzed complicates the analysis. MRLs are fixed at the parts per million level (ppm or mg/kg) or even at the ppb (μg/kg) level depending on the antibiotic.

This study aimed to survey the occurrence and quantification of the probable residues of zeranol in imported frozen beef and livers as well as locally-manufactured beef luncheon, canned beef and pastirma, in addition to some common antimicrobials (chloramphenicol – florfenicol – penicillin – sulphaquinoxaline – tetracycline – tylosin) in chicken meat samples marketed in Egypt. To achieve these goals, we used the LC-MS/MS method validated according to European Commission Decision 2002/657/EC for confirmatory assay **(European Commission, 2002)**.

MATERIALS AND METHOD

Collection Of Samples:

A total of 75 beef and chicken tissues samples, distributed as 20 imported frozen beef and 10 imported frozen cattle livers samples (represented several meat – exporting foreign countries), besides locally - manufactured meat products consisted of 10 samples each of beef luncheon and canned beef, plus 5 samples of pastirma (dried and cured beef) (represented many Egyptian meat plants) purchased from shops and groceries, in addition to 20 chicken breast samples taken from correspondent number of locally – reared birds after being slaughtered and dressed at different Egyptian poultry shops. Each tissue sample was represented by either 100 g (for non – canned tissues) or by a whole can pack (for canned beef). Each of non – canned samples, was individually packed into a clean polyethylene bag, marked, then kept in an ice box and transferred – without delay – to the central laboratory for food and feed/Agricultural Research Center/Giza – Egypt, wherein prepared. Beef, its products and livers samples were investigated for zeranol residues, whereas chicken breast samples analyzed for some common antimicrobials residues.

PREPARATION OF SAMPLES (Chen and Fang, 2011):

Five grams – from each tissue sample – were added to 20 ml of extracting buffer solution in a clean 250 – ml flask then shacked by the aid of electric shaker for 30 minutes. The resultant mixture was put in a stomacher bag and homogenized for 2 minutes. The homogenate was filtered twice, the first by Whatmann filter paper whereas the latter by the

aid of 0.45 μm-syringe filter (PVDF). The filtered extract sample was diluted by adding 990 μL diluent buffer to 10 μL of it. The diluted filtered extract sample kept in a clean tube at 4°C after being marked, awaiting LC – MS/ MS analysis.

Extracting buffer solution composed of a mixture of two solutions: 236.1 ml from (13.9 g sodium phosphate monobasic + 500 ml dist. water) and 36.9 ml from (14.2 g sodium phosphate dibasic + 500 ml dist. water) then completed to 600-ml volume by adding 327 ml dist. water.

Diluent buffer solution used for diluting both tested samples and related reference standard(s) – is a special buffer for LC – MS/ MS analysis and consisted of a mixture of 5 acetonitrile + 95 dist. water + 0.1% formic acid, however the composition of diluent buffer was changeable according to the type of electric charge of analyte ion, as the aforementioned formula was suitable for positive – ions, while addition of 10 mM ammonium acetate (instead of 0.1% formic acid) was effective for - ve analyte ions.

RESULTS & DISSCUSSION

Table (1): Zeranol residues in imported frozen beef and livers and locally manufactured beef luncheon, canned beef and pastirma (dried salted beef) (n = 55).

Kind of analysed tissue samples	Numbers of analysed tissue samples	Numbers and percentages of samples contained zeranol
Imported frozen beef	20	0 (0%)
Imported frozen livers	10	0 (0%)
Locally manufactured beef luncheon	10	0 (0%)
Locally manufactured Canned beef	10	0 (0%)
Locally manufactured pastirma	5	0 (0%)
Total	**55**	**0 (0%)**

Table (2): Antimicrobial residues in chicken breast samples taken from locally- reared birds (n = 20)

Kinds of analysed antimicrobials	Numbers and percentages of breast samples contained antimicrobial
Chloramphenicol	0 (0%)
Florfenicol	7 (35%)
Penicillin	0 (0%)
Sulphaquinoxaline	3 (15%)
Tetracycline	0 (0%)
Tylosin	15 (75%)

n = Number analysed samples.

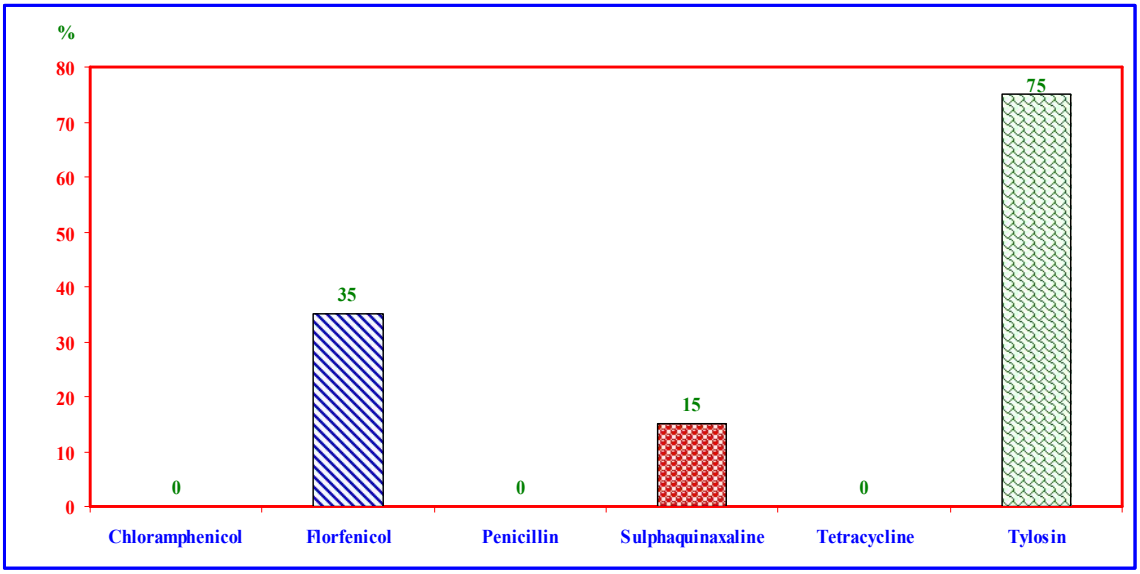

Figure (1): Antimicrobial residues in chicken breast samples taken from locally-reared birds (n = 20).

Table (3): Residue levels (µg/kg) of antimicrobials contained in positive breast samples

Florfenicol	Sulphaquinoxaline	Tylosin
0.032	1.101	0.696
0.152	1.720	0.752
0.190	3.090	0.856
0.303		1.036
0.456		1.076
1.702		1.156
2.363		1.256
		1.494
		1.804
		2.340
		2.944
		2.983
		3.048
		3.954
		8.160

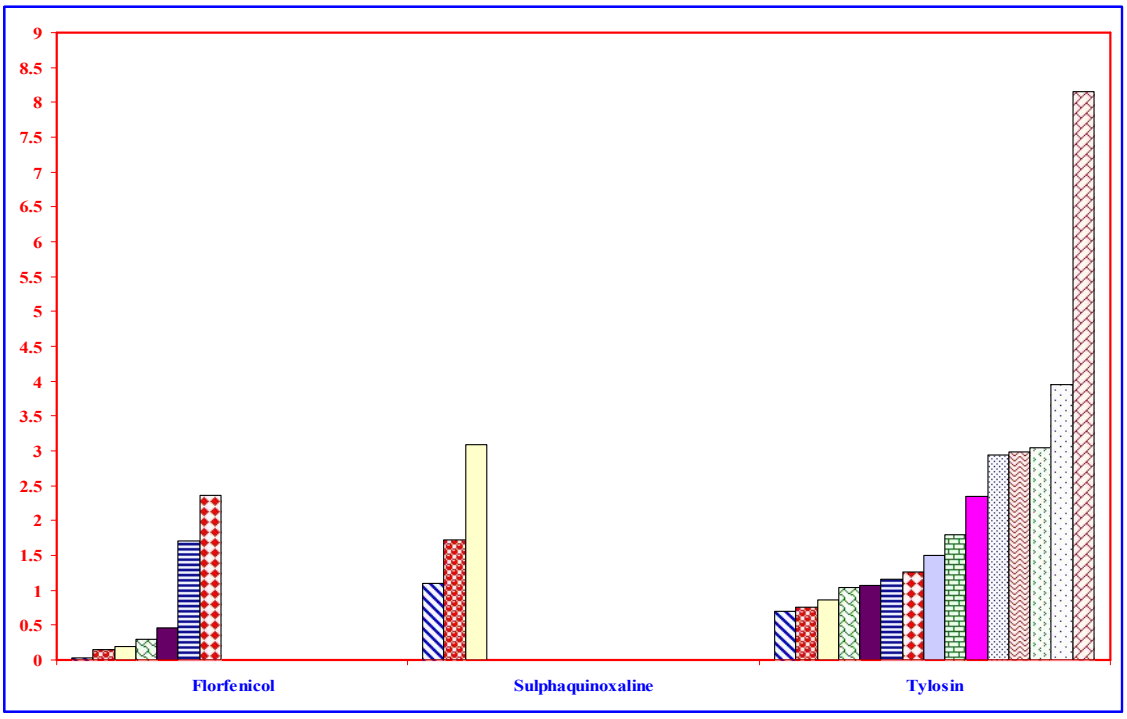

Figure (2): Residue levels (µg/kg) of antimicrobials contained in positive breast samples.

Table (4): Numbers and percentage of positive chicken breast samples contained antimicrobials more then limits recommended by Council Regulation (EEC) (1990)

Types of antimicrobial residues	Maximum residue limits (MRLs)	No. and % of samples contained residues more then MRLs
Florfenicol	100 µg/kg	–
Sulphaquinoxaline	100 µg/kg	–
Tylosin	100 µg/kg	–

Meat and meat products, which play in important role in human nutrition, should be safe and should not contain any factors or substances harmful for human health. However, the anabolic agents used for various purposes in animal husbandry for slaughter tend to leave residue and thus cause some problems in consumer health **(Hoffman, 1996)**.

Inspection of table (1) revealed the absence of zeranol residues in imported frozen beef and cattle livers samples as well as in locally – manufactured beef luncheon, canned beef, and pastirma (dried & cured beef) samples that derived from the imported frozen beef. These findings agreed with the results obtained by **O'keeffe and Hopkins (1987)** who could not detect zeranol residues in 80 Irish beef samples and by **Sadek et al. (1998)** who failed to recognize such residues in beef and cattle livers marketed in Alexandria – Egypt, by using HPLC (High performance Liquid Chromatography). This agreement of the different results may be explained by the sampling time, which determined by the interval from zeranol implanting until slaughtering of the implanted cattle, was > 70 days that reflect a good practice of zeranol use **(Brown, 1980; Scientific Committee on Veterinary Measures Relating to Public Health, 1999** and **Galbraith, 2002)**. On the contrary, **Nazli et al. (2005)** determined zeranol residues in all (100%) tested Turkish meat samples (30 fresh meat and 30 meat products samples) marketed in Istanbul by using ELISA (Enzyme Linked Immuno – Sorbent Assay); out of them 51 (85%) samples contained ≤ 2 µg/kg and the remaining 9 (15%) samples had zeranol levels > 2 µg/kg (> MRL).

It is apparent that the subject of hormone growth promoters in meat is complex with deficiencies in knowledge of biology and chemistry, differences in interpretation of conventional toxicological evaluation, and concerns for human health and implications for trade.

Recently there has been an increasing international and local awareness of the danger of consuming food products with drug resides. Many of them are now classified as carcinogenic, toxic and allergenic **(Mahgoub et al. 2006)**. Growing concern among consumers and public health authorities the presence of antibiotic residues in animal production **(Popelka et al. 2005)**. The presence of antibiotic residues, which are used on a large scale in poultry farming, in chicken muscle and liver samples has been demonstrated by some previously published data **(De Wasch et al., 1998; Tajick and Shohreh, 2006; Miranda et al., 2009 and Tajik et al., 2010).**

Analysis of the residual level of some antimicrobials, commonly used in Egyptian chicken farms, performed by the aid of a validated liquid chromatography coupled with a tandem mass spectrometry (LC–MS/Ms) technique showed that neither chloramphenicol, penicillin, nor tetracycline residues could be detected in tested chicken breast samples. Whereas, the residues of florfenicol were recognized in 7(35%), of sulphaquinoxaline in 3 (15%), and of tylosin in 15 (75%) of such samples (Table 2 and Figure 1). The lowest quantities of the determined antimicrobials residues were 0.032, 1.101, and 0.696 micrograms per each kilogram of chicken breast samples, meanwhile, the highest levels for the same antimicrobials (florfenicol – sulphaquinoxaline – tylosin) were 2.363, 3.090, and 8.160 μg/kg, respectively (Table 3 and Figure 2). By comparison, none of the estimated drug residue levels was exceeded the maximum residue limit (MRL) that recommended by **Council Regulation (EEC) (1990)** as 100 μg/kg (100 ppb) for each drug residue (Table 4).

As regards the chloramphenicol residues, the results obtained in the present work coincided with the MRL for such drug that recommended as zero by several regulatory agencies. On the contrary, **Mehdizadeh et al. (2010)** found these residues in 54.8% out of 31 chicken muscle samples by using ELISA, in addition to the HPLC quantification of the same residues in Nigerian chicken breast samples as a range of 89.33 - 223.05 μg/kg by **Adweuyi et al. (2011)**.

Florfenicol is structural analogue of thiamphenicol, possessing a wide spectrum of activity against both Gram – negative and Gram – positive bacteria **(Syriopoulou et al., 1981)**. Florfenicol was reported to have a greater activity than chloramphenicol and especially against *Pasteurella*, *Salmonella*, *E. coli* and *Staphylococcus aureus*. Florfenicol inhibits peptidyltransferase activity and affect microbial protein synthesis **(Canon et al., 1990)**. The P-nitro group of chloramphenicol is responsible for serious bone marrow toxicity and does –

independent irreversible aplastic anaemia, partially described in humans, but not in animals. For this reasons, the use of chloramphenicol in meat – producing animals had been banned in the USA, the European Union and several other countries **(El-Banna and El–Zorba, 2011)**.

Concerning the penicillin residues in chicken breast samples, **Karmi (2014)** was in agreement with our finding, as he could not detect these residues by the aid of four – plate test (FPT) in such samples.

Sulphaquinoxaline (SQ) residues in chicken muscles could also be detected by higher incidence and quantities by higher incidence and quantities than that obtained in the present work; where **Davitiyanada et al. (1996)** and **Ya-Min et al. (2001)** estimated SQ residues in chicken muscles and livers by levels 580 – 1230 μg/kg; **Elgazzar and El- Lawendy (2005)** found these residues in 100% of 50 chicken breast samples in Egypt, by using liquid chromatography (LC) with levels 170 – 4160 μg/kg (all tested samples had SQ residues > MRL); also **Afify (2010)** determined such residues in 4 (26.67%) out of 15 chicken muscle samples in Egypt, by using HPLC with a mean level of 430 μg/kg, 2 (13.33%) of surveyed samples contained SQ residues quantities > MRL (100 μg/kg). Furthermore, **Mehtabuddin et al. (2012)** recognized sulphonamides residues in 13 (43%) out of 30 chicken breast samples in Pakistan by using HPLC; 7 (23%) of these samples contained such residues by levels > MRL (100 μg/kg), while the remaining 6 (20%) samples possessed < 100 μg/kg; also **Karmi (2014)** detected sulphonamides residues in 44 - 64% of chicken breast samples in Egypt, by using four – plate test (FPT). However, **Salem (1998)** could not determine SQ residues in chicken muscles and livers.

A lot of workers surveyed chicken muscles for their contents of tetracycline(s) residues; among them **De Wasch et al. (1998)** could not found tetracycline residues in 1768 chicken breast samples, by using LC- MS/MS (Liquid Chromatography – Mass Spectrometry / Mass Spectrometry) – in agreement with those findings obtained in our study; also **Cetinkaya et al. (2012)** results were nearly similar to our data, as they found tetracycline residues only in 1 (1.67%) out of 60 chicken muscles samples in turkey with a level 17.2 μg/kg (< MRL = 100 μg/kg) by using LC- MS/MS technique. On the other hand, higher incidence and levels of tetracycline(s) residues were determined in chicken muscles **;** by **Iqabal (2000)** who found tetracycline residues in all (100%) chicken muscles samples in Pakistan, by using HPLC, with level 21.32 - 81.3 μg/g **;** by **Shahid et al. (2007)** who recognized oxytetracycline residues in 11.76% chicken muscles samples, by the aid of HPLC, with a mean level 51 μg/kg **;** by **Afify**

(2010) who determined tetracycline (TC) residues exclusively in 2 (13.33%) out of 15 chicken muscles samples in Egypt, by using HPLC, with a mean level 52.5 μg/kg, none of these samples harboured TC levels > MRL (100 μg/kg) **;** by **Adewuyi et al. (2011)** who estimated oxytetracycline residues in chicken breast samples in Nigeria with a range 670 - 1816 μg/kg more than MRL (100 μg/kg) **;** in addition to the work of **Karmi (2014)** who found tetracyclines (TCs) residues in 48 - 56% of chicken breast samples in Egypt, by using four – plate test (FPT).

Regarding the stability of TC residues in chicken muscles during their cooking, **Al–Ghamdi et al. (2000)** emphasized that boiling water / 20 min could only decrease TC residues, however, **Afify (2010)** assured that boiling water caused complete elimination of such residues (100% reduction) in fully – cooked chicken.

Between 1995 and 1999, **Rose et al. (1999)** demonstrated that residues of a range of veterinary drugs have varying degrees of stability during cooking and therefore, the cooking influences the level of risk posed by such residues. However, the cooking process can not annihilate total amount of these drugs and it can only decrease their levels. Among various agents affecting antibiotics residues after cooking process, cooking time and temperature can play major role about antibiotics residues decreasing. Hence, use of cooking processes that have higher temperature and longer time can lead to the most decrease in antibiotic residues in meat and it can provide an additional margin of safety for consumers, but the effects of metabolites antibiotics residues that can be produced during cooking must toxicologically be studied through future researches **(Javadi et al., 2009)**.

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الملخص العربى

تحليل الكروماتوغرافيا السائلة – قياس الطيف الكتلى/ قياس الطيف الكتلى (الترادفى) لمتبقيات الزيرانول فى اللحم البقرى وأكباد الماشية ومتبقيات بعض مضادات الميكروبات فى لحوم الدواجن

أ.د/ محمد محمد محمد إبراهيم الجزار، *أ. د/ جيهان محمد المغازى ، **ط.ب/ تهانى أحمد أمين العبد
قسم الرقابة الصحية على الأغذية كلية الطب البيطرى جامعة المنصورة.
* المركز الاقليمى للاغذية والاعلاف مركز البحوث الزراعية بالجيزة.
** مديرية الطب البيطرى بدمياط.

الاهتمام بوجود المتبقيات الكيميائية في أنسجة حيوانات الذبيح والدواجن الصالحة للاستهلاك الآدمي – أصبحت الآن هي صيحة العصر في مجال البحث العلمي لاسيما الخاص بتخصص صحة اللحوم. إن وجود تلك المتبقيات لا يعلن عن نفسه حيث لا يغير من الصفات الحسية الطبيعية لتلك الأنسجة لنجد الطعم الطيب واللون الزاهي والقوام المتماسك للحم يحمل بين طياته متبقيات كيميائية لكنه يمثل سمية وخطر داهم علي صحة المستهلكين علي المدي الطويل، لذلك فإن المتبقيات الكيميائية في اللحوم ومنتجاتها تمثل تحديا منقطع النظير للمراقبين الصحيين علي اللحوم ومنتجاتها لأنه من المستحيل التحقق من وجودها بالفحص الظاهري فقط إنما يستلزم تقديرها نوعا وكما تقنيات وأجهزة حديثة من خلال استقصاءات دورية وفحص عينات عشوائية للحوم والدواجن ومنتجاتها. علي الجانب الآخر فإن تعاطي الأدوية البيطرية في مجالات الإنتاج الحيواني والداجني - من أجل مكافحة الأمراض والوقاية منها وكذلك تحفيز النمو- أصبح أمرا حتميا، لذلك أجريت تلك الدراسة علي عدد إجمالي خمس وسبعون عينة عشوائية من أنسجة لحم البقر والدواجن- وزعت كالتالي: عشرون عينة من لحم البقر المجمد المستورد وعشر عينات من أكباد الماشية المجمدة المستوردة (مثلت دولا أجنبيه عديدة مصدرة للحوم) بالإضافة إلي منتجات اللحم البقري المصنعة محليا والتي شملت عشر عينات من كل من لانشون اللحم البقري واللحم البقري المعلب وخمس عينات من البسطرمة (لحم بقري مجفف ومملح) (مثلت مصانع لحوم مصرية عدة) اشتريت من محلات بيع اللحوم والأكباد المجمدة المستوردة وكذلك من محلات البقالة –علاوة علي عشرين عينة من لحم صدور عشرين من مذبوحات الدواجن المرباة محليا وذلك بعد ذبحها وتجهيزها بمحلات الدواجن المصرية المختلفة. تم استكشاف متبقيات الزيرانول (محفز للنمو ويحاكي هرمون الاستروجين في تأثيره ويتم إزدراعه تحت الجلد عند قاعدة الأذن للماشية) في عينات لحم البقر ومنتجاته المختلفة، بينما كان استكشاف بعض مضادات الميكروبات- شائعة الاستعمال البيطري –مقصورا علي عينات لحم صدور الدواجن.

أسفر التقدير النوعي والكمي لجميع المتبقيات المستكشفة في أنسجة عينات لحم البقر والدواجن المختبرة باستعمال التقنية الفعالة والشرعية لكروماتوغرافيا السائلة المقترنة بقياس الطيف الكتلي الترادفي عن غياب متبقيات الزيرانول في عينات لحم البقر ومنتجاته وكذلك في عينات الأكباد. وقد تمخضت نتائج التحاليل أيضا عن غياب مضادات الميكروبات الثلاثة الآتية: كلورامفينكول –بنسلين –تتراسيكلين في عينات لحم صدور الدواجن المختبرة، في حين أمكن التحقق من وجود ثلاث أخر من مضادات الميكروبات في تلك العينات هي فلورفينكول في سبع (٣٥٪) من العينات بمقادير تراوحت بين ٠,٠٣٢ – ٢,٣٦٣ ميكروجرامات/ كيلو جرام (جزء لكل بليون)، سلفاكينوكسالين في ثلاث (١٥٪) من العينات بكميات ١,١٠١ –٣,٠٩٠ ميكروجرامات/ كيلو جرام وتايلوزين في خمس عشرة (٧٥٪) من العينات بمستويات انحسرت بين ٠,٦٩٦- ٨,١٦٠ ميكروجرامات/ كيلو جرام. عند مقارنة مقادير مضادات الميكروبات الثلاثة بالحدود القصوى المسموح بوجودها في اللحوم (١٠٠ ميكروجرام/ كجم لكل نوع من هؤلاء الثلاثة) نجد أنه لا توجد عينة من لحم صدور الدواجن المختبرة احتوت علي مستو أعلي من الحد المسموح به.