PN-ABT-186
91232

FINAL REPORT
PROJECT GRANT AGREEMENT

BETWEEN

REPUBLIC OF TUNISIA
AND
UNITED STATES OF AMERICA

FOR

A SURVEY OF FOOD CONTAMINATION RESEARCH AND CONTROL IN TUNISIA

BY

DR.BECHIR KEKHIA, BIOCHEMIST
PRINICIPAL INVESTIGATOR

AND

CHEKIB ALGUEMI, ASSISTANT

NATIONAL INSTITUTE OF NUTRITION

DR.Z.KALLAL
DIRECTOR

TUNIS/TUNISIA

USAID PROJECT N° 936-5543
DATE: SEPTEMBER 30,1981

DR.C.F.JELINEK. PROJECT OFFICER
US.FOOD AND DRUG ADMINISTRATION
WASHINGTON

BEST AVAILABLE DOCUMENT

ACKNOWLEDGMENT

The Pricipal Investigator wishes to acknowledge the contributions made by Professor Z.KALLAL, Director of the National Institute of Nutrition (INNTA), in the initiation and continued support of the project; by Mr.John LUPIEN, Director of the International affairs Staff, U.S Food And Drug Administration (USFDA), and Miss.Dale GIBB, U.S Agency of International Development, in proposing and obtaining approval for the project, and by DR.Charles JELINEK, Center for Food Safety And Applied Nutrition, USFDA, in providing direction and guidance for this project as Project Officer.

It is also important to acknowledge the valuable contributions made to this project by its Staff of dedicated and competent Investigators:

- Mr.Chékib ALGUEMI, MSc, Assistant	Laboratory adviser/supervisor
- Miss.Neila HAMDOUN, Pharmacist	Gas Chromatography
- Miss.Aida KSOURI, Technician	Extraction/Clean up
- Miss.Afaf DHRAIEF, Technician	Extraction/Clean up
- Mr.Abderrazak ZAGHDOUDI, Technician	Gas Chromatography/Extraction
- Mr.Moungi BOUHANI, Technician	Thin Layer Chromatography
- Mrs.Lilia ZAGHDOUDI,	Glasware cleaning

The interest they have shown, the skill they have employed and the suggestion they made have been responsiLle in large part for the progress made in this project.

Acknowldgement is also given to Mr.Dennis MANSKE, Kansas City District, USFDA for the help in planning this project; Mr.Frank McCULLOUGH, Kansas City District, USFDA for his valuable advice and training of the project Staff; Drs.Gerald MOY and Merton SMITH, International Affairs Staff, USFDA who administered the project and assisted in the procurement of equipment and supplies from the United States, and to Dr.James VERMILLON, U.S-Agency for International Development for facilitating the continued progress of the project.

BEST AVAILABLE DOCUMENT

I. INTRODUCTION

Under USAID Project N° 936-5543 " SURVEY OF FOOD CONTAMINATION RESEARCH AND CONTROL IN TUNISIA", the Tunisian National Institute of Nutrition and Food Technology (INNTA), assisted by the U.S Food and Drug Administration (USFDA) acting under a participating agency service agreement signed February 18, 1982, undertook a program with the following objectives:

a) To detrmine the frequency and levels of pesticide residues and heavy metal content of foods of importance in the Tunisian diet.

b) To develop local skills infield inspection and surveillance, sampling, laboratory analysis, and evaluation of results to establish baseline data.

c) To develop programs to control food contamination problems identified by the Survey.

Following visits to INNTA in March-April, 1983 by a pesticide residue Chemist and Project Officer from the USFDA, the project budget was increased from $ 100,000 to $ 115,000 and completion date was extended to June 30, 1985 because significant delays in obtaining equipment and supplies had occured. Analysis of heavy metals in foods was also deleted from the current project.

After the subsequent visit by USFDA representatives in April-May, 1984, it was agreed that the primary objective of the project would be to analyze 120 food samples selected on the basis of their importance in the Tunisian diet by the general method for residues of organochlorine and organophosphorus pesticides, and in addition, 60 of the non-fatty foods should be analyzed by the Storherr method for residues of organophosphate pesticides. It was also agreed that a maximum of 60 samples of non fatty foods should be analyzed for Carbamate residues, depending on the use pattern. Subsequently, the analysis of carabamate residues was dropped because of delays in arrival of the necessary laboratory equipment.

BEST AVAILABLE DOCUMENT

II. RENOVATIONS AND IMPROVEMENT LABORATORY FACILITIES

Numerous alterations of one area in the National Institute of Nutrition were carried out to convert it into laboratory space and to improve efficiency and safety of laboratory operations. A description of these alterations follows:

The large room was devided by cabinets with lamintaed tops.The division was completed by installing glass panes above the counter top.The resulting north room is 8½ ft x 28 ft. and contains 48 linear feet of counter space plus two sinks. One sink is stainless steel and was installed as part of the renovation. An 8 ft.wide fume hood was installed to improve safety of operations where large amounts of flammable solvents are used. Sample preparation, extraction and clean up are conducted in this area.

The area south of the room divider is 14 ft. x 28 ft. This area contains the explosion-proof centrifuge, rotary evaporation equipment with associated circulating cooling bath, TLC equipment, refrigerator and storage.

Air conditioning was installed with ducts into both areas, sonce hazardous volatile solvents would be used.

The smaller room to the east contains balances, desks and storage.

An additional room, approximately 14½ ft. x 37 ft., formerly used as a library, was converted to an instrument laboratory. This room adjoins the other room to the south. The room contains the HPLC, two GLC's, a refrigerator and storage shelves and cabinets. This room is air conditioned with a window unit.

Each room required some electrical wiring. Laboratory benches, cabinets and shelving were supplied.

In addition, a room on the first floor, approxiamtely 8 ft.x 34 ft. was allocated . It was anticipated that it would be used for samples preparation. It contains an oven for florisil, TLC plates applicator, refrigerator and storage.

BEST AVAILABLE DOCUMENT

The central part of the building, originally was open. Rain water leaked into the room of the first floor, severelly limiting its usefulness. A roof was installed over the entire central part of the building, correcting this problem and creating more usable space.

Separating the instruments from the extraction and clean up laboratory has reduced the danger of cross contamination and improved safety and laboratory efficiency. The fume hood, air conditioning and room divider have improved safety by reducing fire hazards and personnel exposure to vapors.

BEST AVAILABLE DOCUMENT

III. APPARATUS AND REAGENTS

The following equipment was used to prepare food samples:

- EXTRACT PREPARATION EQUIPMENT:
  - Two grinders (IKA-WERK, RFA, model M-20 with 250 ml capacity, and Micro-Culatti).
  - One homogenizer, 250 ml capacity (IKA-WERK, RFA, Ultra-Turrax).
  - One chopper, 25-125 ml capacity (Buhler, RFA).
  - One explosion proof centrifuge (DAMON-ICE, USA) for fatty food extracts.
  - One rotary evaporator equiped with vacuum controller(BUCHI, Switzerland) and refrigerated water bath (LAUDA, RFA).
  - Kuderna Danish concentrators (SUPELCO, Switzerland).

- CLEANUP EQUIPMENT:
  - A Florisil chromatographic column (10 mm i.d x 300 mm) with teflon stopcock and coarse porosity fritted disc (SUPELCO, Switzerland) was used.
  - A Charcoal chromatographic column (25 mm i.d x 400 mm) with vacuum adaptor connetcted to a vacuum pump was used for Storherr method.
  - The gel permeation chromatography was done with a long column, (25 mm i.d x 300 mm), (KONTES, USA) with organic adaptors packed with Biobeads SX3 (BIO-RAD, USA) connected with pump (MILTON-ROY, USA) for organic solvents.

- GAS CHROMATOGRAPHY EQUIPMENT:
  - Two gas chromatographs (TRACOR 565, USA) were used. One was equiped with a nickel 63 electron capture detecteor (ECD) connected with 6 ft.x 4 mm i.d glass column, packed with OV-225 3% on Gas-Chrom Q 100/120 mesh, and with a flame photometric detector (FPD) in the phosphorus mode connected with 6 ft.x 4 mm i.d glasse column, packed with OV-1 5% on supelcoport 80/100 mesh.

BEST AVAILABLE DOCUMENT

The ECD carier gas was argon|methane 95|5 (60 ml|min). The FPD carrier gas was nitrogen (80 ml|min).
The temperature parameters were: 200 °C columns, 215 °C injectors, 200 °C FPD detector and 350 °C ECD detector.
The second gas chromatograph was equipped with a nickel 63 ECD connected with capillary column OV-101 or SE-54 (0,250 mm i.d X 25 m)or with 4 mm i.d X 6 ft. glass column packed with OV-1 5% on supelcoport 80|100 mesh, and with a FPD connected with 4 mm i.d X 6 ft. glass column packed with OV-17 3% on supelcoport 80|100 mesh.
The temperature and the gas flow conditions were the same as the first gas chromatograph, except for the capilary column which was used with the splitless or isothermal mod
The sensitivity of all detectors was set to have 70% full scale deflection with injection 1 ng of chlorpyrifos in hexane.

- THIN LAYER CHROMATOGRAPHY EQUIPMENT:

The standard CAMAG|SWITZERLAND equipment was used for the confirmation tests. Plates were prepared with CAMAG automatic coater. Samples were spotted with Nanomat or Linomat III and detected with UV-Camag Cabinet.

- REAGENTS:

All reagents were of analytical grade. Solvents were from MERCK and were of pesticide grade.

BEST AVAILABLE DOCUMENT

IV. PROCEDURES

The methods applied were those described in the Pesticide Analytical Manuel (PAM) with some modifications (see below).

GENEREAL METHOD FOR NON FATTY FOODS (PAM 212):

After the acetonitrile extraction of the food samples, a portion (20%) of the total filtrated extract was taken for the STORHERR method. A second portion (30%) was taken for the determination of both organophosphorus and organochlorines by the general method (1).
The Florisil column cleanup procedure was done according to the W.L.SAXTON method (2) which uses 10 mm i.d X 300 mm glass column packed with 4 g of Florisil in petrolium ether. Three fractions 35 ml 6%, 35 ml 15%, and 40 ml 50% ethyl ether in petrolium ether were eluted from the column and concentrated just to dryness. The final volume of each fraction was adjusted to 2 ml before gas chromatography injection.
The STORHERR procedure was done according to PAM 232.3. We used 6 g of Celite 245 AW|MgO|Charcoal mixture instead of 9 g as described by L.J.CARSON. One fraction (180 ml benzene|acentonitrile 1|1) was eluted from the column and concentrated just to dryness. The final volume was adjusted to 2 ml with hexane before gas chromatography injection.

GENEREAL METHOD FOR FATTY FOODS (PAM 211):

After extraction of the food sample with petrolium ether, a portion corresponding to 1 g of fat was purified on a gel permeation column using a Biobeads X 3 gel according to the M.L.HOPPER method (4).
One fraction (80 ml hexane|methylene chloride 1|1) was eluted from the column and concentrated to about 5 ml before the Florisil cleanup.
The eluted fractions from the Florisil cleanup were concentrated just to dryness and the final volume adjusted to 5 ml with hexane.

BEST AVAILABLE DOCUMENT

BEST AVAILABLE DOCUMENT

GAS CHROMATOGRAPHY IDENTIFICATION:

Each fraction from Florisil or Charcoal columns was injected on OV-225 (ECD) and on OV-1 5% (FPD) GC columns. Fraction not showing peaks was descared and considered as not contaminated. Fractions showing peaks were re-injected on OV-1 5% (ECD), OV-101 (capillary|ECD), SE-54 or OV-17 3% (FPD).
Relative retention times to chlorpyrifos on each column were compared to the PAM tables. A peak was considered as identified when the RRT|C ratio was in accordance with PAM values.

CONFORMITY TESTS|

The P-value test was used according to PAM 621 in hexane|acetonitile system.
TLC for organochlorines according to PAM 412, method II was also used as a confirming test.
Sandwich injection test developed in our laboratory wasalso used: this test constists of injecting tha analyzed fraction which probably contained pesticide with the known corresponding standard in the same syringe. If the chromatogram shows no separation between peaks, the probable peak in addition to the other tests cab be identified more easily.

CALUCULATION:

All calculations were made by using the HP 3390 integrator with the following formula:

$$\frac{\text{Peak height(fraction)| ul}}{\text{Peak height(standard)| ul}} \text{ X (Standard conc.)Xfract.Vol}$$

Results were corrected according to PAM formula for each group of food and expressed as ppm. For fatty foods, results were expressed as ppm f

FOOD SAMPLES ANALYSIS:

Each analyzed food sample set was run with a blank and spiked sample for the recovery estimation.

V. METHODS CONTROL

The pesticide residue analytical methods described in the PAM for the determination of organochlorine and organophosphorus pesticide residues in foods were controlled in the INNTA Laboratories prior to the initial survey. This control consisted of a study of the recovery of two standard pesticide mixtures added to different food samples.

The first mixture contained diazinon (0,03 ppm), malathion (0,05 ppm), lindane (0,01 ppm), diledrin (0,01 ppm), and DDT-PP' (0,03 ppm).

The second mixture contained the same pesticide standards except that each one was three times more concentrated.

During each recovery study, an unspiked or blank sample was utilized as a control.

DESCRIPTION OF RESULTS FOR RECOVERY STUDIES:

The results of the recovery studies using the general procedure (PAM I, 212) for non-fatty foods spiked at the lower concentrations of pesticide are presented in Table II. Average percent recoveries for all non fatty foods used and for all pesticides added ranged from 71,7% to 81,5%. The recovery rates for organochlorine pesticides averaged slightly higher (76,1%) than those obtained for the organophosphorus pesticides (73,6%). The coefficients of variation (CV) ranged from 35,3 to 44,9 for recoveries of all pesticides at the low spiking levels. The average CV for organochlorine pesticides was 37,1 and for organophosphorus pesticides was 44,3.

BEST AVAILABLE DOCUMENT

The results of the recovery studies for the non fatty foods which were spiked at the higher concentration level of pesticide (3 times higher) are shown in Table III. The range of recoveries under these conditions was 61,9% to 79,3% for organochlorine and organophosphorus compounds respectively. The average coefficient of variation calculated for the organochlorines (17,1%) was considerably lower than that of the organophosphorus compounds (45,9%). The average recoveries for organochlorine pesticides spiked at both high and low levels were greater than those for organophosphorus compounds.

The recovery studies using the general procedure (PAM I, 211) for fatty foods which were spiked with the lower concentrations of pesticides are shown in Table IV. As can be seen, the average recovery for organochlorine compounds (80,6%) was higher than for tha organophosphorus compounds (60,8%). The CV values did not differ significantly except that the value for diazinon recoveries was extremely high (70,6%). In this concentration erratic results are to be expected with diazinon in the analytical methodology used.

Table V presents the results of the three fatty foods recoveries spiked at the higher concentrations . Recovery rates were poor for all pesticides studied except malathion which showed an 83,2% recovery. The average recovery for organochlorines was 42,2% and the average for the organophosphorus pesticides was 61,0%. It should be kept in mind that the data in this table represents only a few analyses (1 to 3) for the different pesticides.

Table VI presents the recovery results for the lower spiked levels of pesticides using the Storherr method (PAM I, 232.3). The range of average recoveries was 80,0% to 86,1% with an average recoveries of 83,0% for organophosphorus pesticides using this method.

The recoveries for the foods spiked at the higher concentrations are shown in Table VII. The range of average recoveries was 57,8% to 68,3% with an overall average for these organophosphorus pesticides of 63,0%.

BEST AVAILABLE DOCUMENT

Acceptable recoveries and coefficients of variation at low residue levels are are addressed by P.A.GREVE in The World Health Organization publication (5). He presents a compilation of results from three authors, which shows that recoveries should be within the range of 70% to 110% with a mean greater than 80% after removal of outliers. The level of spiking is not mentioned.

He lists acceptable coefficients of variation for different residue levels as :

Residu level	Coefficients of variation (%)	
	Repeatability	Reproductibility
0,01	50	100
0,10	25	50
1,00	12,5	25

The recoveries and CV's obtained are in the range expected when three factors are considered:

1) The range of spiking is 0,01 to 0,15 mg|kg

2) No values were removed as outliers

3) Using the GPC column clean-up with the general method for fatty foods (PAM 211), often results in low recoveries of diazinon, which may partially elute with the lipid fraction.

BEST AVAILABLE DOCUMENT

VI.SURVEY RESULTS

The analysis of (125) Tunisian food samples from different locations in this initial survey showed the contamination of two cheese samples with HCB (level 80,0 and 67,4 ppb). But we faound (12) samples prbably contamintaed with organochlorines or organophosphorus. In both cases, it was not possible to confirm whether these compounds were pesticides or not.

In the food samples submitted by other Government Agencies, we found a 36% contamination rate. The foodscontaminated were barley, wheat and black pepper by malathion, DDT-PP' and HCB. In all samples contamination level was less than 1 ppm.

BEST AVAILABLE DOCUMENT

VII. DISCUSSION OF RESULTS

The basic objectives of this initial study were to establish a laboratory capability in INNTA for the analysis of organochlorine and organophosphorus pesticide residues in food products and to determine the level of occurrence of these pesticides in Tunisian foods obtained from the market place.

As described in the results section, the initial survey which was conducted indicates possible common occurrence of pesticides in cheese, barley, wheat and black pepper. However, because the numberof samples obtained to date is small, the survey is not large or representative enough in order to make any valid conclusions regarding the occurrence of pesticides in food products consumed in Tunisia. For this reason it is important for INNTA to continue the survey that has been initiated in order to obtain more samples . Additionally, it is important to modify the survey procedures in the following ways in order to obtain more valid conclusions:

. obtain more samples from foods especially those of major importance in the Tunisian diet; for exemple milk and wheat.
. obtain samples from all the major food-producing areas of Tunisia.
. obtain food samples close to the location of production.
. obtain samples of major food imports.
. obtain samples which are large enough to be representative of the food lot which was sampled (It is recommended that in most cases at least a 5 kg sample be taken from the lot).

It is important that the sample site of origin and other such information be occurately recorded in order to facilitate any followup actions which may be necessary.

BEST AVAILABLE DOCUMENT

The INNTA laboratory has not beed able to analyze any samples for carbamate residues or to perform any recovery studies on carbamate pesticides because it has experienced considerable delays in the procurement of some of the equipment needed for analysis. It was also necessary to return some of the parts of the fluorescence detection which were not functioning properly.

On the other side, because there was a need to confirm the results of the analyses for suspected organochlorine residues by TLC significant time was required. If a chloride specific microcoulometric detector was available, more efficient and more positive confirmations could be obtained with greater sensitivity could be obtained for organochlorine residues.

Because only one EC detector was available for the organochlorin analyses, extraction and cleanup were carried outmore rapidly than the GLC analyses could be carried out. For this reason, the availability of another gas chromatograph with EC detector is necessary to followup analyses.

BEST AVAILABLE DOCUMENT

VIII. CONCLUSIONS

From the investigations and accomplishments under this project, the following conclusions can be drawn:

a) Significant improvements were made by INNTA in its physical facilities in order to carry out pesticide analyses of foods efficiently and accurately.

b) The pesticide laboratory of INNTA has the capability to carry out sensitive and acurate analyses for organochlorine and organophosphorus pesticides in all type of foods.

c) The output of laboratory could be increased significantly by the addition of selected instruments to enable more direct and efficient analyses and confirmation of findings of organochlorine pesticides.

d) The survey for these pesticides in foods which was carried out identified some commodities in which contamination appears to occur commonly, but it was not large or representative enough to establish baseline data on the frequency and levels of pesticide residues in foods of importance in the Tunisian diet.

e) There is a need to carry out the collection of food samples as close to the location of production as feasible and to obtain larger samples which are representative of a food lot.

f) The development of the capability to analyze for carbamate pesticides was seriously compromised by delays in equipment prourement, and the need to return some of the original items purchased.

BEST AVAILABLE DOCUMENT

IX. RECOMMENDATIONS

The following recommendations are made for the followup to this project:

a) Additional samples of foods of importance should be obtained for analysis from all sections in Tunisia, as close to the site of production as feasible. These samples should be large enough to be representative of the food lot concerned. These additional steps should be carried out in order that valid conclusions can be made regarding the incidence of pesticides in the Tunisian food supply.

b) Large pieces of food preparation equipment should be obtained for preparation of representative samples of food for laboratory analysis.

c) A gas chromatograph with chloride selective microcoulometric detector and a gas chromatograph with an electron capture detector should be purchased. This would greatly increase laboratory efficiency by allowing rapid,positive identification of organochlorine residues.

d) The capability to analyze foods for residues of carbamate insecticides should be established. Samples of appropriate foods should then be analyzed for these pesticides.

e) Samples for additional pesticide analytical standards and of replacements were needed should be obtained, through the assistance of FDA if posible, to aid the measurment and confirmation of pesticide residues.

BEST AVAILABLE DOCUMENT

X. REFERENCES

(1) Pesticide Analytical Manual Volume I.
U.S Department of Health and Human Services - Food And Drug Administration - WASHINGTON.

(2) W.L,SAXTON - Partial Miniaturization of the M.O.G Pesticide Screening Method - Laboratory Information Bulletin, DFST/EDRO, Dec.30, 1981.

(3) L.J,CARSON - Modified Storherr Procedure for the Analysis of Organophosphorus pesticides - Laboratory Information Bulletin, Dec. 17, 1979.

(4) M.L,HOPPER - Automated Gel Permeation System For Rapid Separation of Industrial Chemicals Organophosphate and Chlorinated Pesticides From Fats - Laboratory Information Bulletin, March 1, 1982.

(5) P.A,GREVE - Good Laboratory Practice In Pesticide Residue Analysis - Health Aspects Of Chemical Safety - Interim Document 14 - Pesticide Residue Analysis - W.H.O/Copenhagen, Page 281, 1984.

BEST AVAILABLE DOCUMENT

Table II: Recovery of pesticides at low level in non fatty foods

RECOVERY STUDIES

NON FATTY FOODS, GENERAL METHOD (PAM I, 212)

FOOD PRODUCT	DIAZINON (0.03 PPM)	MALATHION (0.05 PPM)	LINDANE (0.01 PPM)	DIELDRIN (0.01 PPM)	DDT-PP' (0.03 PPM)	FILE No
BARLEY	48,6		78,2	66,0	54,1	74
	80,3	58,2	89,3	82,5	67,6	73
BLACKPEPPER			60,4	50,4	78,7	35
			49,2	45,1	100,8	36
	83,8		74,3	72,5	85,4	38
CARROTS			44,4		50.0	14
CELERI			21,7		32,8	12
	68,9	45,5	80,5	48,1	55,8	112
FENNEL	35,6	71,4	70,6	48,3	53,0	76
HARISSA	113,0	130,0	137,0	112,0	93,0	82
MELON	119,5	36,8	131,0	133,5	134,0	39
	26,3		60,5	77,5	113,6	32
	29,6	69,8	69,1	92,9	92,5	41
	81,0	20,9	101,7	100,5	93,1	24
ORANGE	80,9	64,6	78,8	70,6	65,9	108
PARSLEY	150,0	133,1	104,9	95,4	62,8	78
POTATOES			78,3	42,8	95,9	11
		99,3		25,4	30,0	20
	114,3	97,9	95,6	83,6	64,4	22
	54,4	69,0	46,9	57,5	64,6	81
SPAGHETTI	11,6	40,4	46,6	44,5	40,3	52
	67,2	83,5	62,8	62,1	55,5	51
	68,6	96,8	88,6	74,9	71,3	62
	73,5	133,2	82,8	44,3	67,8	63
TANGERINE	40,7	39,2	55,4	51,0	78,3	65
	64,9	93,0	95,3	57,4	89,8	68

BEST AVAILABLE DOCUMENT

RECOVERY STUDIES

NON FATTY FOODS, GENERAL METHOD (PAM I, 212) - (CONTINUED)

FOOD PRODUCT	DIAZINON (0.03 PPM)	MALATHION (0.05 PPM)	LINDANE (0.01 PPM)	DIELDRIN (0.01 PPM)	DDT-PP' (0.03 PPM)	FILE No
TOMATOES	101,4	63,7	112,5	114,8	101,3	21
	117,2	21,4	132,2	112,2	109,2	42
	34,3	25,9	28,0	69,0	23,8	54
	56,3	58,6	124,3	66,3	62,9	19
	80,2	68,9	98,8	79,0	58,3	56
	88,0	63,6	100,2	88,2	50,5	53
WATER MELON	42,4		52,7	57,5	45,3	30
	97,7	139,3	87,1	155,6	80,8	40
WHEAT			19,1		101,9	16
		90,1	88,6	74,6	69,6	31
WHOLE EGG	108,7	105,6	122,1	101,2	79,1	67
	62,7	80,4	56,3	31,0	18,9	115
YOGURT	22,4	93,7	79,5	74,6	70,2	71
	72,4	49,5	139,5	97,6	96,2	77
	74,6	99,0	113,5	99,0	79,3	99
$\bar{X}$	71,84	75,5	81,45	75,24	71,66	
S	32,26	33,0	31,22	28,41	25,3	
CV %	44,9	43,7	38,3	37,75	35,3	
n	33	31	40	38	41	

BEST AVAILABLE DOCUMENT

Table III: Recovery of pesticides at higher level in non fatty foods

RECOVERY STUDIES

NON FATTY FOODS, GENERAL METHOD (PAM I, 212)

FOOD PRODUCT	DIAZINON (0.09 PPM)	MALATHION (0.15 PPM)	LINDANE (0.03 PPM)	DIELDRIN (0.03 PPM)	DDT-PP' (0.09 PPM)	FILE No
COURGETTE	22,2	56,6	62,5	53,6	61,6	121
LETTUCE	69,8	64,0	84,2	65,8	82,4	123
	88,1	132,0	58,8	77,1	89,3	124
SALAD	61,9	116,0	67,0	51,9	79,9	128
SPINACH	93,8	38,7	81,2	55,3	66,4	125
TOMATOES	35,7	68,5	86,5	81,7	65,3	118
$\bar{X}$	61,9	79,3	73,6	74,2	74,1	
S	28,4	36,4	12,0	13,0	11,2	
CV	45,8	46,0	16,4	19,9	15,1	
n	6	6	6	6	6	

BEST AVAILABLE DOCUMENT

Table 12 : Recovery of pesticides at low level in fatty foods

RECOVEY STUDIES

FATTY FOODS, GENERAL METHOD (PAM I, 211)

FOOD PRODUCT	DIAZINON (0.03 PPM)	MALATHION (0.05 PPM)	LINDANE (0.01 PPM)	DIELDRIN (0.01 PPM)	DDT-PP' (0.03 PPM)	FILE No
BUTTER	14,3	25,8	72,1	57,2	60,3	75
	95,9	109,5	111,0	105,3	103,8	28
CHESSE			100,2	86,5	80,2	83
			210	77,3	82,4	85
			87,4	51,9	69,9	86
	35,1	55,4	65,7	65,7	60,3	59
	38,7	60,9	113,3	92,3	96,6	64
EGGS (YELLOW)	25,2			61,6	34,4	60
FISH						49
MEAT (BEEF)	58,0	58,5	82,7	78,9	85,8	119
MEAT (COW)	140,0	139,0	139,0	157,0	157,0	47
MILK	11,4	63,6	78,5	83,6	79,3	61
	11,5	21,8	34,6	31,6	32,5	58 BIS
	17,8	64,3	99,4	41,3	73,2	72
	35,6	56,4	68,3	66,6	71,6	58
OLIVES		89,5	163,6	144,4	69,3	66
			27,4	52,1	31,8	70
OLIVES OIL	102,7	71,1	63,4	68,3	64,6	69
	108,6	115,0	121,7	57,9	51,5	27
	35,4	48,5	65,3	82,4	74,4	45
	69,1	73,0	83,5	82,4	74,4	29
	79,2	22,9	45,3	74,2	37,0	25
TUNNY						90
YOGURT	53,3	96,8	88,3	76,3	69,0	106
$\bar{X}$	54,8	69,8	90,9	78,0	72,8	
S	38,7	32,7	41,6	29,4	28,2	
CV	70,6	47,4	45,8	37,6	38,8	
n	18	17	22	23	23	

BEST AVAILABLE DOCUMENT

Table V: Recovery of pesticides at higher level in fatty foods

RECOVERY STUDIES

FATTY FOODS, GENERAL METHOD (PAM I, 211)

FOOD PRODUCT	DIAZINON (0.09 PPM)	MALATHION (0.15 PPM)	LINDANE (0.03 PPM)	DIELDRIN (0.03 PPM)	DDT-PP' (0.09 PPM)	FILE No
CHICKEN	64,4	83,2	67,1	69,4	65,2	120
FISH	13,2		17,3	22,3		126
MERGUEZ			25,6	37,3	28,6	133
$\bar{X}$	38,8	83,2	36,7	43,0	46,9	
S	-	-	-	-	-	
CV	-	-	-	-	-	
n	2	1	3	3	2	

BEST AVAILABLE DOCUMENT

Table VI: Recovery of pesticides at low level (Storherr method).

RECOVERY STUDIES

NON FATTY FOODS, STORHERR METHOD (PAM I, 232.3)

FOOD PRODUCT	DIAZINON (0.03 PPM)	MALATHION (0.05 PPM)	FILE No
CELERI	81,3	100,6	112
FENNEL	100,9	60,6	76
ORANGE	77,0	93,1	108
PARSLEY	62,1	62,2	78
POTATOES	86,2	82,4	81
TANGERINE	74,5	86,7	65
	75,0	98,0	68
WHOLE EGG	75,3	84,5	115
YOGURT	63,1	62,8	71
	87,9	98,7	77
	97,2	117,2	99
$\bar{x}$	80,0	86,1	
s	12,5	18,2	
cv	15,4	21,1	
n	11	11	

BEST AVAILABLE DOCUMENT

Table VII: Recovery of pesticides (Storherr method) at higher level.

RECOVERY STUDIES

NON FATTY FOODS, STORHERR METHOD (PAM I, 232.3)

FOOD PRODUCT	DIAZINON (0.09 PPM)	MALATHION (0.15 PPM)	FILE No
COURGETTE	24,2	22,9	121
LETTUCE	39,5	48,0	123
SALAD(GREEN)	80,5	98,0	128
SPINACH	79,2	96,7	125
TOMATOES	65,6	75,4	118
$\bar{x}$	57,8	68,3	
s	25,0	32,5	
CV	43,3	4[illegible],6	
n	5	5	

BEST AVAILABLE DOCUMENT

Table VIII:

CONTAMINATED FOOD SAMPLES (TUNISIA)

NAME	ORIGIN	RESULTS	FILE
Cheese Camambert	STIL	HCB (80,0 ppb Fat)	85
Cheese double cream	STIL	HCB (67,4 ppb Fat)	87

BEST AVAILABLE DOCUMENT

BEST AVAILABLE DOCUMENT

Table IX:

CONTAMINATED FOOD SAMPLES NON IDENTIFIED ORGANOCHLORINE PESTICIDES (TUNISIA)

NAME	ORIGIN	FILE
Blett	INNTA	139
Celeri	INNTA	112
Fennels	INNTA	76
Fish	INNTA	132
Onion	INNTA	138
Parsley	INNTA	78
Salad green	INNTA	128
Tangerine	INNTA	68
Tunny (Canned)	Super Market	90

BEST AVAILABLE DOCUMENT

Table X:

CONTAMINATED FOOD SAMPLES NON IDENTIFIED
ORGANOPHOSPHORUS PESTICIDES
(TUNISIA)

NAME	ORIGIN	FILE
Fish	INNTA	136
Tomatoes	INNTA	53
Yogurt	STIL	71

BEST AVAILABLE DOCUMENT

Table XI-1:

NON CONTAMINATED FOOD SAMPLES (TUNISIA)

NAME	ORIGIN	FILE
Barley	Hospital(R)	146
Barley Bran	INNTA	74
Blackpepper	OCT/TUNIS	43
Blett	INNTA	6
Butter	INNTA	75
Butter	INNTA	28
Cacao Nuts	Agruculture	109
Carrots	INNTA	117
Carrots	INNTA	141
Carrots	INNTA	114
Carrots	INNTA	80
Carrots	INNTA	8
Carrots	INNTA	14
Celeri	INNTA	12
Cheese (Riki)	INNTA	59
Cheese	INNTA	64
Cheese Roquefort	INNTA	84
Cheese Gruyere	Super Market	86
Cheese Holland	Super Market	83
Chick-Pea	Hospital(R)	144
Chicken	INNTA	120
Cougettes	INNTA	121
Cougettes	INNTA	7
Couscous	INNTA	129
Egg Whole	INNTA	115
Egg Whole	INNTA	67

BEST AVAILABLE DOCUMENT

Table XI-2:

NAME	ORIGIN	FILE
Egg yellow	INNTA	60
Fennels	INNTA	110
Fish	INNTA	126
Fish	INNTA	127
Fish	INNTA	142
Fish	INNTA	49
Fish	INNTA/Market	143
Flour	INNTA	131
Haricot bean	TUNIS	145
Harissa (canned)	PACNA	82
Lettuce	INNTA	123
Letuce	INNTA	124
Lettuce	INNTA	3
Macaroni	INNTA	134
Macaroni	INNTA	135
Meat bovine	INNTA	119
Meat bovine	INNTA	47
Meat bovine	INNTA	55
Meat mutton	INNTA	48
Melon	Borj Toumi	32
Melon	Borj Toumi	39
Melon	Borj Toumi	41
Melon	Borj Toumi	24
Mergueze (Sausages)	Menzah VI	133
Milk	STIL	61
Milk conc.Gloria	INNTA	72
Milk Pasteurized	STIL	58
Milk	STIL	88

BEST AVAILABLE DOCUMENT

Table XI-3:

NAME	ORIGIN	FILE
Olive oil	CAPBON	29
Olive oil	INNTA	69
Olive oil	INNTA	25
Olive oil	INNTA	27
Olives	ARIANA	66
Olives (black)	MENZAH	70
Onion	INNTA	10
Oranges	INNTA	108
Parsley	INNTA	137
Parsley	INNTA	111
Parsley	INNTA	13
Potatoes	INNTA	116
Potatoes	INNTA	113
Potatoes	INNTA	81
Potatoes	INNTA	20
Potatoes	INNTA	22
Potatoes	INNTA	1
Potatoes	INNTA	11
Salad green	INNTA	5
Sardine (canned)	ONP/Sidi Daoud	91
Sardine (canned)	Super Market	97
Sardine (canned)	Super Market	96
Sardine (canned)	Super Market/Chaba	93
Sardine (canned)	Super Market/Alcazar	95
Sardine (canned)	Super Market/Mahdia	92
Sardine (canned)	Super Market/Sidi Daoud	94

BEST AVAILABLE DOCUMENT

Table XI-4:

NAME	ORIGIN	FILE
Spinach	INNTA	122
Spinach	INNTA	125
Tangerine	INNTA	65
Tomatoes	Borj Toumi	42
Tomatoes	INNTA	118
Tomatoes	INNTA	107
Tomatoes	INNTA	19
Tomatoes	INNTA	21
Tomatoes	INNTA	54
Tomatoes	INNTA	56
Tomatoes	INNTA	57
Tomatoes	INNTA	2
Tomatoes	INNTA	9
Tunny (canned)	Super Market	89
Water melon	Borj Toumi	40
Water melon	INNTA	30
Wheat	Borj Toumi	31
Wheat	INNTA	18
Wheat crushed	INNTA	130
Yogurt	STIL	67
Yogurt	BELDI	104
Yogurt	STIL	105
Yogurt	STIL	106
Yogurt	STIL	98
Yogurt	BELDI	101
Yogurt	STIL	102
Yogurt	STIL	103
Yogurt	STIL	99

BEST AVAILABLE DOCUMENT

SAMPLES SUBMITTED BY GOVERNMENT AGENCIES - CONTAMINATED.

FOOD PRODUCTS	ORIGIN	PESTICIDES FOUND	LEVEL (PPM)	FILE No
WHEAT	TUNIS	Malathion	0,40	15
Blackpepper	RADES	DDT-PP'	0,06	26
Blackpepper	TUNIS	DDT-PP'	Trace	23
Blackpepper	TUNIS	DDT-PP'	0.030	17
Blackpepper	TUNIS	DDT-PP'	0,025	35
Blackpepper	TUNIS	DDT-PP'	0,150	33
Blackpepper	TUNIS	DDT-PP'	0,022	38
BARLEY	GROMBALIA	Malathion	0,175	79
BARLEY BRAN	TUNIS	HCB	Trace	73

BEST AVAILABLE DOCUMENT

SAMPLES SUBMITTED BY GOVERNMENT AGENCIES - NON CONTAMINATED.

FOOD PRODUCTS	ORIGIN	FILE
WHEAT	TUNIS	16
WHEAT	SOUSSE	50
CUCUMBER	MONASTIR	149
SPAGHETTI	MSAKEN	51
SPAGHETTI	MSAKEN	52
SPAGHETTI	SIDI-BOUZID	63
SPAGHETTI	SIDI-BOUZID	62
EGGS	MONASTIR	147
BLACKPEPPER	TUNIS	44
BLACKPEPPER	TUNIS	33
INDIAN BLACKPEPPER	PARIS	37
GREEN PEPPER	PARIS	34
YOGURT	MONASTIR	148
OLIVE OIL	MONASTIR	45
OLIVE OIL	SFAX	153
OLIVE OIL	SFAX	156

BEST AVAILABLE DOCUMENT