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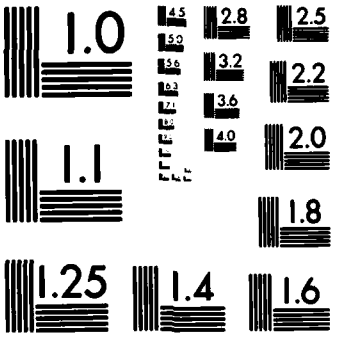

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## Abstract

HMX can be detected in dosing solutions and in diets prepared for toxicity studies by reverse phase HPLC.

Dietary preparations were stable for at least 21 days when stored in ambient conditions in the dark.

Almost all the dosing solutions and diets prepared for a range of studies were shown to contain dose to the desired concentrations of HMX. There was, however, the possibility of an interchange between 2 groups on one occasion on the 13 week rat study.

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IRI Report No. 2832

# HMX: Analysis of Dosing Formulations Used in Acute, Sub-acute and Sub-chronic Toxicity Studies

Final Report by:

M.S. Henderson

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Musselburgh, EH21 7UB, Scotland

Contracting Officer's Technical Representative:

Jesse J. Barkley, Jr.
U.S. Army Medical Bioengineering Research
and Development Laboratory
Fort Detrick, Frederick, Maryland 21701-5010

## FOREWORD

"I, the undersigned, hereby declare that this work was performed under my supervision, according to the procedures herein described and that this report represents a true and accurate record of the results obtained."

A.B. Wilson, B.V.Sc., M.R.C.V.S.,
D.A.B.T.
Principal Investigator

## QUALITY ASSURANCE AUTHENTICATION

The execution of this type of short-term study is not individually inspected. The processes involved are inspected at intervals according to a pre-determined schedule.

This report has been audited by IRI Quality Assurance Personnel according to the appropriate Standard Operating Procedure and is considered to describe the methods and procedures used in the study. The reported results accurately reflect the original data of the study.

IRI Project No. 415669 MD
415669 CR
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416877

Report No. 2832

Signed: Andrew Waddell
(Quality Assurance Manager)

Date: 3rd March 1986.

## CONTENTS

## SUMMARY

An analytical method for HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) in dosing suspensions has been developed. Aliquots of the formulation are dissolved in acetonitrile and diluted until a solution is obtained whose ultraviolet absorbance at 228 nm can be recorded.

Ten suspensions of HMX in distilled water were prepared in order to assess the accuracy of the preparation procedure and the homogeneity of the formulation. In all samples the results were within ± 10% of the target values and were considered satisfactory.

The method was used to analyse the samples prepared during studies to evaluate the acute toxicological effects of HMX. The results are reported and, as they were within ± 10% of the target values, were considered satisfactory.

As part of the study concerning the toxicology of HMX following dietary administration to rats and mice it was necessary to formulate HMX in diet and analyse the formulations in order to assess both the accuracy of preparation and their homogeneity. The stability of the formulated test substance on storage for up to 21 days was also assessed.

The principal findings were as follows:

1. HMX can be readily determined in diets using a procedure based on reverse phase high performance liquid chromatography (HPLC) with spectrophotometric detection. Tetryl (tetranitromethylaniline) is used as internal standard.

2. The accuracy of dietary preparation (defined as % mean found/target concentration) and the homogeneity of the diet (defined as co-efficient of variation at each of the test concentrations) were as indicated below:

| Test Sample Concentration (ppm) | Accuracy (% Recovery) | Co-efficient of Variation n = 10 |
|---|---|---|
| 1,250 | 94.2 | 6.8 |
| 10,000 | 102.0 | 5.4 |
| 25,000 | 99.3 | 2.5 |

3. Re-analysis of the same samples after storage in darkness at ambient temperature for 21 days showed no significant degradation of HMX, i.e. values for re-assay after storage are within 2 $\sigma$ of the initial non-stored values.

4. Re-analysis of the same samples after storage at 40°C for 14 days showed no significant degradation of HMX.

Prior to commencement of the main 90 day subchronic studies, 14 day dose range finding studies were undertaken. As part of the latter, it was necessary to carry out routine chemical analysis of HMX in the formulated diets. Accordingly aliquots were collected and analysed at commencement and on Day 8 of the study programme.

The results of the analyses were considered acceptable since other than Group 3♀ mixed 23 October 1980 the mean values were within ± 10% of the target value.

During the 90 day studies and the 14 day rat study aliquots of diet for consumption by animal dose groups were analysed at commencement and at Weeks 1, 2, 3, 6, 9 and at termination of the study. The results are presented in Tables 28-41.

The results are generally (>90%) acceptable since the mean values were within 10% of the nominal values.

Two exceptions to this were found during the analysis of the formulations for the rat study and it is suggested on one occasion (15 December 1980) the Group 5♀ diet may have been sampled twice and on the other (29 December 1980) the diets had been interchanged although it is not possible to state whether this occurred during the formulation or the sampling of the diet.

## INTRODUCTION

The U.S. Army have undertaken a programme of toxicity testing of HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) following both dietary and oral (by gavage) administration to rats and mice. In addition, one study was concerned with the establishment of percutaneous toxicity of HMX when administered to rabbits. As an essential part of these studies it was necessary to optimise the procedure for the preparation of HMX in suspension in liquid vehicles. There was also a requirement to analyse the suspensions prior to and during the dosing phases of the study in order to assess both the homogeneity and the accuracy of the preparation of doses.

As an essential part of the dietary studies it was necessary to optimise the procedures for the preparation of the diet, to analyse the dietary mixes and to assess the stability of HMX in dietary formulations after storage.

The analytical experiments described in this report were performed at the Inveresk Gate laboratories of Inveresk Research International Limited, Musselburgh, Scotland between July 1980 and April 1981.

The formulation and sampling of the diets and the preparation of the dosing suspensions were carried out in the dispensary at the Elphinstone Research Centre.

All data generated and recorded during this study will be stored in the Scientific Archives of Inveresk Research International Limited.

## GENERAL EXPERIMENTAL PROCEDURES

### Materials

HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) and RDX (hexahydro-1,3,5,-trinitro-1,3,5-triazine) were supplied from the Royal Ordnance Factory, Bridgewater, England as white suspensions containing approximately 20% by weight of water. The dry materials were obtained by drying to constant weight in a water heated oven.

Tetryl (trinitrophenylmethylnitramine) for use as internal standard was supplied by the Ministry of Defence, Waltham Abbey, England from wartime stock.

DNB (1,3-dinitrobenzene) (organic analytical standard grade) was supplied by BDH Limited, Poole, Dorset, England.

SEX (octahydro-1-acetyl-3,5,7-trinitro-1,3,5,7-tetrazocine) and TAX (hexahydro-1-acetyl-3,5-dinitro-1,3,5-triazocine) were supplied by the U.S. Army (Holston Defence Corporation).

Acetonitrile (of HPLC or "S" grade) was purchased from Rathburn Chemicals Limited, Walkerburn, Scotland.

### Moisture Content of Supplied HMX

Approximately 1 g of the supplied HMX was weighed accurately into a glass scintillation vial and placed in a water-jacketted oven at 90°C for several hours. The vial was removed, placed in a desiccator for 30 min to cool and then reweighed. This process was continued until the vial reached constant weight. The experiment was performed in triplicate and the mean value used. It was found that samples could be dried to constant weight at 90°C in 3½-4 h.

Where the dry material was used for the preparation of formulations it was assumed to be fully dry when no further loss in weight was detected. No further calculation of water content was made for these samples.

## EXPERIMENTAL PROCEDURES

### SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies

#### Measurement of the Extinction Co-efficient for HMX

Five solutions of HMX in acetonitrile were prepared and their ultraviolet spectra recorded (see Figure 1 for typical spectrum).

The data are presented below.

| Initial Concentration mg.100 $ml^{-1}$ | Concentration After Dilution mg.100 $ml^{-1}$ | $\Delta A$ | $\varepsilon$ | Mean Value |
|---|---|---|---|---|
| 30.4 | 1.52 | 1.07 | 20837 | |
| 29.1 | 1.455 | 1.07 | 21768 | 21346 |
| 24.7 | 1.235 | 0.88 | 21091 | $\pm$ 454 |
| 21.7 | 1.085 | 0.795 | 21688 | |

Calculation:

$$\varepsilon = \frac{\Delta A \times MW \times 10^{2}}{C}$$

where $\varepsilon$ = Extinction co-efficient for HMX

$\Delta A$ = Absorbance of solution at 228 nm

C = Concentration of measured solution (mg.100 $ml^{-1}$)

MW = Molecular weight

#### Final Analytical Methodology for the Analysis of HMX in Suspensions

The supplied formulation was mixed thoroughly on a Vortex mixer and a 1 ml aliquot of the suspension transferred to a 100 ml volumetric flask. This was made to the mark with acetonitrile (HPLC grade). For complete dissolution of the HMX it was necessary to shake thoroughly for at least 1 min or keep in an ultrasonic bath. A suitable aliquot (V.ml) was removed and transferred to a 100 ml or 250 ml volumetric flask and made to the mark with acetonitrile (S grade). The ultraviolet spectrum was recorded using as reference a solution prepared as above from 1 ml of 0.5% low-viscosity carboxymethylcellulose (CMC).

The results are tabulated (Tables 8-10).

Calculation:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times d \times 296 \times 10^4}{W \times V \times \varepsilon}$$

where $\Delta A$ = Absorbance at 228 nm

$d$ = Density of suspension

$W$ = Weight of aliquot

$V$ = Volume removed for second dilution

$\varepsilon$ = Extinction co-efficient for HMX (21346)

For 1% high-viscosity CMC:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times 172.5}{W \times V}$$

For physiological saline:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times 156.1}{W \times V}$$

It was also necessary to calculate the density of HMX as a suspension in the 2 vehicles. Aliquots (1 ml) of a 60% w/w suspension were removed and transferred to preweighed 1 ml volumetric flasks. The flasks were reweighed and the density calculated. The results are tabulated (Table 11).

The sampling schedules relating test sampling to animal dosing are shown below (Tables 1-2).

SECTION 2: Analysis of HMX in Dietary Formulations Analytical Method Development

Preliminary HPLC of HMX and Related Explosives

Solvent 1: Methanol:Water (30:70 v/v)

Solvent 2: Acetonitrile:Water (20:30 v/v)

System A

Column: 100 x 5 mm stainless steel packed with Hypersil ODS

Solvent: 1

Flow: 1 $ml.min^{-1}$

Wavelength: 228 nm

Detector: Pye LCUV or LC3UV

Range: 0.32 AUFS

Pump: Altex 110A

Recorder: 10 mV

Chart Speed: 600 $mm.h^{-1}$

The chromatograms obtained are shown in Figure 2.

System B

Column: 100 x 5 mm stainless steel packed with Hypersil ODS

Solvent: 2

The chromatograms obtained are shown in Figures 3-6.

System C

Column: 250 x 5 mm stainless steel packed with LiChrosorb RP 2

Solvent: 2

Flow: 2 $ml.min^{-1}$

System D

Column: 250 x 5 mm stainless steel packed with LiChrosorb RP 8

Solvent: 2

Flow: 2 $ml.min^{-1}$

Using conditions described under System A it was possible to separate the compounds shown (Figure 2) but it was not always possible to obtain single peaks due to solvent effects caused by the poor solubility of HMX in aqueous methanol solutions. For this reason it was decided that the preliminary HPLC experiments would be carried out using conditions described under System B. As seen from Figure 5 this system does not separate HMX from RDX or SEX from TAX, but does permit separation of HMX from tetryl which was used as internal standard (Figure 6).

During the course of the study tetryl became unavailable and 1,3-dinitrobenzene was used as internal standard (Figure 7).

## Preliminary Analysis of HMX in Diet

### (a) Experimental Laboratory Preparation of Diet

To 100 g of BP diet (for typical analysis see Appendix 1) was added a solution of HMX (50 mg) in acetonitrile (10 ml). After addition of 200 ml of acetonitrile the mixture was shaken mechanically for 5 min and then evaporated on a Buchi rotary evaporator for 5 h at 50°C. This gave a diet containing 500 ppm HMX.

### (b) Method of Analysis

3 x 10 g samples of the diet were weighed into clear 8 oz glass jars. To each sample was added 1.5 ml of internal standard solution (38.3 mg tetryl in 10 ml acetonitrile) and acetonitrile (50 ml) as extracting solvent. The bottles were shaken mechanically for 1 h and the contents allowed to settle. An aliquot of the supernatant (10 µl) was taken for analysis by HPLC using conditions described under System B. The peak height ratios were compared with those from extracts of diet to which known amounts of HMX and tetryl were added. The mean value obtained was 520 ± 12 ppm (relative error 4%). Comparison of peak heights obtained from extracted HMX and from known concentrations of HMX indicated that complete extraction had taken place.

## Purity of HMX

Using the HPLC conditions described under System A in which HMX can be distinguished from RDX it was calculated that the supplied material contained a maximum of 0.4% RDX (Figure 8).

## Final Analytical Procedure for the Determination of HMX in Diets

Approximately 5 g of the supplied diet was weighed accurately into an 8 oz clear glass jar. To the sample was added 1 ml of internal standard solution (tetryl or 1,3-dinitrobenzene in acetonitrile) and 50 ml of acetonitrile as extracting solvent. The mixture was shaken mechanically for 1 h, and the contents left to settle, with centrifugation (15 min, 3,000 r.p.m.) if necessary. An appropriate aliquot (5-50 µl) was taken for analysis by HPLC.

Standard solutions of HMX were prepared by adding a known amount of HMX (equivalent to that of the groups being analysed) to a sample of untreated diet. To these were added internal standard solution and extracting solvent as described above. The experimental details and the results obtained from the analyses are presented in Tables 12-14.

It should be noted that the HMX used for the preparation of the calibration standards and quality control samples was from the same dried batch as that used for the preparation of the bulk diet.

Quality control samples were prepared by an independent operator at a level unknown to the analyst and included in each batch of samples processed. Standards and samples were extracted and analysed concurrently.

HPLC Conditions

Column: 100 x 5 mm stainless steel packed with Hypersil ODS

Solvent: Acetonitrile:Water (2:3 v/v)

Flow Rate: 1 $ml.min^{-1}$

Wavelength: 228 nm

Equipment: Hewlett Packard 1084B liquid chromatograph with a variable wavelength ultraviolet detector and an autosampler. This instrument integrates the output from the detector and calculates peak areas. When calibrated and appropriately programmed it calculates the concentration of HMX in a sample from the peak area ratios and the sample weight. The information is printed out after each chromatogram.

This method was used for the analysis of HMX in all samples of diet prepared for animal consumption during the toxicology studies.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice

Preparation of Dietary Formulations

The formulations were prepared by mixing the correct amount of dry test material with the animal feedstuff in plastic drums on a Winkworth Change Drum Tumble Mixer for 20 min. Aliquots for analysis (3 x 10 g) were removed from three places through the mix.

## Analytical Procedure for the Determination of HMX in Diets

The procedure used for the analysis of diets prepared for animal consumption during the toxicology studies was that described on page 9, Final Analytical Procedure for the Determination of HMX in Diets.

## Sampling Schedules

Aliquots of diet (3 x 10 g) for consumption by animal dose groups 1-5♂ and 1-5♀ were analysed at commencement and on Day 8 of the 14 day study.

For the 90 day studies aliquots of diet for animal dose groups 1-6♂ and 1-6♀ were analysed at commencement and at Weeks 1,2,3,6,9 and termination of the study.

## RESULTS AND DISCUSSION

SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies.

### Method Development

Ten suspensions of HMX in distilled water were supplied for analysis in order to assess the accuracy of the preparation procedures and the homogeneity of the suspensions. Each sample was analysed in duplicate and the results are shown in Table 3. In all samples the results were within ± 10% of the target values and were considered satisfactory.

### Analysis of HMX in Suspensions for the Range Finding and Main Acute Toxicology Studies in Rats and Mice

The formulations for the range finding studies were prepared in 0.5% low viscosity carboxymethylcellulose (CMC). Aliquots for analysis were removed at intervals during the dosing of each group of animals. Tables 4-7 show the results obtained. In all samples the results were within ± 10% of the target values and were considered satisfactory.

### Analysis of HMX in Suspensions During Acute Dermal Toxicity Testing in Rabbits

Samples were prepared in 1% high-viscosity CMC and physiological saline. The analysis were performed in duplicate and the results are shown in Tables 8-10.

SECTION 2: Analysis of HMX in Dietary Formulations. Analytical Method Development.

### Optimisation of Dietary Preparation Procedures

Bulk samples of HMX were supplied wetted with approximately 20% water. Since it was not considered practicable to use the wetted material in diet preparations, the material was dried to constant weight prior to formulation. The weighed quantity of test material was then mixed in plastic drums on a Winkworth Change Drum Tumble Mixer for 20 min.

Three stock diets containing HMX at concentrations of 1,250, 10,000 and 25,000 ppm were prepared and analysed to assess both the accuracy of the preparation procedure and the homogeneity of the mix. Ten samples from each batch were analysed using the method described above and the results are shown in Tables 12-14. Since the values found were within ± 10% of the theoretical value it was considered that the method of preparation was satisfactory.

### Stability of HMX in Diets

Since it was necessary to provide evidence for the stability of the test compound for the lifetime of the formulation prior to commencement of the feeding trials the following stability tests were undertaken. Samples of

formulated diets were subjected to accelerated ageing by storage in an environmental cabinet for 14 days at 40°C. Further samples were stored at room temperature in darkness for 21 days.

Analysis of the stored samples gave the results shown in Tables 15-20. Table 21 summarises the results obtained and compares them with those from freshly formulated diet. All are well within the values of + 2 $\sigma$ of the initial non-stored values and were regarded as acceptable. There was no evidence that significant degradation had occurred during the 21 days test period.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice.

14 Day Studies

The results obtained are presented in Tables 22-23 (the rat study) and Tables 25-26 (the mouse study). The results were considered satisfactory since all but one gave a mean value which fell within + 10% of the target value. The exception (Group 3♀ in the 14 day rat study mixed 23 October 1980) was also considered acceptable (mean value 10.6%).

90 Day Studies

The results are tabulated (Tables 28-41). The concentrations quoted for the standard and quality control samples are expressed in mg while those of the formulated diet extracts are in ppm.

The formulated diets were generally considered acceptable since the mean values fell within + 2 $\sigma$ of the nominal concentration ($\sigma$ being arbitrarily assigned as 5%). Where the nominal concentration was less than 50 ppm the results are considered acceptable where the mean value fell within the range of + 5 ppm of the nominal value.

Several exceptions to the above were found, the most notable being the diets formulated on 15 December 1980 and 29 December 1980 for the 13 week rat study. On the former occasion the Group 6♀ diet on analysis gave a result of 4882 ppm rather than 12577 ppm. This value (4882 ppm) is not significantly different from that of 5019 ppm obtained from the Group 5♀ sample (nominal concentration 5151 ppm). The dispensary weighing records are consistent with the preparation of a 12577 ppm diet and it is suggested that the Group 5♀ diet may have been sampled twice. On the latter occasion, the dispensary weighing records are consistent with the preparation of the required diets at 467 ppm and 1062 ppm. The analytical results bear this out except that the analytical sample of diet labelled 467 ppm gave a result of 896 ppm while that labelled 1062 ppm diet gave 548 ppm, suggesting that the samples have been interchanged. It is not possible to state whether this happened during the formulation or the sampling stages of the procedures.

There were also several groups (detailed in tables) where the results were outwith the defined limits of acceptability which when re-analysed gave results which were considered satisfactory.

## CONCLUSION

SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies.

By using a simple analysis based on ultraviolet spectroscopy it was possible to analyse HMX when formulated as a suspension in distilled water, carboxymethylcellulose or physiological saline.

The test suspensions analysed were, in all but 2 samples, found to be acceptable since the mean values were within $\pm$ 2 $\sigma$ of the target value where $\sigma$ is arbitrarily assigned as 5%. The 2 samples which were outwith this 10% value were 10.1 and 10.5% and were also regarded as acceptable.

SECTION 2: Analysis of HMX in Dietary Formulations. Analytical Method Development.

HMX can be readily determined in diets using a procedure based on reverse phase high performance liquid chromatography with spectrophotometric detection. The accuracy of dietary preparation was considered acceptable since analysis of 3 different batches gave mean values which were within $\pm$ 10% of the theoretical value.

HMX formulated in diet is not degraded on storage either at 40°C for 14 days or at ambient for 21 days.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice.

The test diets analysed were generally (>90%) found to be acceptable since the mean values were within $\pm$ 2 $\sigma$ where $\sigma$ is arbitrarily assigned as 5% (IRI SOP/ACH/106). Where the nominal value is less than 50 ppm the results are acceptable if the mean value is within $\pm$ 5 ppm.

Two exceptions to this were found during the analysis of the formulations for the rat study and it is suggested that on one occasion (15 December 1980) the Group 5♀ diet may have been sampled twice and on the other (29 December 1980) the diets had been interchanged although it is not possible to state whether this occurred during the formulation or the sampling of the diet (see Tables 28 and 30).

TABLE 1

Analysis of HMX in Dosing Suspensions During Percutaneous Toxicity Testing in Rabbits

Sampling Schedule: 1

Vehicle: Physiological saline

Sample 1 taken

2 rabbits dosed (1 $ml.kg^{-1}$)

Sample 2 taken

2 rabbits dosed (3 $ml.kg^{-1}$)

Sample 3 taken

2 rabbits dosed (5 $ml.kg^{-1}$)

Sample 4 taken

Vehicle: 1% High viscosity CMC

Sample 5 taken

2 rabbits dosed (0.5 $ml.kg^{-1}$)

Sample 6 taken

2 rabbits dosed (1 $ml.kg^{-1}$)

Sample 7 taken

2 rabbits dosed (3 $ml.kg^{-1}$)

Sample 8 taken

2 rabbits dosed (5 $ml.kg^{-1}$)

TABLE 2

Analysis of HMX in Dosing Suspensions During Percutaneous Toxicity Testing in Rabbits

Sampling Schedule: 2 (Non-abraded groups)

Sample 1 taken

Group 14, 200-203♂ dosed

Sample 2 taken

Group 14, 204-207♀ dosed

Sample 3 taken

Group 16, 216-219♂ dosed

Sample 4 taken

Group 16, 220-223♀ dosed

Sample 5 taken

Group 18, 232-235♂ dosed

Sample 6 taken

Group 18, 236-239♀ dosed

Sample 7 taken

Group 20, 248-251♂ dosed

Sample 8 taken

Group 20, 252-255♀ dosed

Sample 9 taken

TABLE 2 (continued)

Sampling Schedule: 3 (Abraded groups)

Sample 10 taken

Group 15, 208-211♂ dosed

Sample 11 taken

Group 15, 212-215♀ dosed

Sample 12 taken

Group 17, 224-227♂ dosed

Sample 13 taken

Group 17, 228-231♀ dosed

Sample 14 taken

Group 19, 240-243♂ dosed

Sample 15 taken

Group 19, 244-247♀ dosed

Sample 16 taken

Group 21, 256-259♂ dosed

Sample 17 taken

Group 21, 260-263♀ dosed

Sample 18 taken

TABLE 3

Analysis of HMX In Dosing Suspensions In Distilled Water (Method Development)

| Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Deviation from Nominal (Dry) ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 50.6 | 40.5 | 1 | A | 0.93 | 3.0 | 43.0 | 43.9 | 3.4 | 8.4 |
| | | | B | 0.97 | 3.0 | 44.8 | | | |
| 100.6 | 80.5 | 2 | A | 0.78 | 1.5 | 72.1 | 76.8 | 3.7 | 4.6 |
| | | | B | 0.88 | 1.5 | 81.4 | | | |
| 199.9 | 159.9 | 3 | A | 1.255 | 1.0 | 174.0 | 170.9 | 11.0 | 6.9 |
| | | | B | 1.21 | 1.0 | 167.8 | | | |
| 302.4 | 241.9 | 4 | A | 0.97 | 0.5 | 269.0 | 255.2 | 13.3 | 5.5 |
| | | | B | 0.87 | 0.5 | 241.3 | | | |
| 699.6 | 559.7 | 5 | A | 0.85 | 0.5 | 589.3 | † 617.1 | 57.4 | 10.2 |
| | | | B | 0.93 | 0.5 | 644.8 | | | |

Date of Formulation: 2 July 1980
Date of Analysis: 3 July 1980

† = Second dilution = 250 ml

TABLE 3 (continued)

| Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Deviation from Nominal (Dry) ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 49.8 | 39.8 | 6 | A | 0.84 | 3.0 | 38.8 | 38.4 | 1.4 | 3.5 |
| | | | B | 0.82 | 3.0 | 37.9 | | | |
| 100.0 | 80.0 | 7 | A | 0.93 | 1.5 | 86.0 | 85.6 | 5.6 | 7.0 |
| | | | B | 0.92 | 1.5 | 85.1 | | | |
| 250.0 | 200.0 | 8 | A | 1.53 | 1.0 | 212.2 | 213.6 | 13.6 | 6.8 |
| | | | B | 1.55 | 1.0 | 214.9 | | | |
| 500.0 | 400.0 | 9 | A | 1.57 | 0.5 | 435.4 | 434.1 | 34.1 | 8.5 |
| | | | B | 1.56 | 0.5 | 432.7 | | | |
| 750.0 | 600.0 | 10 | A | 0.94 | 0.5 | 651.7 | 651.7 | 51.7 | 8.6 |
| | | | B | 0.94 | 0.5 | 651.7 | | | |

Date of Formulation: 4 July 1980
Date of Analysis: 7 July 1980

TABLE 4

Analysis of HMX In Dosing Suspensions Formulated In Carboxymethylcellulose During the Range Finding Study In Rats

| Group | Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Deviation from Nominal ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 15.00 | 12.00 | 101 | 0.85 | 10.0 | 11.79 | 12.16 | 0.16 | 1.3 |
| | | | 102 | 0.94 | 10.0 | 13.03 | | | |
| | | | 103 | 0.84 | 10.0 | 11.65 | | | |
| 2 | 35.00 | 28.00 | 104 | 0.82 | 4.0 | 28.43 | 29.45 | 1.45 | 5.2 |
| | | | 105 | 1.06 | 5.0 | 29.40 | | | |
| | | | 106 | 1.10 | 5.0 | 30.51 | | | |
| 3 | 75.00 | 60.00 | 107 | 0.79 | 2.0 | 54.77 | 61.47 | 1.47 | 2.5 |
| | | | 108 | 0.90 | 2.0 | 62.40 | | | |
| | | | 109 | 0.97 | 2.0 | 67.25 | | | |
| 4 | 250.00 | 200.00 | 110 | 1.48 | 1.0 | 205.23 | 201.53 | 1.53 | 0.8 |
| | | | 111 | 1.36 | 1.0 | 188.59 | | | |
| | | | 112 | 1.52 | 1.0 | 210.78 | | | |
| 5 | 750.00 | 600.00 | 113 | 0.77 | 0.5 | 533.87 | 561.61 | 38.39 | 6.4 |
| | | | 114 | 0.82 | 0.5 | 568.54 | | | |
| | | | 115 | 0.84 | 0.5 | 582.41 | | | |

Date of Formulation: 9 July 1980
Date of Analysis: 11 July 1980

TABLE 5

Analysis of HMX in Dosing Suspensions Formulated in Carboxymethylcellulose During the Range Finding Study in Mice

| Group | Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Deviation from Nominal ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 15.00 | 12.00 | 116 | 0.40 | 4.5 | 12.33 | 13.25 | 1.25 | 10.4 |
| | | | 117 | 0.88 | 9.0 | 13.56 | | | |
| | | | 118 | 1.00 | 10.0 | 13.87 | | | |
| 2 | 35.00 | 28.00 | 119 | 1.09 | 5.0 | 30.23 | 29.98 | 1.98 | 7.1 |
| | | | 120 | 0.97 | 4.5 | 29.89 | | | |
| | | | 121 | 0.86 | 4.0 | 29.81 | | | |
| 3 | 75.00 | 60.00 | 122 | 0.94 | 2.0 | 65.17 | 61.24 | 1.24 | 2.1 |
| | | | 123 | 0.81 | 2.0 | 56.16 | | | |
| | | | 124 | 0.90 | 2.0 | 62.40 | | | |
| 4 | 250.00 | 200.00 | 125 | 1.50 | 1.0 | 208.00 | 205.23 | 5.23 | 2.6 |
| | | | 126 | 1.59 | 1.0 | 220.49 | | | |
| | | | 127 | 1.35 | 1.0 | 187.20 | | | |
| 5 | 750.00 | 600.00 | 128 | 0.78 | 0.5 | 540.81 | 584.72 | 15.28 | 2.5 |
| | | | 129 | 0.93 | 0.5 | 644.81 | | | |
| | | | 130 | 0.82 | 0.5 | 568.54 | | | |

Date of Formulation: 9 July 1980
Date of Analysis: 14 July 1980

TABLE 6

Analysis of HMX In Dosing Suspensions Formulated In Carboxymethylcellulose During Acute Toxicology Studies In Rats

| Group | Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Group Mean ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 150.00 | 121.05 | 1 | 1.10 | 1.2 | 127.11 | 127.25 | 127.83 | 5.6 |
| | | | 2 | 1.05 | 1.1 | 132.37 | | | |
| | | | 3 | 0.97 | 1.1 | 122.28 | | | |
| | | | 4 | 1.13 | 1.2 | 130.58 | 134.43 | | |
| | | | 5 | 1.19 | 1.2 | 137.51 | | | |
| | | | 6 | 1.17 | 1.2 | 135.20 | | | |
| | | | 7 | 1.11 | 1.2 | 128.27 | 121.80 | | |
| | | | 8 | 0.93 | 1.0 | 128.96 | | | |
| | | | 9 | 0.78 | 1.0 | 108.16 | | | |
| 2 | 225.00 | 181.58 | 10 | 0.94 | 0.7 | 186.21 | 183.57 | 184.20 | 1.4 |
| | | | 11 | 0.99 | 0.7 | 196.21 | | | |
| | | | 12 | 0.85 | 0.7 | 168.39 | | | |
| | | | 13 | 0.51 | 0.4 | 176.80 | 182.58 | | |
| | | | 14 | 0.49 | 0.4 | 169.87 | | | |
| | | | 15 | 0.58 | 0.4 | 201.07 | | | |
| | | | 16 | 0.98 | 0.8 | 169.78 | 186.44 | | |
| | | | 17 | 1.48 | 1.0 | 205.23 | | | |
| | | | 18 | 0.93 | 0.7 | 184.23 | | | |
| 3 | 337.50 | 272.36 | 19 | 0.91 | 0.5 | 252.38 | 262.66 | 272.48 | 0 |
| | | | 20 | 0.94 | 0.4 | 244.40 | | | |
| | | | 21 | 1.05 | 0.5 | 292.21 | | | |
| | | | 22 | 1.32 | 0.7 | 261.49 | 281.30 | | |
| | | | 23 | 1.06 | 0.5 | 293.98 | | | |
| | | | 24 | 1.04 | 0.5 | 288.43 | | | |
| | | | 25 | 1.13 | 0.6 | 261.16 | 273.49 | | |
| | | | 26 | 1.60 | 0.75 | 295.83 | | | |
| | | | 27 | 0.76 | 0.4 | 263.47 | | | |

Date of Formulation: 22 July 1980
Date of Completion of Analysis: 25 July 1980
HMX contained 19.3% water
Second Dilution = 100 ml

TABLE 6 (continued)

| Group | Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Group Mean ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| | | | 28 | 0.845 | 0.3 | 390.59 | | | |
| | | | 29 | 0.855 | 0.3 | 395.21 | 385.00 | | |
| | | | 30 | 1.065 | 0.4 | 369.21 | | | |
| | | | 31 | 1.455 | 0.5 | 403.53 | | | |
| 4 | 506.25 | 408.24 | 32 | 1.455 | 0.5 | 403.53 | 402.61 | 396.75 | 2.9 |
| | | | 33 | 1.445 | 0.5 | 400.76 | | | |
| | | | 34 | 0.955 | 0.3 | 441.73 | | | |
| | | | 35 | 1.015 | 0.4 | 351.88 | 402.64 | | |
| | | | 36 | 1.495 | 0.5 | 414.62 | | | |
| | | | 37 | 0.96 | 0.2 | 665.62 | | | |
| | | | 38 | 0.92 | 0.2 | 637.88 | 660.99 | | |
| | | | 39 | 0.98 | 0.2 | 679.48 | | | |
| | | | 40 | 0.87 | 0.2 | 603.21 | | | |
| 5 | 759.38 | 612.82 | 41 | 0.97 | 0.2 | 672.55 | 637.88 | 639.42 | 4.3 |
| | | | 42 | NO SAMPLE | | | | | |
| | | | 43 | 1.12 | 0.25 | 621.24 | | | |
| | | | 44 | 1.18 | 0.25 | 654.52 | 619.39 | | |
| | | | 45 | 0.84 | 0.2 | 582.41 | | | |

TABLE 7

Analysis of HMX in Gavage Suspensions Formulated in Carboxymethylcellulose During Acute Toxicology Studies in Mice

| Group | Nominal Concentration (Wet) ($mg \cdot ml^{-1}$) | Nominal Concentration (Dry) ($mg \cdot ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg \cdot ml^{-1}$) | Mean Concentration ($mg \cdot ml^{-1}$) | Group Mean ($mg \cdot ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 60.00 | 48.00 | 46 | 0.93 | 2.5 | 51.59 | 46.32 | 45.90 | 4.4 |
| | | | 47 | 0.66 | 2.0 | 45.76 | | | |
| | | | 48 | 0.45 | 1.5 | 41.60 | | | |
| | | | 49 | 0.89 | 2.5 | 49.37 | 44.85 | | |
| | | | 50 | 0.77 | 2.4 | 44.49 | | | |
| | | | 51 | 0.44 | 1.5 | 40.68 | | | |
| | | | 52 | 0.75 | 2.0 | 52.00 | 46.53 | | |
| | | | 53 | 0.49 | 1.5 | 45.30 | | | |
| | | | 54 | 0.61 | 2.0 | 42.29 | | | |
| 2 | 102.00 | 81.6 | 55 | 0.82 | 1.5 | 75.81 | 73.34 | 79.15 | 3.0 |
| | | | 56 | 0.78 | 1.5 | 72.11 | | | |
| | | | 57 | 1.04 | 2.0 | 72.11 | | | |
| | | | 58 | 0.56 | 1.0 | 77.66 | 80.74 | | |
| | | | 59 | 0.97 | 1.5 | 89.67 | | | |
| | | | 60 | 0.81 | 1.5 | 74.88 | | | |
| | | | 61 | 1.25 | 2.0 | 86.76 | 83.36 | | |
| | | | 62 | 0.73 | 1.2 | 84.36 | | | |
| | | | 63 | 0.57 | 1.0 | 79.04 | | | |
| 3 | 173.4 | 138.72 | 64 | 0.90 | 1.0 | 124.80 | 124.46 | 126.88 | 8.5 |
| | | | 65 | 0.83 | 1.0 | 115.10 | | | |
| | | | 66 | 0.77 | 0.8 | 133.47 | | | |
| | | | 67 | 1.22 | 1.3 | 130.14 | 134.44 | | |
| | | | 68 | 1.04 | 1.0 | 144.22 | | | |
| | | | 69 | 0.93 | 1.0 | 128.96 | | | |
| | | | 70 | 0.82 | 0.9 | 126.34 | 121.73 | | |
| | | | 71 | 0.86 | 1.0 | 119.26 | | | |
| | | | 72 | 0.69 | 0.8 | 119.60 | | | |

Date of Formulation: 22 July 1980
Date of Completion of Analysis: 25 July 1980
HMX contained 20% water

TABLE 7 (continued)

| Group | Nominal Concentration (Wet) ($mg.ml^{-1}$) | Nominal Concentration (Dry) ($mg.ml^{-1}$) | Sample No. | ΔA | V (ml) | Concentration Found ($mg.ml^{-1}$) | Mean Concentration ($mg.ml^{-1}$) | Group Mean ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| 4 | 294.78 | 235.82 | 73 | 1.04 | 0.6 | 240.36 | 244.84 | 240.16 | 1.8 |
| | | | 74 | 0.97 | 0.55 | 244.56 | | | |
| | | | 75 | 0.99 | 0.55 | 249.61 | | | |
| | | | 76 | 0.70 | 0.4 | 242.27 | 239.90 | | |
| | | | 77 | 1.26 | 0.7 | 249.61 | | | |
| | | | 78 | 0.82 | 0.5 | 227.42 | | | |
| | | | 79 | 0.53 | 0.3 | 244.98 | 235.74 | | |
| | | | 80 | 0.51 | 0.3 | 235.74 | | | |
| | | | 81 | 0.49 | 0.3 | 226.49 | | | |
| 5 | 501.13 | 400.90 | 82 | 1.06 | 0.35 | 419.97 | 427.89 | 413.34 | 3.1 |
| | | | 83 | 1.02 | 0.35 | 404.12 | | | |
| | | | 84 | 1.16 | 0.35 | 459.59 | | | |
| | | | 85 | 1.31 | 0.40 | 454.14 | 402.84 | | |
| | | | 86 | 0.56 | 0.20 | 388.28 | | | |
| | | | 87 | 0.66 | 0.25 | 366.09 | | | |
| | | | 88 | NO SAMPLE | | | 409.28 | | |
| | | | 89 | 1.10 | 0.35 | 435.82 | | | |
| | | | 90 | 0.69 | 0.25 | 382.73 | | | |

TABLE 8

Analysis of HMX in Dosing Suspensions Formulated During Percutaneous Toxicity Testing in Rabbits
Nominal Concentration 600 $mg.ml^{-1}$

| Vehicle | Sample No. | | Weight of HMX Suspension (g) | V (ml) | ΔA | Concentration Found ($mg.ml^{-1}$) | Mean Value ($mg.ml^{-1}$) | Mean Value ($mg.ml^{-1}$) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|---|---|
| Physiological Saline | 1 | A | 1.1101 | 0.3 | 1.12 | 525.0 | 537.1 | 537.1 ± 10.1 (1.9%) | 10.5 |
| | | B | 0.3193* | 0.65 | 1.46 | 549.1 | | | |
| | 2 | A | 1.4153 | 0.7† | 1.34 | 524.1 | 530.0 | | |
| | | B | 0.0801 | 1.8 | 0.99 | 535.9 | | | |
| | 3 | A | 0.1278* | 1.5 | 1.30 | 529.3 | 539.0 | | |
| | | B | 1.3352 | 0.65† | 1.22 | 548.6 | | | |
| | 4 | A | 0.7626* | 0.35† | 0.74 | 541.0 | 542.4 | | |
| | | B | 0.6460 | 1.2† | 1.08 | 543.7 | | | |
| 1% High Viscosity Carboxy-methyl-cellulose | 5 | A | 0.4121 | 0.8 | 1.10 | 575.6 | 567.4 | 539.3 ± 20.2 (3.7%) | 10.1 |
| | | B | 0.0877 | 3.5 | 0.995 | 559.2 | | | |
| | 6 | A | 0.9301 | 0.45 | 1.28 | 527.5 | 533.3 | | |
| | | B | 0.4480 | 0.9 | 1.26 | 539.1 | | | |
| | 7 | A | 0.4600 | 0.7 | 0.99 | 530.4 | 521.1 | | |
| | | B | 0.4467 | 0.8 | 1.06 | 511.7 | | | |
| | 8 | A | 0.7542 | 0.4 | 0.95 | 543.2 | 535.3 | | |
| | | B | 0.5280 | 0.7 | 1.13 | 527.4 | | | |

*Date of Formulation:* 11 August 1980
Date of Analysis: 14 August 1980
* = Initial volume 50 ml
† = Final volume 250 ml

TABLE 9

Analysis of HMX in Dosing Suspensions Formulated During Percutaneous Toxicity Testing in Rabbits (Non-abraded Groups)
Nominal Concentration 600 $mg.ml^{-1}$ (60%)

| Sample No. | Weight of HMX Suspension (g) | V (ml) | ΔA | Concentration Found ($mg.ml^{-1}$) | Concentration Found ($mg.ml^{-1}$) | Mean Value (%) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|
| 1 | 0.7670 | 0.25 | 0.735 | 661.2 | 66.1 | | |
| 2 | 0.8020 | 0.3 | 0.96 | 688.3 | 68.8 | | |
| 3 | 0.8524 | 0.5 | 1.50 | 607.1 | 60.7 | | |
| 4 | 0.9495 | 0.4 | 1.39 | 631.3 | 63.1 | 63.0 | |
| 5 | 0.8924 | 0.4 | 1.29 | 623.4 | 62.3 | ± 3.7 | 5.0 |
| 6 | 0.6855 | 0.55 | 1.34 | 613.1 | 61.3 | (6%) | |
| 7 | 0.8845 | 0.35 | 1.045 | 582.2 | 58.2 | | |
| 8 | 0.6811 | 0.4 | 0.93 | 588.8 | 58.9 | | |
| 9 | 0.7070 | 0.3 | 0.83 | 675.0 | 67.5 | | |

Vehicle: 1% High-viscosity carboxymethylcellulose
Date of Formulation: 14 October 1980
Date of Analysis: 15 October 1980

TABLE 10

Analysis of HMX in Dosing Suspensions Formulated During Percutaneous Toxicity Testing in Rabbits (Abraded Groups)
Nominal Concentration 600 $mg.ml^{-1}$ (60%)

| Sample No. | Weight of HMX Suspension (g) | V (ml) | ΔA | Concentration Found ($mg.ml^{-1}$) | Concentration Found (%) | Mean Value (%) | % Deviation from Nominal |
|---|---|---|---|---|---|---|---|
| 10 | 0.7784 | 0.25 | 0.74 | 656.0 | 65.6 | | |
| 11 | 0.7367 | 0.45 | 1.27 | 660.8 | 66.1 | | |
| 12 | 0.6172 | 0.55 | 1.285 | 653.0 | 65.3 | | |
| 13 | 0.8084 | 0.4 | 1.19 | 634.8 | 63.5 | 64.4 | |
| 14 | 0.6880 | 0.45 | 1.14 | 635.2 | 63.5 | ± 1.9 | 7.3 |
| 15 | 0.9135 | 0.3 | 1.1045 | 657.8 | 65.8 | (2.9%) | |
| 16 | 0.8206 | 0.35 | 1.085 | 651.7 | 65.2 | | |
| 17 | 0.7328 | 0.4 | 1.02 | 600.3 | 60.0 | | |
| 18 | 0.7868 | 0.4 | 1.18 | 646.8 | 64.7 | | |

Vehicle: 1% High-viscosity carboxymethylcellulose
Date of Formulation: 15 October 1980
*Date of Analysis: 20 October 1980*

TABLE 11

Calculation of Density of HMX Suspensions

| Sample No. | Vehicle | Weight Flask (g) | Weight Flask + HMX (g) | Weight HMX (g) | Density |
|---|---|---|---|---|---|
| 1 | 1% High Viscosity Carboxymethyl-cellulose | 4.9587 | 6.2116 | 1.2529 | 1.244 |
| 2 | | 6.4865 | 7.7638 | 1.2773 | |
| 3 | | 5.5393 | 6.7776 | 1.2383 | |
| 4 | | 6.8965 | 8.1077 | 1.2112 | |
| 5 | | 7.0406 | 8.2804 | 1.2398 | |
| 6 | Physiological Saline | 5.3692 | 6.4963 | 1.1271 | 1.126 |
| 7 | | 5.2857 | 6.4068 | 1.1211 | |
| 8 | | 6.9042 | 8.0174 | 1.1132 | |
| 9 | | 6.6210 | 7.7641 | 1.1431 | |
| 10 | | 5.6160 | 6.7406 | 1.1246 | |

TABLE 12

Analysis of HMX in Diets (Method Development)
Nominal Concentration 1,250 ppm

Date of Mixing: 8 September 1980
Date of Analysis: 10 September 1980

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Peak Height Compound (mm) | Peak Height Internal Standard (mm) | Peak Height Ratio | Concentration Found (mg) | Concentration (ppm) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 5.09 | - | - | 50 | 0 | 0 | 0 | | |
| 2 | 5.10 | 1 | 1 | 50 | 77 | 56.5 | 1.363 | | |
| 3 | 4.93 | 1 | 1 | 50 | 167 | 124 | 1.347 | | |
| 4 | 4.99 | 1 | 1 | 50 | 120 | 87.5 | 1.371 | | |
| 5 | 4.89 | - | 1 | 50 | 143 | 98 | 1.459 | 6.458 | 1321 |
| 6 | 5.25 | - | 1 | 50 | 114 | 82.5 | 1.382 | 6.117 | 1165 |
| 7 | 5.15 | - | 1 | 50 | 121 | 86 | 1.407 | 6.228 | 1209 |
| 8 | 4.99 | - | 1 | 50 | 97 | 77 | 1.260 | 5.577 | 1118 |
| 9 | 4.93 | - | 1 | 50 | 85 | 70 | 1.214 | 5.374 | 1090 |
| 10 | 5.00 | - | 1 | 50 | 127 | 98 | 1.296 | 5.737 | 1147 |
| 11 | 4.86 | - | 1 | 50 | 93 | 76 | 1.224 | 5.418 | 1115 |
| 12 | 5.09 | - | 1 | 50 | 80 | 53 | 1.509 | 6.680 | 1312 |
| 13 | 5.06 | - | 1 | 50 | 119 | 92 | 1.293 | 5.723 | 1131 |
| 14 | 5.14 | - | 1 | 50 | 153 | 113 | 1.354 | 5.993 | 1166 |

HMX solution 6.02 $mg.ml^{-1}$
Internal standard solution 6.16 $mg.ml^{-1}$

Mean value 1177 $\pm$ 80 (6.8%). Deviation of mean from nominal 73 ppm (5.8%)

TABLE 13

Analysis of HMX in Diets (Method Development)
Nominal Concentration 10,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Peak Height Compound (mm) | Peak Height Internal Standard (mm) | Peak Height Ratio | Concentration Found (mg) | Concentration (ppm) |
|---|---|---|---|---|---|---|---|---|---|
| 15 | 2.48 | - | - | 104 | 0 | 0 | 0 | | |
| 16 | 2.51 | 4 | 1 | 100 | 148.5 | 80 | 1.856 | | |
| 17 | 2.53 | 4 | 1 | 100 | 171.5 | 90.5 | 1.895 | | |
| 18 | 2.50 | 4 | 1 | 100 | 67 | 34 | 1.971 | | |
| 19 | 2.57 | - | 1 | 104 | 175 | 85.5 | 2.047 | 27.76 | 10800 |
| 20 | 2.56 | - | 1 | 104 | 180 | 100.5 | 1.791 | 24.29 | 9487 |
| 21 | 2.47 | - | 1 | 104 | 140.5 | 68 | 2.066 | 28.02 | 11344 |
| 22 | 2.50 | - | 1 | 104 | 165.5 | 90.5 | 1.829 | 24.80 | 9920 |
| 23 | 2.48 | - | 1 | 104 | 165 | 87.5 | 1.886 | 25.58 | 10313 |
| 24 | 2.57 | - | 1 | 104 | 156.5 | 79 | 1.981 | 26.86 | 10453 |
| 25 | 2.55 | - | 1 | 104 | 165 | 87.5 | 1.886 | 25.58 | 10030 |
| 26 | 2.51 | - | 1 | 104 | 148 | 80 | 1.850 | 25.09 | 9996 |
| 27 | 2.55 | - | 1 | 104 | 143 | 78 | 1.833 | 24.86 | 9748 |
| 28 | 2.49 | - | 1 | 104 | 161.5 | 89 | 1.815 | 24.61 | 9885 |

HMX solution 6.465 $mg.ml^{-1}$
Internal standard solution 20.36 $mg.ml^{-1}$

Mean value 10198 $\pm$ 548 (5.4%). Deviation of mean from nominal 198 ppm (2.0%)

TABLE 14

Analysis of HMX in Diets (Method Development)
Nominal Concentration 25,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Peak Height Compound (mm) | Peak Height Internal Standard (mm) | Peak Height Ratio | Concentration Found (mg) | Concentration (ppm) |
|---|---|---|---|---|---|---|---|---|---|
| 29 | 2.52 | - | - | 110 | 0 | 0 | 0 | | |
| 30 | 2.48 | 10 | 1 | 100 | 109.5 | 73 | 1.500 | | |
| 31 | 2.49 | 10 | 1 | 100 | 151 | 99 | 1.525 | | |
| 32 | 2.47 | 10 | 1 | 100 | 155 | 104 | 1.490 | | |
| 33 | 2.53 | - | 1 | 110 | 133 | 89 | 1.494 | 63.97 | 25280 |
| 34 | 2.51 | - | 1 | 110 | 128 | 88 | 1.455 | 62.30 | 24820 |
| 35 | 2.51 | - | 1 | 110 | 113 | 76.5 | 1.477 | 63.24 | 25200 |
| 36 | 2.54 | - | 1 | 110 | 128 | 87.5 | 1.463 | 62.64 | 24660 |
| 37 | 2.51 | - | 1 | 110 | 128 | 87 | 1.471 | 62.98 | 25090 |
| 38 | 2.56 | - | 1 | 110 | 125 | 87 | 1.437 | 61.53 | 24040 |
| 39 | 2.49 | - | 1 | 110 | 133 | 95 | 1.400 | 59.94 | 24070 |
| 40 | 2.55 | - | 1 | 110 | 140 | 98 | 1.429 | 61.19 | 24000 |
| 41 | 2.51 | - | 1 | 110 | 126.5 | 86 | 1.471 | 62.98 | 25090 |
| 42 | 2.47 | - | 1 | 110 | 142 | 95 | 1.495 | 64.01 | 25910 |

HMX solution 6.444 $mg.ml^{-1}$
Internal standard solution 62.27 $mg.ml^{-1}$

Mean value 24816 $\pm$ 629 (2.5%). Deviation of mean from nominal 184 ppm (0.7%)

TABLE 15

Analysis of HMX in Diets (Method Development)
(After Storage at 40°C for 14 Days)
Nominal Concentration 1,250 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 43 | 5.07 | - | - | 50 | - |
| 44 | 5.01 | 1 | 1 | 50 | - |
| 45 | 5.02 | 1 | 1 | 50 | - |
| 46 | 4.99 | 1 | 1 | 50 | - |
| 47 | 5.01 | - | 1 | 50 | 1296 |
| 48 | 5.00 | - | 1 | 50 | 1287 |
| 49 | 4.98 | - | 1 | 50 | 1259 |
| 50 | 4.97 | - | 1 | 50 | 1266 |
| 51 | 5.02 | - | 1 | 50 | 1197 |
| 52 | 5.00 | - | 1 | 50 | 1206 |
| 53 | 4.99 | - | 1 | 50 | 1272 |
| 54 | 4.98 | - | 1 | 50 | 1207 |
| 55 | 4.97 | - | 1 | 50 | 1171 |
| 56 | 5.00 | - | 1 | 50 | 1182 |

HMX solution 6.06 $mg.ml^{-1}$
Internal standard solution 6.00 $mg.ml^{-1}$

Mean value 1234 $\pm$ 46 (3.8%). Deviation of mean from nominal 16 ppm (1.3%)

TABLE 16

Analysis of HMX in Diets (Method Development)
(After Storage at 40°C for 14 Days)
Nominal Concentration 10,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 57 | 2.50 | - | - | 104 | - |
| 58 | 2.55 | 4 | 1 | 100 | - |
| 59 | 2.44 | 4 | 1 | 100 | - |
| 60 | 2.49 | 4 | 1 | 100 | - |
| 61 | 2.53 | - | 1 | 104 | 10152 |
| 62 | 2.51 | - | 1 | 104 | 10701 |
| 63 | 2.48 | - | 1 | 104 | 10315 |
| 64 | 2.51 | - | 1 | 104 | 10551 |
| 65 | 2.55 | - | 1 | 104 | 10196 |
| 66 | 2.52 | - | 1 | 104 | 9459 |
| 67 | 2.49 | - | 1 | 104 | 9769 |
| 68 | 2.48 | - | 1 | 104 | 10137 |
| 69 | 2.51 | - | 1 | 104 | 9829 |
| 70 | 2.50 | - | 1 | 104 | 10186 |

HMX solution 6.485 $mg.ml^{-1}$
Internal standard solution 20.00 $mg.ml^{-1}$

Mean value 10130 $\pm$ 368 (3.6%). Deviation of mean from nominal 130 ppm (1.3%)

TABLE 17

Analysis of HMX in Diets (Method Development)
(After Storage at 40°C for 14 Days)
Nominal Concentration 25,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 71 | 2.50 | - | - | 110 | - |
| 72 | 2.54 | 10 | 1 | 100 | - |
| 73 | 2.49 | 10 | 1 | 100 | - |
| 74 | 2.50 | 10 | 1 | 100 | - |
| 75 | 2.48 | - | 1 | 110 | 25443 |
| 76 | 2.48 | - | 1 | 110 | 26467 |
| 77 | 2.49 | - | 1 | 110 | 25742 |
| 78 | 2.48 | - | 1 | 110 | 25354 |
| 79 | 2.48 | - | 1 | 110 | 24993 |
| 80 | 2.48 | - | 1 | 110 | 25428 |
| 81 | 2.52 | - | 1 | 110 | 25202 |
| 82 | 2.50 | - | 1 | 110 | 25238 |
| 83 | 2.50 | - | 1 | 110 | 23481 |
| 84 | 2.52 | - | 1 | 110 | 22690 |

HMX solution 6.494 $mg.ml^{-1}$
Internal standard solution 62.42 $mg.ml^{-1}$

Mean value 25004 $\pm$ 1103 (4.4%). Deviation of mean from nominal 4 ppm (0%)

TABLE 18

Analysis of HMX in Diets (Method Development)
(After Storage at Ambient for 21 Days)
Nominal Concentration 1,250 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 85 | 4.94 | - | - | 50 | - |
| 86 | 5.02 | 1 | 1 | 50 | - |
| 87 | 5.00 | 1 | 1 | 50 | - |
| 88 | 5.02 | 1 | 1 | 50 | - |
| 89 | 5.01 | - | 1 | 50 | 1268 |
| 90 | 5.01 | - | 1 | 50 | 1327 |
| 91 | 5.04 | - | 1 | 50 | 1269 |
| 92 | 5.02 | - | 1 | 50 | 1380 |
| 93 | 5.00 | - | 1 | 50 | 1270 |
| 94 | 5.03 | - | 1 | 50 | 1307 |
| 95 | 5.00 | - | 1 | 50 | 1336 |
| 96 | 5.03 | - | 1 | 50 | 1311 |
| 97 | 5.00 | - | 1 | 50 | 1217 |
| 98 | 5.02 | - | 1 | 50 | 1228 |

HMX solution 5.94 $mg.ml^{-1}$
Internal standard solution 5.86 $mg.ml^{-1}$

Mean value 1291 $\pm$ 50 (3.9%). Deviation of mean from nominal 41 ppm (3.3%)

TABLE 19

Analysis of HMX in Diets (Method Development)
(After Storage at Ambient for 21 Days)
Nominal Concentration 10,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 99 | 2.51 | - | 1 | 104 | - |
| 100 | 2.55 | 4 | 1 | 100 | - |
| 101 | 2.50 | 4 | 1 | 100 | - |
| 102 | 2.51 | 4 | 1 | 100 | - |
| 103 | 2.55 | - | 1 | 104 | 10740 |
| 104 | 2.50 | - | 1 | 104 | 10521 |
| 105 | 2.49 | - | 1 | 104 | 10002 |
| 106 | 2.54 | - | 1 | 104 | 10030 |
| 107 | 2.53 | - | 1 | 104 | 10568 |
| 108 | 2.51 | - | 1 | 104 | 10560 |
| 109 | 2.44 | - | 1 | 104 | 10062 |
| 110 | 2.51 | - | 1 | 104 | 10485 |
| 111 | 2.50 | - | 1 | 104 | 9807 |
| 112 | 2.51 | - | 1 | 104 | 10331 |

HMX solution 6.52 $mg.ml^{-1}$
Internal standard solution 20.01 $mg.ml^{-1}$

Mean value 10311 $\pm$ 312 (3.0%). Deviation of mean from nominal 311 ppm (3.1%)

TABLE 20

Analysis of HMX in Diets (Method Development)
(After Storage at Ambient for 21 Days)
Nominal Concentration 25,000 ppm

| Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution (ml) | Volume Internal Standard (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) |
|---|---|---|---|---|---|
| 113 | 2.48 | - | - | 110 | - |
| 114 | 2.51 | 10 | 1 | 100 | - |
| 115 | 2.48 | 10 | 1 | 100 | - |
| 116 | 2.53 | 10 | 1 | 100 | - |
| 117 | 2.53 | - | 1 | 110 | 24905 |
| 118 | 2.46 | - | 1 | 110 | 25945 |
| 119 | 2.50 | - | 1 | 110 | 25432 |
| 120 | 2.49 | - | 1 | 110 | 27191 |
| 121 | 2.53 | - | 1 | 110 | 24044 |
| 122 | 2.50 | - | 1 | 110 | 26280 |
| 123 | 2.53 | - | 1 | 110 | 25983 |
| 124 | 2.50 | - | 1 | 110 | 24876 |
| 125 | 2.50 | - | 1 | 110 | 26241 |
| 126 | 2.52 | - | 1 | 110 | 25081 |

HMX solution 6.518 $mg.ml^{-1}$
Internal standard solution 64.31 $mg.ml^{-1}$

Mean value 25598 $\pm$ 907 (3.5%). Deviation of mean from nominal 598 ppm (2.4%)

TABLE 21

Analysis of HMX In Diets (Method Development)
Summary of Results

| Nominal Concentration (ppm) | Concentration (ppm) Found | | |
|---|---|---|---|
| | Freshly Prepared | Storage at 40°C for 14 Days | Storage at Ambient for 21 Days |
| 1,250 | 1177 ± 80 | 1234 ± 46 | 1291 ± 50 |
| 10,000 | 10198 ± 548 | 10130 ± 368 | 10311 ± 312 |
| 25,000 | 24816 ± 629 | 25004 ± 1103 | 25598 ± 907 |

TABLE 22

Analysis of HMX In Diets Formulated During the 14 Day Toxicity Study In Rats (at commencement)

Date of Mixing: 16 October 1980
Date of Analysis: 27 October 1980

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 5.00 | - | - | 100 | 0 | | |
| 0 | 1♀♂ | 2 | 5.01 | - | - | 100 | 0 | 0 | 0 |
| | | 3 | 5.03 | - | - | 100 | 0 | | |
| | | 4 | 5.00 | - | 1 | 100 | 2723 | | |
| 2714 | 2♂ | 5 | 5.02 | - | 1 | 100 | 2842 | 2744 | 1.1 |
| | | 6 | 5.00 | - | 1 | 100 | 2667 | | |
| | | 7 | 5.00 | - | 1 | 100 | 2744 | | |
| 2642 | 2♀ | 8 | 5.00 | - | 1 | 100 | 2648 | 2765 | 4.7 |
| | | 9 | 5.01 | - | 1 | 100 | 2903 | | |
| | | 10 | 5.00 | 1 | 1 | 100 | | | |
| Standards | | 11 | 5.02 | 1 | 1 | 100 | | | |
| | | 12 | 5.02 | 1 | 1 | 100 | | | |

HMX solution 12.60 $mg.ml^{-1}$
Internal standard solution 12.08 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 13 | 3.00 | - | 1 | 104 | 7366 | | |
| 7650 | 3♂ | 14 | 3.00 | - | 1 | 104 | 7558 | 7642 | 0.1 |
| | | 15 | 3.00 | - | 1 | 104 | 8002 | | |
| | | 16 | 3.00 | - | 1 | 104 | 8430 | | |
| 8133 | 3♀ | 17 | 3.00 | - | 1 | 104 | 7855 | 8051 | 1.0 |
| | | 18 | 3.00 | - | 1 | 104 | 7867 | | |
| | | 19 | 3.03 | 4 | 1 | 100 | | | |
| Standards | | 20 | 3.00 | 4 | 1 | 100 | | | |
| | | 21 | 3.02 | 4 | 1 | 100 | | | |

HMX solution 6.135 $mg.ml^{-1}$
Internal standard solution 24.05 $mg.ml^{-1}$

TABLE 22 (continued)

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 22 | 2.50 | - | 1 | 110 | 23322 | | |
| 23550 | 4♂ | 23 | 2.50 | - | 1 | 110 | 22527 | 23113 | 1.9 |
| | | 24 | 2.49 | - | 1 | 110 | 23489 | | |
| | | 25 | 2.50 | - | 1 | 110 | 25327 | | |
| 24800 | 4♀ | 26 | 2.50 | - | 1 | 110 | 25647 | 25376 | 2.3 |
| | | 27 | 2.50 | - | 1 | 110 | 25155 | | |
| | | 28 | 2.50 | 10 | 1 | 100 | | | |
| Standards | | 29 | 2.50 | 10 | 1 | 100 | | | |
| | | 30 | 2.51 | 10 | 1 | 100 | | | |

HMX solution 6.560 $mg.ml^{-1}$
Internal standard solution 61.465 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 31 | 2.50 | - | 1 | 130 | 67087 | | |
| 72000 | 5♂ | 32 | 2.49 | - | 1 | 130 | 70922 | 69955 | 2.8 |
| | | 33 | 2.50 | - | 1 | 130 | 71856 | | |
| | | 34 | 2.50 | - | 1 | 130 | 72017 | | |
| 72000 | 5♀ | 35 | 2.50 | - | 1 | 130 | 69442 | 70756 | 1.7 |
| | | 36 | 2.50 | - | 1 | 130 | 70809 | | |
| | | 37 | 2.50 | 30 | 1 | 100 | | | |
| Standards | | 38 | 2.50 | 30 | 1 | 100 | | | |
| | | 39 | 2.50 | 30 | 1 | 100 | | | |

HMX solution 6.075 $mg.ml^{-1}$
Internal standard solution 179.92 $mg.ml^{-1}$

TABLE 23

Analysis of HMX in Diets Formulated During the 14 Day Toxicity Study in Rats (at Day 8)

Date of Mixing: 23 October 1980
Date of Analysis: 3 November 1980

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 82 | 5.00 | - | - | 104 | 0 | | |
| 0 | 1♀♂ | 83 | 5.01 | - | - | 104 | 0 | 0 | 0 |
| | | 84 | 5.01 | - | - | 104 | 0 | | |
| | | 85 | 5.00 | - | 1 | 104 | 5107 | | |
| 4995 | 2♀ | 86 | 5.00 | - | 1 | 104 | 4893 | 4930 | 1.3 |
| | | 87 | 5.00 | - | 1 | 104 | 4791 | | |
| | | 88 | 5.00 | - | 1 | 104 | 4055 | | |
| 3869 | 2♂ | 89 | 5.00 | - | 1 | 104 | 3876 | 3935 | 1.7 |
| | | 90 | 5.00 | - | 1 | 104 | 3873 | | |
| | | 91 | 5.00 | 4 | 1 | 100 | | | |
| Standards | | 92 | 5.00 | 4 | 1 | 100 | | | |
| | | 93 | 5.00 | 4 | 1 | 100 | | | |

HMX solution 5.01 $mg.ml^{-1}$
Internal standard solution 19.905 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 94 | 2.50 | - | 1 | 105 | 16969 | | |
| 15000 | 3♀ | 95 | 2.51 | - | 1 | 105 | 16941 | 16586 | 10.6 |
| | | 96 | 2.50 | - | 1 | 105 | 15847 | | |
| | | 97 | 2.50 | - | 1 | 105 | 10752 | | |
| 11448 | 3♂ | 98 | 2.50 | - | 1 | 105 | 10933 | 10785 | 5.8 |
| | | 99 | 2.51 | - | 1 | 105 | 10671 | | |
| | | 100 | 2.50 | 5 | 1 | 100 | | | |
| Standards | | 101 | 2.50 | 5 | 1 | 100 | | | |
| | | 102 | 2.50 | 5 | 1 | 100 | | | |

HMX solution 7.49 $mg.ml^{-1}$
Internal standard solution 28.04 $mg.ml^{-1}$

TABLE 23 (continued)

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| 49000 | 4♀ | 103 | 2.50 | - | 1 | 120 | 48703 | 48739 | 0.5 |
| | | 104 | 2.50 | - | 1 | 120 | 48370 | | |
| | | 105 | 2.50 | - | 1 | 120 | 49143 | | |
| 36720 | 4♂ | 106 | 2.50 | - | 1 | 120 | 37858 | 36961 | 0.7 |
| | | 107 | 2.50 | - | 1 | 120 | 36512 | | |
| | | 108 | 2.49 | - | 1 | 120 | 36514 | | |
| Standards | | 109 | 2.50 | 20 | 1 | 100 | | | |
| | | 110 | 2.50 | 20 | 1 | 100 | | | |
| | | 111 | 2.50 | 20 | 1 | 100 | | | |

HMX solution 5.96 $mg.ml^{-1}$
Internal standard solution 100.06 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 175500 | 5♀ | 112 | 2.00 | - | 4 | 160 | 175287 | 177482 | 1.1 |
| | | 113 | 2.00 | - | 4 | 160 | 181643 | | |
| | | 114 | 2.00 | - | 4 | 160 | 175516 | | |
| Standards | | 115 | 2.00 | 60 | 4 | 100 | | | |
| | | 116 | 2.00 | 60 | 4 | 100 | | | |
| | | 117 | 2.00 | 60 | 4 | 100 | | | |

HMX solution 5.99 $mg.ml^{-1}$
Internal standard solution 90.086 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 120600 | 5♂ | 118 | 2.00 | - | 3 | 140 | 129395 | 125781 | 4.3 |
| | | 119 | 2.00 | - | 3 | 140 | 125597 | | |
| | | 120 | 2.00 | - | 3 | 140 | 122352 | | |
| Standards | | 121 | 2.00 | 40 | 3 | 100 | | | |
| | | 122 | 2.00 | 40 | 3 | 100 | | | |
| | | 123 | 2.00 | 40 | 3 | 100 | | | |

HMX solution 6.004 $mg.ml^{-1}$
Internal standard solution 79.89 $mg.ml^{-1}$

TABLE 24

Analysis of HMX in Diet: Results of Quality Control Sample Analyses (Analysed Concurrently with Diets Formulated During the 14 Day Toxicity Study in Rats (at Day 8))

| Group | Nominal Weight (mg) | Analytical Sample No. | Weight Found (mg) | Mean Weight (mg) | Deviation of Mean (%) |
|---|---|---|---|---|---|
| 1♂♀ | | 124 | 18.32 | | |
| 2♂♀ | 18.52 | 125 | 18.93 | 19.20 | 3.8 |
| | | 126 | 20.34 | | |
| | | 127 | 25.35 | | |
| 3♂♀ | 25.60 | 128 | 25.52 | 25.56 | 0.2 |
| | | 129 | 25.80 | | |
| | | 130 | 126.56 | | |
| 4♂♀ | 127.40 | 131 | 126.35 | 125.41 | 1.6 |
| | | 132 | 123.32 | | |
| | | 133 | 423.58 | | |
| 5♀ | 413.42 | 134 | 409.84 | 412.95 | 0.1 |
| | | 135 | 405.43 | | |
| | | 136 | 260.57 | | |
| 5♂ | 278.35 | 137 | 281.61 | 270.47 | 2.8 |
| | | 138 | 269.22 | | |

TABLE 25

Analysis of HMX in Diets Formulated During the 14 Day Toxicity Study in Mice (at commencement)

Date of Mixing: 20 October 1980
Date of Analysis: 3 November 1980

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 40 | 5.00 | - | - | 100 | 0 | | |
| 0 | 1♀♂ | 41 | 5.01 | - | - | 100 | 0 | 0 | 0 |
| | | 42 | 5.02 | - | - | 100 | 0 | | |
| | | 43 | 5.00 | - | 1 | 100 | 527 | | |
| 483 | 2♂ | 44 | 5.00 | - | 1 | 100 | 478 | 521 | 7.9 |
| | | 45 | 5.00 | - | 1 | 100 | 559 | | |
| | | 46 | 5.00 | 1 | 1 | 100 | | | |
| Standards | | 47 | 5.00 | 1 | 1 | 100 | | | |
| | | 48 | 5.00 | 1 | 1 | 100 | | | |

HMX solution 2.40 $mg.ml^{-1}$
Internal standard solution 2.468 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 49 | 5.01 | - | 1 | 50 | 1295 | | |
| 1331 | 2♀ | 50 | 5.00 | - | 1 | 50 | 1293 | 1320 | 0.8 |
| | | 51 | 5.00 | - | 1 | 50 | 1371 | | |
| | | 52 | 5.02 | - | 1 | 50 | 1534 | | |
| 1513 | 3♂ | 53 | 5.00 | - | 1 | 50 | 1515 | 1547 | 2.2 |
| | | 54 | 5.00 | - | 1 | 50 | 1593 | | |
| | | 55 | 5.00 | 1 | 1 | 50 | | | |
| Standards | | 56 | 5.00 | 1 | 1 | 50 | | | |
| | | 57 | 5.00 | 1 | 1 | 50 | | | |

HMX solution 6.68 $mg.ml^{-1}$
Internal standard solution 7.032 $mg.ml^{-1}$

TABLE 25 (continued)

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 58 | 5.02 | - | 1 | 104 | 3088 | | |
| 3325 | 3♀ | 59 | 5.00 | - | 1 | 104 | 3115 | 3152 | 5.2 |
| | | 60 | 5.00 | - | 1 | 104 | 3253 | | |
| | | 61 | 5.00 | - | 1 | 104 | 4833 | | |
| 4856 | 4♂ | 62 | 4.99 | - | 1 | 104 | 4562 | 5278 | 8.7 |
| | | 63 | 5.01 | - | 1 | 104 | 6439 | | |
| | | 64 | 5.00 | 4 | 1 | 100 | | | |
| Standards | | 65 | 5.02 | 4 | 1 | 100 | | | |
| | | 66 | 5.02 | 4 | 1 | 100 | | | |

HMX solution 4.992 $mg.ml^{-1}$
Internal standard solution 20.136 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 67 | 2.50 | - | 1 | 105 | 7878 | | |
| 7926 | 4♀ | 68 | 2.50 | - | 1 | 105 | 7835 | 8109 | 2.3 |
| | | 69 | 2.49 | - | 1 | 105 | 8614 | | |
| | | 70 | 2.50 | - | 1 | 105 | 11852 | | |
| 12547 | 5♂ | 71 | 2.52 | - | 1 | 105 | 12904 | 12820 | 2.2 |
| | | 72 | 2.51 | - | 1 | 105 | 13705 | | |
| | | 73 | 2.50 | 5 | 1 | 100 | | | |
| Standards | | 74 | 2.50 | 5 | 1 | 100 | | | |
| | | 75 | 2.50 | 5 | 1 | 100 | | | |

HMX solution 5.008 $mg.ml^{-1}$
Internal standard solution 24.77 $mg.ml^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 76 | 2.50 | - | 1 | 110 | 22237 | | |
| 21078 | 5♀ | 77 | 2.51 | - | 1 | 110 | 22115 | 22227 | 5.5 |
| | | 78 | 2.52 | - | 1 | 110 | 22328 | | |
| | | 79 | 2.50 | 10 | 1 | 100 | | | |
| Standards | | 80 | 2.50 | 10 | 1 | 100 | | | |
| | | 81 | 2.50 | 10 | 1 | 100 | | | |

HMX solution 5.058 $mg.ml^{-1}$
Internal standard solution 50.184 $mg.ml^{-1}$

TABLE 26

Analysis of HMX in Diets Formulated During the 14 Day Toxicity Study in Mice (at Day 8)

Date of Mixing: 27 October 1980
Date of Analysis: 3 November 1980

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | 140 | 5.00 | - | - | 100 | 0 | | |
| 0 | 1♂♀ | 141 | 5.00 | - | - | 100 | 0 | 0 | 0 |
| | | 142 | 5.00 | - | - | 100 | 0 | | |
| | | 143 | 5.00 | - | 1 | 100 | 548 | | |
| 500 | 2♂ | 144 | 5.00 | - | 1 | 100 | 505 | 515 | 3.0 |
| | | 145 | 5.00 | - | 1 | 100 | 493 | | |
| | | 146 | 5.00 | - | 1 | 100 | 1463 | | |
| 1458 | 2♀ | 147 | 5.00 | - | 1 | 100 | 1547 | 1521 | 4.3 |
| | | 148 | 5.00 | - | 1 | 100 | 1553 | | |
| | | 149 | 5.00 | 1 | 1 | 100 | | | |
| Standards | | 150 | 5.00 | 1 | 1 | 100 | | | |
| | | 151 | 5.00 | 1 | 1 | 100 | | | |

HMX solution 5.064 mg.ml$^{-1}$
Internal standard solution 5.012 mg.ml$^{-1}$

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 152 | 5.00 | - | 1 | 104 | 2026 | | |
| 2014 | 3♂ | 153 | 5.00 | - | 1 | 104 | 2127 | 2082 | 3.4 |
| | | 154 | 5.00 | - | 1 | 104 | 2093 | | |
| | | 155 | 5.00 | - | 1 | 104 | 4698 | | |
| 4556 | 3♀ | 156 | 5.00 | - | 1 | 104 | 4916 | 4852 | 6.5 |
| | | 157 | 5.00 | - | 1 | 104 | 4941 | | |
| | | 158 | 5.00 | 4 | 1 | 100 | | | |
| Standards | | 159 | 5.00 | 4 | 1 | 100 | | | |
| | | 160 | 5.00 | 4 | 1 | 100 | | | |

HMX solution 3.995 mg.ml$^{-1}$
Internal standard solution 15.920 mg.ml$^{-1}$

TABLE 26 (continued)

| Nominal Concentration (ppm) | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Concentration Found (ppm) | Mean Concentration Found (ppm) | Deviation of Mean Concentration (%) |
|---|---|---|---|---|---|---|---|---|---|
| 11613 | 4♀ | 161 | 2.50 | - | 1 | 105 | 11221 | 11209 | 3.5 |
| | | 162 | 2.50 | - | 1 | 105 | 11256 | | |
| | | 163 | 2.50 | - | 1 | 105 | 11151 | | |
| Standards | | 164 | 2.50 | 5 | 1 | 100 | | | |
| | | 165 | 2.50 | 5 | 1 | 100 | | | |
| | | 166 | 2.50 | 5 | 1 | 100 | | | |

HMX solution 5.996 $mg.ml^{-1}$
Internal standard solution 29.888 $mg.ml^{-1}$

TABLE 27

Analysis of HMX in Diets: Results of Quality Control Sample Analyses [Analysed Concurrently with Diets Formulated During the 14 Day Toxicity Study in Mice (at Day 8)]

| Group | Nominal Weight (mg) | Analytical Sample No. | Weight Found (mg) | Mean Weight (mg) | Deviation of Mean (%) |
|---|---|---|---|---|---|
| 1♂♀ | | 167 | 5.24 | | |
| 2♂♀ | 5.34 | 168 | 5.47 | 5.39 | 0.9 |
| | | 169 | 5.45 | | |
| | | 170 | 18.03 | | |
| 3♂♀ | 17.92 | 171 | 17.78 | 17.80 | 0.4 |
| | | 172 | 17.59 | | |
| | | 173 | 34.96 | | |
| 4♀ | 36.03 | 174 | 35.33 | 35.00 | 2.8 |
| | | 175 | 34.68 | | |

TABLE 28

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 23 October 1980
Date of Analysis: 3 November 1980

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 1♀♂ | 176 | 5.02 | - | - | 100 | 0 | 0 | 0 |
| | | 177 | 4.98 | - | - | 100 | 0 | | |
| | | 178 | 5.03 | - | - | 100 | 0 | | |
| 2.02 | Standards | 179 | 5.00 | 1 | 1 | 100 | | | |
| | | 180 | 4.99 | 1 | 1 | 100 | | | |
| | | 181 | 4.93 | 1 | 1 | 100 | | | |
| 2.13 | QC | 182 | 4.97 | 1 | 1 | 100 | 1.73 | 1.98 | 7.0 |
| | | 183 | 5.01 | 1 | 1 | 100 | 2.10 | | |
| | | 184 | 5.00 | 1 | 1 | 100 | 2.10 | | |
| 420 | 2♂ | 185 | 5.07 | - | 1 | 100 | 417 | 409 | 2.6 |
| | | 186 | 5.07 | - | 1 | 100 | 413 | | |
| | | 187 | 4.98 | - | 1 | 100 | 398 | | |
| 415 | 2♀ | 188 | 5.02 | - | 1 | 100 | 438 | 438 | 5.5* |
| | | 189 | 5.02 | - | 1 | 100 | 637 | | |
| | | 190 | 5.00 | - | 1 | 100 | 438 | | |

HMX solution 2.02 $mg.ml^{-1}$
Internal standard solution 1.97 $mg.ml^{-1}$
QC solution of HMX 2.13 $mg.ml^{-1}$
* = Mean of 2 results only

TABLE 28 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 5.51 | Stand-ards | 191 | 5.03 | 1 | 1 | 100 | | | |
| | | 192 | 5.01 | 1 | 1 | 100 | | | |
| | | 193 | 4.92 | 1 | 1 | 100 | | | |
| 5.20 | QC | 194 | 5.03 | 1 | 1 | 100 | 5.34 | 5.37 | 3.3 |
| | | 195 | 5.00 | 1 | 1 | 100 | 5.50 | | |
| | | 196 | 4.95 | 1 | 1 | 100 | 5.27 | | |
| 1250 | 3♂ | 197 | 4.99 | - | 1 | 100 | 1281 | 1274 | 1.9 |
| | | 198 | 4.97 | - | 1 | 100 | 1283 | | |
| | | 199 | 4.96 | - | 1 | 100 | 1258 | | |
| 864 | 3♀ | 200 | 5.02 | - | 1 | 100 | 896 | 877 | 1.5 |
| | | 201 | 5.00 | - | 1 | 100 | 873 | | |
| | | 202 | 5.00 | - | 1 | 100 | 861 | | |

HMX solution 5.51 $mg.ml^{-1}$

Internal standard solution 4.94 $mg.ml^{-1}$

QC solution of HMX 5.20 $mg.ml^{-1}$

TABLE 28 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 12.15 | Stand-ards | 203 | 5.00 | 3 | 1 | 100 | | | |
| | | 204 | 4.97 | 3 | 1 | 100 | | | |
| | | 205 | 5.10 | 3 | 1 | 100 | | | |
| 11.91 | QC | 206 | 5.01 | 3 | 1 | 100 | 12.05 | 11.81 | 0.8 |
| | | 207 | 4.95 | 3 | 1 | 100 | 11.98 | | |
| | | 208 | 4.99 | 3 | 1 | 100 | 11.41 | | |
| 2305 | 4♀ | 209 | 5.03 | - | 1 | 103 | 2360 | 2309 | 0.2 |
| | | 210 | 5.10 | - | 1 | 103 | 2290 | | |
| | | 211 | 5.10 | - | 1 | 103 | 2276 | | |

HMX solution 4.05 $mg.ml^{-1}$
Internal standard solution 12.295 $mg.ml^{-1}$
QC solution of HMX 3.97 $mg.ml^{-1}$

TABLE 28 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 22.575 | Stand-ards | 212 | 5.04 | 5 | 1 | 100 | | | |
| | | 213 | 5.00 | 5 | 1 | 100 | | | |
| | | 214 | 4.99 | 5 | 1 | 100 | | | |
| 22.78 | QC | 215 | 5.02 | 5 | 1 | 100 | 22.75 | | |
| | | 216 | 5.08 | 5 | 1 | 100 | 22.42 | 22.76 | 0 |
| | | 217 | 5.00 | 5 | 1 | 100 | 23.11 | | |
| 3930 | 4♂ | 218 | 5.05 | - | 1 | 105 | 3973 | | |
| | | 219 | 4.97 | - | 1 | 105 | 3859 | 3936 | 0.2 |
| | | 220 | 4.89 | - | 1 | 105 | 3977 | | |
| 5151 | 5♀ | 221 | 4.97 | - | 1 | 105 | 4918 | | |
| | | 222 | 5.00 | - | 1 | 105 | 4933 | 5019 | 2.6 |
| | | 223 | 4.94 | - | 1 | 105 | 5205 | | |

HMX solution 4.515 $mg.ml^{-1}$
Internal standard solution 22.3 $mg.ml^{-1}$
QC solution of HMX 4.555 $mg.ml^{-1}$

TABLE 28 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 30.07 | Stand-ards | 224 | 5.07 | 6 | 1 | 100 | | | |
| | | 225 | 5.02 | 6 | 1 | 100 | | | |
| | | 226 | 5.05 | 6 | 1 | 100 | | | |
| 29.71 | QC | 227 | 5.03 | 6 | 1 | 100 | 30.63 | 30.27 | 1.8 |
| | | 228 | 5.00 | 6 | 1 | 100 | 30.03 | | |
| | | 229 | 5.00 | 6 | 1 | 100 | 30.06 | | |
| 11520 | 5♂ | 230 | 5.07 | - | 1 | 106 | 11135 | 11293 | 2.0 |
| | | 231 | 5.00 | - | 1 | 106 | 11093 | | |
| | | 232 | 5.02 | - | 1 | 106 | 11650 | | |
| 12577 | 6♀ | 233 | 5.02 | - | 1 | 106 | 4814 | 4882 | 61.2** |
| | | 234 | 4.90 | - | 1 | 106 | 5004 | | |
| | | 235 | 5.00 | - | 1 | 106 | 4827 | | |

HMX solution 5.012 $mg.ml^{-1}$

Internal standard solution 30.01 $mg.ml^{-1}$

QC solution of HMX 4.952 $mg.ml^{-1}$

** = It is suggested that Group 5♀ may have been sampled in error since the dispensary weighing records are correct for the preparation of a nominal 12577 ppm diet

TABLE 28 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 81.94 | Stand-ards | 236 | 4.97 | 17 | 1 | 100 | | | |
| | | 237 | 5.03 | 17 | 1 | 100 | | | |
| | | 238 | 5.00 | 17 | 1 | 100 | | | |
| 84.49 | QC | 239 | 4.99 | 17 | 1 | 100 | 85.38 | 85.49 | 1.2 |
| | | 240 | 4.99 | 17 | 1 | 100 | 85.25 | | |
| | | 241 | 5.03 | 17 | 1 | 100 | 85.85 | | |
| 33600 | 6♂ | 242 | 5.04 | - | 1 | 117 | 32233 | 32925 | 2.0 |
| | | 243 | 4.99 | - | 1 | 117 | 32656 | | |
| | | 244 | 5.02 | - | 1 | 117 | 33887 | | |

HMX solution 4.82 $mg.ml^{-1}$
Internal standard solution 85.01 $mg.ml^{-1}$
QC solution of HMX 4.97 $mg.ml^{-1}$

TABLE 29

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 22 December 1980 (Week 1)
Date of Analysis: 23 January 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 13♀ | 245 | 5.32 | - | - | 100 | 0 | 0 | 0 |
| | | 246 | 5.45 | - | - | 100 | 0 | | |
| | | 247 | 5.00 | - | - | 100 | 0 | | |
| 1.98 | Standards | 251 | 5.18 | 1 | 1 | 100 | | | |
| | | 252 | 4.94 | 1 | 1 | 100 | | | |
| | | 253 | 5.29 | 1 | 1 | 100 | | | |
| 2.56 | QC | 254 | 5.16 | - | 1 | 100 | 2.28 | 2.28 | 10.9 |
| | | 255 | 5.36 | 1 | 1 | 100 | 2.27 | | |
| | | 256 | 5.38 | 1 | 1 | 100 | 2.28 | | |
| 450 | 2♂ | 257 | 6.72 | - | 1 | 100 | 468 | 426 | 5.4 |
| | | 258 | 5.15 | - | 1 | 100 | 409 | | |
| | | 259 | 5.50 | - | 1 | 100 | 400 | | |
| 431 | 2♀ | 263 | 5.11 | - | 1 | 100 | 412 | 408 | 5.3* |
| | | 264 | 5.32 | - | 1 | 100 | 404 | | |
| | | 265 | 5.15 | - | 1 | 100 | 569 | | |

HMX solution 1.98 $mg.ml^{-1}$
Internal standard solution 2.428 $mg.ml^{-1}$
QC solution of HMX 2.56 $mg.ml^{-1}$
* = Mean of 2 results only

TABLE 29 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 5.66 | Stand-ards | 269 | 5.44 | 1 | 1 | 100 | | | |
| | | 270 | 5.30 | 1 | 1 | 100 | | | |
| | | 271 | 5.89 | 1 | 1 | 100 | | | |
| 4.86 | QC | 272 | 6.49 | 1 | 1 | 100 | 5.47 | 5.50 | 13.2 |
| | | 273 | 6.05 | 1 | 1 | 100 | 5.53 | | |
| | | 274 | 5.21 | 1 | 1 | 100 | 5.51 | | |
| 1400 | 3♂ | 275 | 4.83 | - | 1 | 100 | 1439 | 1418 | 1.3 |
| | | 276 | 5.65 | - | 1 | 100 | 1359 | | |
| | | 277 | 4.92 | - | 1 | 100 | 1457 | | |
| 1080 | 3♀ | 281 | 5.57 | - | 1 | 100 | 1169 | 1149 | 6.4 |
| | | 282 | 5.34 | - | 1 | 100 | 1134 | | |
| | | 283 | 5.10 | - | 1 | 100 | 1143 | | |

HMX solution 5.66 $mg.ml^{-1}$
Internal standard solution 5.776 $mg.ml^{-1}$
QC solution of HMX 4.86 $mg.ml^{-1}$

TABLE 29 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 11.595 | Standards | 287 | 5.00 | 3 | 1 | 100 | | | |
| | | 288 | 5.93 | 3 | 1 | 100 | | | |
| | | 289 | 5.75 | 3 | 1 | 100 | | | |
| 10.86 | QC | 290 | 5.66 | 3 | 1 | 100 | 11.30 | 11.19 | 3.0 |
| | | 291 | 5.35 | 3 | 1 | 100 | 11.19 | | |
| | | 292 | 5.05 | 3 | 1 | 100 | 11.07 | | |
| 2916 | 4♀ | 293 | 6.85 | - | 1 | 103 | 2772 | 2795 | 4.2 |
| | | 294 | 5.25 | - | 1 | 103 | 2788 | | |
| | | 295 | 5.87 | - | 1 | 103 | 2824 | | |

HMX solution 3.865 $mg.ml^{-1}$
Internal standard solution 11.836 $mg.ml^{-1}$
QC solution of HMX 3.62 $mg.ml^{-1}$

TABLE 29 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 23.06 | Stand-ards | 299 | 5.12 | 5 | 1 | 100 | | | |
| | | 300 | 5.18 | 5 | 1 | 100 | | | |
| | | 301 | 4.82 | 5 | 1 | 100 | | | |
| 20.43 | QC | 302 | 4.96 | 5 | 1 | 100 | 20.47 | | |
| | | 303 | 4.96 | 5 | 1 | 100 | 20.49 | 20.70 | 1.3 |
| | | 304 | 5.04 | 5 | 1 | 100 | 21.15 | | |
| 4345 | 4♂ | 305 | 6.36 | - | 1 | 105 | 4320 | | |
| | | 306 | 5.17 | - | 1 | 105 | 4392 | 4357 | 0.3 |
| | | 307 | 6.31 | - | 1 | 105 | 4358 | | |
| 6526 | 5♀ | 311 | 5.60 | - | 1 | 105 | 6632 | | |
| | | 312 | 6.50 | - | 1 | 105 | 6958 | 6721 | 3.0 |
| | | 313 | 5.30 | - | 1 | 105 | 6573 | | |

HMX solution 4.612 $mg.ml^{-1}$
Internal standard solution 22.812 $mg.ml^{-1}$
QC solution of HMX 4.085 $mg.ml^{-1}$

TABLE 29 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 31.20 | Stand-ards | 317 | 2.46 | 6 | 1 | 100 | | | |
| | | 318 | 2.99 | 6 | 1 | 100 | | | |
| | | 319 | 2.91 | 6 | 1 | 100 | | | |
| 29.30 | QC | 320 | 2.94 | 6 | 1 | 100 | 30.07 | 29.31 | 0 |
| | | 321 | 2.92 | 6 | 1 | 100 | 29.41 | | |
| | | 322 | 2.60 | 6 | 1 | 100 | 28.44 | | |
| 12921 | 5♂ | 323 | 3.23 | - | 1 | 106 | 12882 | 13166 | 1.9 |
| | | 324 | 2.99 | - | 1 | 106 | 13147 | | |
| | | 325 | 2.42 | - | 1 | 106 | 13470 | | |
| 15833 | 6♀ | 329 | 2.47 | - | 1 | 106 | 16760 | 16580 | 4.7* |
| | | 330 | 3.08 | - | 1 | 106 | 16400 | | |
| | | 331 | 2.26 | - | 1 | 106 | 24167 | | |

HMX solution 5.20 $mg.ml^{-1}$

Internal standard solution 30.072 $mg.ml^{-1}$

QC solution of HMX 4.884 $mg.ml^{-1}$

* = Mean of 2 results only

TABLE 29 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 88.20 | Stand-ards | 335 | 2.87 | 17 | 1 | 100 | | | |
| | | 336 | 2.50 | 17 | 1 | 100 | | | |
| | | 337 | 2.38 | 17 | 1 | 100 | | | |
| 81.311 | QC | 338 | 2.90 | 17 | 1 | 100 | 81.36 | 82.11 | 1.0 |
| | | 339 | 2.63 | 17 | 1 | 100 | 83.43 | | |
| | | 340 | 2.49 | 17 | 1 | 100 | 81.55 | | |
| 37538 | 63 | 341 | 2.38 | - | 1 | 117 | 36085 | 36915 | 1.7 |
| | | 342 | 3.03 | - | 1 | 117 | 37335 | | |
| | | 343 | 2.46 | - | 1 | 117 | 37325 | | |

HMX solution 5.188 $mg.ml^{-1}$
Internal standard solution 87.608 $mg.ml^{-1}$
QC solution of HMX 4.783 $mg.ml^{-1}$

TABLE 30

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 29 December 1980 (Week 2)
Date of Analysis: 23 January 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 1♂♀ | 248 | 4.97 | - | - | 100 | 0 | 0 | 0 |
| | | 249 | 5.20 | - | - | 100 | 0 | | |
| | | 250 | 5.13 | - | - | 100 | 0 | | |
| 480 | 2♂ | 260 | 4.97 | - | 1 | 100 | 483 | 482 | 0.4 |
| | | 261 | 5.68 | - | 1 | 100 | 474 | | |
| | | 262 | 5.10 | - | 1 | 100 | 488 | | |
| 467 | 2♂ | 266 | 5.37 | - | 1 | 100 | 911 | 896 | • |
| | | 267 | 5.17 | - | 1 | 100 | 866 | | |
| | | 268 | 5.14 | - | 1 | 100 | 911 | | |
| 1389 | 3♂ | 278 | 4.74 | - | 1 | 100 | 1453 | 1494 | 7.6 |
| | | 279 | 5.42 | - | 1 | 100 | 1507 | | |
| | | 280 | 6.24 | - | 1 | 100 | 1522 | | |

N.B. Analysed with diet prepared 22 December 1980 using the same standard solutions

• = Since the values obtained are markedly different from the nominal concentrations the archive samples for these groups have also been analysed (see p. 65)

TABLE 30 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1062 | 3♀ | 284 | 5.34 | - | 1 | 100 | 521 | 548 | • |
| | | 285 | 4.88 | - | 1 | 100 | 570 | | |
| | | 286 | 5.04 | - | 1 | 100 | 552 | | |
| 2527 | 4♀ | 296 | 5.18 | - | 1 | 103 | 2819 | 2776 | 9.9 |
| | | 297 | 4.85 | - | 1 | 103 | 2771 | | |
| | | 298 | 5.80 | - | 1 | 103 | 2739 | | |
| 4288 | 4♂ | 308 | 4.84 | - | 1 | 105 | 4735 | 4655 | 8.6 |
| | | 309 | 5.27 | - | 1 | 105 | 4695 | | |
| | | 310 | 5.81 | - | 1 | 105 | 4536 | | |
| 5654 | 5♀ | 314 | 4.86 | - | 1 | 105 | 6185 | 5929 | 4.6 |
| | | 315 | 5.21 | - | 1 | 105 | 6054 | | |
| | | 316 | 5.28 | - | 1 | 105 | 5547 | | |

° = Since the values obtained are markedly different from the nominal concentrations the archive samples for these groups have also been analysed (see p. 65)

TABLE 30 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 12764 | 5♂ | 326 | 2.82 | - | 1 | 106 | 13099 | 12959 | 1.5 |
| | | 327 | 2.60 | - | 1 | 106 | 12813 | | |
| | | 328 | 2.36 | - | 1 | 106 | 12964 | | |
| 13065 | 6♀ | 332 | 2.47 | - | 1 | 106 | 12874 | 13817 | 5.8 |
| | | 333 | 2.45 | - | 1 | 106 | 15603 | | |
| | | 334 | 2.93 | - | 1 | 106 | 12973 | | |
| 36500 | 6♂ | 344 | 2.93 | - | 1 | 117 | 31888 | 34104 | 6.6 |
| | | 345 | 2.51 | - | 1 | 117 | 37009 | | |
| | | 346 | 2.52 | - | 1 | 117 | 33414 | | |

TABLE 30 (continued)

Analysis of Samples from Archives

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 3.84 | Standards | 434 | 4.96 | 1 | 1 | 100 | | | |
| | | 435 | 4.99 | 1 | 1 | 100 | | | |
| | | 436 | 4.94 | 1 | 1 | 100 | | | |
| 467 | 2♀ | 437 | 5.34 | - | 1 | 100 | 1053 | | ϕ |
| | | 438 | 5.16 | - | 1 | 100 | 1091 | 1072 | 1.0 |
| 1062 | 3♀ | 439 | 4.89 | - | 1 | 100 | 504 | | |
| | | 440 | 5.79 | - | 1 | 100 | 460 | 482 | 3.2 |

HMX solution 3.84 $mg.ml^{-1}$

Internal standard solution 4.01 $mg.ml^{-1}$

ϕ = Assumes Group 2♀ and Group 3♀ samples have been interchanged

TABLE 31

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 5 January 1981 (Week 3)
Date of Analysis: 21 January 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 18♀ | 347 | 4.88 | - | - | 100 | 0 | 0 | 0 |
| | | 348 | 4.91 | - | - | 100 | 0 | | |
| | | 349 | 5.09 | - | - | 100 | 0 | | |
| 1.68 | Standards | 350 | 5.21 | 1 | 1 | 100 | | | |
| | | 351 | 4.90 | 1 | 1 | 100 | | | |
| | | 352 | 4.98 | 1 | 1 | 100 | | | |
| 1.66 | QC | 353 | 5.18 | 1 | 1 | 100 | 1.76 | 1.82 | 9.6 |
| | | 354 | 5.11 | 1 | 1 | 100 | 1.85 | | |
| | | 355 | 5.61 | 1 | 1 | 100 | 1.84 | | |
| 506 | 2♂ | 356 | 5.31 | - | 1 | 100 | 552 | 549 | 8.5 |
| | | 357 | 5.70 | - | 1 | 100 | 543 | | |
| | | 358 | 5.32 | - | 1 | 100 | 551 | | |
| 474 | 2♀ | 359 | 5.70 | - | 1 | 100 | 517 | 533 | 12.4 |
| | | 360 | 5.69 | - | 1 | 100 | 541 | | |
| | | | | | | | 542 | | |
| | | 361 | 5.43 | - | 1 | 100 | 539 | | |
| | | | | | | | 524 | | |

HMX solution 1.68 $mg.ml^{-1}$
Internal standard solution 1.732 $mg.ml^{-1}$
QC solution of HMX 1.66 $mg.ml^{-1}$

TABLE 31 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 5.00 | Standards | 362 | 4.87 | 1 | 1 | 100 | | | |
| | | 363 | 4.79 | 1 | 1 | 100 | | | |
| | | 364 | 5.58 | 1 | 1 | 100 | | | |
| 5.74 | QC | 365 | 5.77 | 1 | 1 | 100 | 5.52 | | |
| | | 366 | 5.93 | 1 | 1 | 100 | 5.09 | 5.28 | 8.0 |
| | | 367 | 5.02 | 1 | 1 | 100 | 5.24 | | |
| 1482 | ♂ | 368 | 5.52 | - | 1 | 100 | 1459 | | |
| | | 369 | 5.57 | - | 1 | 100 | 1447 | 1449 | 2.2 |
| | | 370 | 5.16 | - | 1 | 100 | 1440 | | |
| 1114 | ♀ | 371 | 5.41 | - | 1 | 100 | 1169 | | |
| | | 372 | 5.71 | - | 1 | 100 | 1171 | 1156 | 3.7 |
| | | 373 | 5.31 | - | 1 | 100 | 1127 | | |

HMX solution 5.00 $mg.ml^{-1}$

Internal standard solution 4.432 $mg.ml^{-1}$

QC solution of HMX 5.74 $mg.ml^{-1}$

TABLE 31 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 12.24 | Stand-ards | 374 | 4.78 | 3 | 1 | 100 | | | |
| | | 375 | 5.22 | 3 | 1 | 100 | | | |
| | | 376 | 5.45 | 3 | 1 | 100 | | | |
| 9.12 | QC | 377 | 5.68 | 3 | 1 | 100 | 9.02 | | |
| | | 378 | 5.19 | 3 | 1 | 100 | 9.12 | 9.07 | 0.5 |
| | | 379 | 5.41 | 3 | 1 | 100 | 9.08 | | |
| 2614 | 4♀ | 380 | 5.77 | - | 1 | 103 | 2496 | | |
| | | 381 | 5.37 | - | 1 | 103 | 2387 | 2487 | 4.8 |
| | | 382 | 4.91 | - | 1 | 103 | 2579 | | |

HMX solution 4.08 $mg.ml^{-1}$
Internal standard solution 11.828 $mg.ml^{-1}$
QC solution of HMX 3.04 $mg.ml^{-1}$

TABLE 31 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 22.30 | Stand-ards | 383 | 5.65 | 5 | 1 | 100 | | | |
| | | 384 | 5.00 | 5 | 1 | 100 | | | |
| | | 385 | 5.00 | 5 | 1 | 100 | | | |
| 15.56 | QC | 386 | 5.24 | 5 | 1 | 100 | 15.28 | 15.91 | 2.2 |
| | | 387 | 5.91 | 5 | 1 | 100 | 16.30 | | |
| | | 388 | 5.61 | 5 | 1 | 100 | 16.15 | | |
| 4500 | 4♂ | 389 | 5.53 | - | 1 | 105 | 4674 | 4532 | 0.7 |
| | | 390 | 5.89 | - | 1 | 105 | 4195 | | |
| | | 391 | 4.84 | - | 1 | 105 | 4727 | | |
| 5850 | 5♀ | 392 | 5.53 | - | 1 | 105 | 5827 | 6020 | 2.9 |
| | | 393 | 6.38 | - | 1 | 105 | 6132 | | |
| | | 394 | 5.71 | - | 1 | 105 | 6100 | | |

HMX solution 4.46 $mg.ml^{-1}$
Internal standard solution 22.18 $mg.ml^{-1}$
QC solution of HMX 5.19 $mg.ml^{-1}$

TABLE 31 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 31.34 | Standards | 395 | 2.59 | 6 | 1 | 100 | | | |
| | | 396 | 2.64 | 6 | 1 | 100 | | | |
| | | 397 | 2.50 | 6 | 1 | 100 | | | |
| 29.52 | QC | 398 | 2.48 | 6 | 1 | 100 | 28.92 | | |
| | | 399 | 2.26 | 6 | 1 | 100 | 28.84 | 29.05 | 1.7 |
| | | 400 | 2.68 | 6 | 1 | 100 | 29.39 | | |
| 13331 | 5♂ | 401 | 2.72 | - | 1 | 106 | 14306 | | |
| | | 402 | 3.19 | - | 1 | 106 | 13337 | 13775 | 3.3 |
| | | 403 | 3.14 | - | 1 | 106 | 13681 | | |
| 13893 | 6♀ | 404 | 2.70 | - | 1 | 106 | 13455 | | |
| | | 405 | 2.53 | - | 1 | 106 | 13950 | 13607 | 2.1 |
| | | 406 | 2.81 | - | 1 | 106 | 13416 | | |

HMX solution 5.224 $mg.ml^{-1}$
Internal standard solution 30.50 $mg.ml^{-1}$
QC solution of HMX 4.92 $mg.ml^{-1}$

TABLE 31 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 86.04 | Standards | 407 | 2.22 | 17 | 1 | 100 | | | |
| | | 408 | 2.70 | 17 | 1 | 100 | | | |
| | | 409 | 2.40 | 17 | 1 | 100 | | | |
| 85.085 | QC | 410 | 2.65 | 17 | 1 | 100 | 95.78 | | |
| | | 411 | 2.46 | 17 | 1 | 100 | 85.08 | 89.06 | 4.7 |
| | | 412 | 2.18 | 17 | 1 | 100 | 86.33 | | |
| 38000 | 63 | 413 | 2.29 | - | 1 | 117 | 37141 | | |
| | | 414 | 2.37 | - | 1 | 117 | 36315 | 36757 | 3.3 |
| | | 415 | 2.44 | - | 1 | 117 | 36814 | | |

HMX solution 5.061 $mg.ml^{-1}$
Internal standard solution 81.69 $mg.ml^{-1}$
QC solution of HMX 5.005 $mg.ml^{-1}$

TABLE 32

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 26 January 1981 (Week 6)
Date of Analysis: 5 February 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| | Stand- | 441 | 5.34 | 1 | 1 | 50 | | | |
| 2.29 | ards | 442 | 5.96 | 1 | 1 | 50 | | | |
| | | 443 | 6.25 | 1 | 1 | 50 | | | |
| | | 444 | 6.61 | 1 | 1 | 50 | 4.02 | | |
| 3.86 | QC | 445 | 5.57 | 1 | 1 | 50 | 4.03 | 4.02 | 4.1 |
| | | 446 | 5.53 | 1 | 1 | 50 | 4.02 | | |
| | | 447 | 4.68 | - | 1 | 50 | 0 | | |
| 0 | 13♀ | 448 | 5.08 | - | 1 | 50 | 0 | 0 | 0 |
| | | 449 | 5.43 | - | 1 | 50 | 0 | | |
| | | 450 | 5.61 | - | 1 | 50 | 602 | | |
| 659 | 2♂ | 451 | 6.88 | - | 1 | 50 | 593 | 607 | 7.9 |
| | | 452 | 5.59 | - | 1 | 50 | 626 | | |
| | | 453 | 5.50 | - | 1 | 50 | 594 | | * |
| 561 | 2♀ | 454 | 5.94 | - | 1 | 50 | 601 | 598 | 6.6 |
| | | 455 | 6.08 | - | 1 | 50 | | | |

HMX solution 2.29 $mg.ml^{-1}$
Internal standard solution 3.32 $mg.ml^{-1}$
QC solution of HMX 3.86 $mg.ml^{-1}$
* = Mean of 2 results only

TABLE 32 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 7.94 | Stand-ards | 456 | 5.39 | 2 | 1 | 100 | | | |
| | | 457 | 5.38 | 2 | 1 | 100 | | | |
| | | 458 | 5.70 | 2 | 1 | 100 | | | |
| 8.54 | QC | 459 | 5.45 | 2 | 1 | 100 | 8.77 | | |
| | | 460 | 5.38 | 2 | 1 | 100 | 8.81 | 8.85 | 3.6 |
| | | 461 | 5.44 | 2 | 1 | 100 | 8.98 | | |
| 1982 | 3♂ | 462 | 5.00 | - | 1 | 102 | 1783 | | |
| | | 463 | 4.69 | - | 1 | 102 | 1766 | 1811 | 8.6 |
| | | 464 | 6.17 | - | 1 | 102 | 1833 | | |
| 1294 | 3♀ | 465 | 5.31 | - | 1 | 102 | 1269 | | |
| | | 466 | 4.96 | - | 1 | 102 | 1245 | 1272 | 1.7 |
| | | 467 | 5.65 | - | 1 | 102 | 1303 | | |

HMX solution 3.97 $mg.ml^{-1}$
Internal standard solution 9.83 $mg.ml^{-1}$
QC solution of HMX 4.27 $mg.ml^{-1}$

TABLE 32 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 26.43 | Stand-ards | 468 | 6.57 | 5 | 1 | 100 | | | |
| | | 469 | 4.99 | 5 | 1 | 100 | | | |
| | | 470 | 5.07 | 5 | 1 | 100 | | | |
| 27.05 | QC | 471 | 4.42 | 5 | 1 | 100 | 26.98 | | |
| | | 472 | 4.94 | 5 | 1 | 100 | 26.72 | 26.80 | 0.9 |
| | | 473 | 4.95 | 5 | 1 | 100 | 26.71 | | |
| 5732 | 4♂ | 474 | 5.93 | - | 1 | 105 | 5197 | | |
| | | 475 | 4.71 | - | 1 | 105 | 5458 | 5308 | 7.4 |
| | | 476 | 4.97 | - | 1 | 105 | 5268 | | |
| 3076 | 4♀ | 477 | 5.40 | - | 1 | 105 | 3323 | | |
| | | 478 | 6.84 | - | 1 | 105 | 2926 | 3056 | 0.7 |
| | | 479 | 5.18 | - | 1 | 105 | 2919 | | |
| 7027 | 5♀ | 480 | 4.90 | - | 1 | 105 | 7041 | | |
| | | 481 | 5.31 | - | 1 | 105 | 7013 | 7011 | 0.2 |
| | | 482 | 4.97 | - | 1 | 105 | 6978 | | |

HMX solution 5.285 $mg.ml^{-1}$
Internal standard solution 27.29 $mg.ml^{-1}$
QC solution of HMX 5.41 $mg.ml^{-1}$

TABLE 32 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| | Stand- | 483 | 2.60 | 10 | 1 | 90 | | | |
| 42.82 | ards | 484 | 2.86 | 10 | 1 | 90 | | | |
| | | 485 | 2.90 | 10 | 1 | 90 | | | |
| | | 486 | 2.80 | 10 | 1 | 90 | 45.63 | | |
| 43.82 | QC | 487 | 2.66 | 10 | 1 | 90 | 44.84 | 45.10 | 2.9 |
| | | 488 | 2.38 | 10 | 1 | 90 | 44.82 | | |
| | | 489 | 3.27 | - | 1 | 100 | 14939 | | + |
| 17441 | 5♂ | 490 | 2.20 | - | 1 | 100 | 14159 | 14426 | 17.3 |
| | | 491 | 2.51 | - | 1 | 100 | 14179 | | |
| | | 492 | 3.35 | - | 1 | 100 | 14178 | | |
| 16565 | 6♀ | 493 | 2.91 | - | 1 | 100 | 15900 | 15033 | 9.2 |
| | | 494 | 3.23 | - | 1 | 100 | 15020 | | |

HMX solution 4.282 $mg.ml^{-1}$

Internal standard solution 41.9 $mg.ml^{-1}$

QC solution of HMX 4.382 $mg.ml^{-1}$

+ = Repeated using different standard solution of HMX (see p. 77)

TABLE 32 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 127.28 | Standards | 495 | 2.83 | 25 | 1 | 75 | | | |
| | | 496 | 2.22 | 25 | 1 | 75 | | | |
| | | 497 | 2.25 | 25 | 1 | 75 | | | |
| 125.45 | QC | 498 | 2.24 | 25 | 1 | 75 | 125.01 | 124.71 | 0.6 |
| | | 499 | 2.76 | 25 | 1 | 75 | 123.91 | | |
| | | 500 | 3.09 | 25 | 1 | 75 | 125.20 | | |
| 49325 | 63 | 501 | 2.15 | - | 1 | 100 | 45065 | 46616 | 5.5 |
| | | 502 | 2.06 | - | 1 | 100 | 47071 | | |
| | | 503 | 2.58 | - | 1 | 100 | 47713 | | |

HMX solution 5.09 $mg.ml^{-1}$
Internal standard solution 121.3 $mg.ml^{-1}$
QC solution of HMX 5.02 $mg.ml^{-1}$

TABLE 32 (continued)

Repeat Analyses

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 50.8 | Stand-ards | 483R | 2.50 | 10 | 1 | 90 | | | |
| | | 484R | 2.50 | 10 | 1 | 90 | | | |
| | | 485R | 2.50 | 10 | 1 | 90 | | | |
| 17441 | 5δ | 489R | 3.32 | - | 1 | 100 | 16129 | 16966 | 2.7 |
| | | 490R | 3.25 | - | 1 | 100 | 18000 | | |
| | | 491R | 3.41 | - | 1 | 100 | 16769 | | |

HMX solution 5.08 $mg.ml^{-1}$
Internal standard solution 48.86 $mg.ml^{-1}$

TABLE 33

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 16 February 1981 (Week 9)
Date of Analysis: 19 February 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 3.23 | Standards | 504 | 4.88 | 1 | 1 | 50 | | | |
| | | 505 | 5.88 | 1 | 1 | 50 | | | |
| | | 506 | 4.94 | 1 | 1 | 50 | | | |
| 3.40 | QC | 507 | 4.94 | 1 | 1 | 50 | 3.45 | | |
| | | 508 | 5.44 | 1 | 1 | 50 | 3.49 | 3.47 | 2.1 |
| | | 509 | 5.99 | 1 | 1 | 50 | 3.47 | | |
| 0 | 1♂♀ | 510 | 5.92 | - | 1 | 50 | 0 | | |
| | | 511 | 4.92 | - | 1 | 50 | 0 | 0 | 0 |
| | | 512 | 4.76 | - | 1 | 50 | 0 | | |
| 715 | 2♂ | 513 | 5.12 | - | 1 | 50 | 704 | | |
| | | 514 | 6.00 | - | 1 | 50 | 718 | 698 | 2.4 |
| | | 515 | 5.05 | - | 1 | 50 | 672 | | |
| 606 | 2♀ | 516 | 5.41 | - | 1 | 50 | 651 | | |
| | | 517 | 5.90 | - | 1 | 50 | 513 | 590 | 2.6 |
| | | 518 | 5.85 | - | 1 | 50 | 606 | | |

HMX solution 3.23 $mg.ml^{-1}$
Internal standard solution 2.93 $mg.ml^{-1}$
QC solution of HMX 3.40 $mg.ml^{-1}$

TABLE 33 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 7.86 | Stand-ards | 519 | 5.40 | 2 | 1 | 100 | | | |
| | | 520 | 5.32 | 2 | 1 | 100 | | | |
| | | 521 | 5.67 | 2 | 1 | 100 | | | |
| 9.04 | QC | 522 | 5.47 | 2 | 1 | 100 | 8.80 | | |
| | | 523 | 6.00 | 2 | 1 | 100 | 8.93 | 8.87 | 1.9 |
| | | 524 | 5.28 | 2 | 1 | 100 | 8.88 | | |
| 2158 | 3♂ | 525 | 5.72 | - | 1 | 100 | 2107 | | |
| | | 526 | 5.68 | - | 1 | 100 | 2117 | 2115 | 2.0 |
| | | 527 | 5.44 | - | 1 | 100 | 2121 | | |
| 1377 | 3♀ | 528 | 5.30 | - | 1 | 100 | 1303 | | |
| | | 529 | 5.02 | - | 1 | 100 | 1343 | 1345 | 2.3 |
| | | 530 | 6.24 | - | 1 | 100 | 1390 | | |

HMX solution 3.93 $mg.ml^{-1}$
Internal standard solution 8.09 $mg.ml^{-1}$
QC solution of HMX 4.52 $mg.ml^{-1}$

TABLE 33 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 26.03 | Stand-ards | 531 | 4.95 | 5 | 1 | 100 | | | |
| | | 532 | 6.22 | 5 | 1 | 100 | | | |
| | | 533 | 5.21 | 5 | 1 | 100 | | | |
| 30.20 | QC | 534 | 5.90 | 5 | 1 | 100 | 30.41 | | |
| | | 535 | 5.31 | 5 | 1 | 100 | 30.10 | 30.37 | 0.6 |
| | | 536 | 6.00 | 5 | 1 | 100 | 30.60 | | |
| 6440 | 4♂ | 537 | 5.50 | - | 1 | 105 | 6083 | | |
| | | 538 | 5.28 | - | 1 | 105 | 6574 | 6268 | 2.7 |
| | | 539 | 5.13 | - | 1 | 105 | 6146 | | |
| 3348 | 4♀ | 540 | 5.07 | - | 1 | 105 | 3301 | | |
| | | 541 | 5.43 | - | 1 | 105 | 3263 | 3297 | 1.5 |
| | | 542 | 5.57 | - | 1 | 105 | 3326 | | |
| 7372 | 5♀ | 543 | 5.42 | - | 1 | 105 | 7292 | | |
| | | 544 | 5.93 | - | 1 | 105 | 7179 | 7295 | 1.0 |
| | | 545 | 5.46 | - | 1 | 105 | 7413 | | |

HMX solution 5.205 $mg.ml^{-1}$
Internal standard solution 30.225 $mg.ml^{-1}$
QC solution of HMX 6.04 $mg.ml^{-1}$

TABLE 33 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 49.16 | Standards | 546 | 2.69 | 10 | 1 | 100 | | | |
| | | 547 | 2.45 | 10 | 1 | 100 | | | |
| | | 548 | 2.95 | 10 | 1 | 100 | | | |
| 48.92 | QC | 549 | 2.88 | 10 | 1 | 100 | 48.92 | | |
| | | 550 | 2.97 | 10 | 1 | 100 | 49.68 | 49.00 | 0.2 |
| | | 551 | 2.90 | 10 | 1 | 100 | 48.41 | | |
| 19105 | 5♂ | 552 | 2.61 | - | 1 | 110 | 15202<br>15754 | | |
| | | 553 | 2.78 | - | 1 | 110 | 15776<br>16788 | 16582 | 13.2 |
| | | 554 | 2.50 | - | 1 | 110 | 17796<br>18178 | | |
| 18289 | 6♀ | 555 | 2.66 | - | 1 | 110 | 17900 | | |
| | | 556 | 3.16 | - | 1 | 110 | 17367 | 17463 | 4.5 |
| | | 557 | 2.71 | - | 1 | 110 | 17121 | | |

HMX solution 4.916 $mg.ml^{-1}$
Internal standard solution 47.83 $mg.ml^{-1}$
QC solution of HMX 4.892 $mg.ml^{-1}$

TABLE 33 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 139.325 | Standards | 558 | 3.10 | 25 | 1 | 75 | | | |
| | | 559 | 2.36 | 25 | 1 | 75 | | | |
| | | 560 | 2.44 | 25 | 1 | 75 | | | |
| 137.25 | QC | 561 | 2.84 | 25 | 1 | 75 | 133.72 | 135.50 | 1.3 |
| | | 562 | 2.61 | 25 | 1 | 75 | 134.47 | | |
| | | 563 | 3.50 | 25 | 1 | 75 | 138.32 | | |
| 53864 | 6♂ | 564 | 2.72 | - | 1 | 100 | 57551 | 52468 | 2.6 |
| | | 565 | 2.50 | - | 1 | 100 | 48764 | | |
| | | 566 | 2.53 | - | 1 | 100 | 51089 | | |

HMX solution 5.573 $mg.ml^{-1}$
Internal standard solution 133.62 $mg.ml^{-1}$
QC solution of HMX 5.49 $mg.ml^{-1}$

TABLE 34

Analysis of HMX In Diets During 90 Day Toxicity Testing In Rats

Date of Mixing: 9 March (termination)
Date of Analysis: 20 March 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 3.90 | Stand-ards | 567 | 5.06 | 1 | 1 | 50 | | | |
| | | 568 | 5.06 | 1 | 1 | 50 | | | |
| | | 569 | 5.02 | 1 | 1 | 50 | | | |
| 3.44 | QC | 570 | 5.15 | 1 | 1 | 50 | 3.53 | | |
| | | 571 | 4.94 | 1 | 1 | 50 | 3.45 | 3.52 | 2.3 |
| | | 572 | 5.38 | 1 | 1 | 50 | 3.58 | | |
| 0 | 1♂♀ | 573 | 5.20 | - | 1 | 50 | 0 | | |
| | | 574 | 4.80 | - | 1 | 50 | 0 | 0 | 0 |
| | | 575 | 4.86 | - | 1 | 50 | 0 | | |
| 729 | 2♂ | 576 | 6.75 | - | 1 | 50 | 705 | | |
| | | 577 | 5.15 | - | 1 | 50 | 698 | 716 | 1.8 |
| | | 578 | 5.89 | - | 1 | 50 | 746 | | |
| 618 | 2♀ | 579 | 5.21 | - | 1 | 50 | 665 | | |
| | | 580 | 4.53 | - | 1 | 50 | 663 | 661 | 7.0 |
| | | 581 | 5.85 | - | 1 | 50 | 655 | | |

HMX solution 3.90 $mg.ml^{-1}$
Internal standard solution 3.86 $mg.ml^{-1}$
QC solution of HMX 3.44 $mg.ml^{-1}$

TABLE 34 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 10.38 | Stand-ards | 582 | 5.00 | 2 | 1 | 100 | | | |
| | | 583 | 5.00 | 2 | 1 | 100 | | | |
| | | 584 | 5.00 | 2 | 1 | 100 | | | |
| 20.40 | QC | 585 | 5.00 | 2 | 1 | 100 | 19.72 | 19.57 | 4.1 |
| | | 586 | 5.00 | 2 | 1 | 100 | 19.69 | | |
| | | 587 | 5.00 | 2 | 1 | 100 | 19.31 | | |
| 2214 | 3♂ | 588 | 6.44 | - | 1 | 100 | 2095 | 2157 | 2.6 |
| | | 589 | 5.68 | - | 1 | 100 | 2178 | | |
| | | 590 | 5.62 | - | 1 | 100 | 2199 | | |
| 1373 | 3♀ | 591 | 4.72 | - | 1 | 100 | 1367 | 1350 | 2.0 |
| | | 592 | 4.52 | - | 1 | 100 | 1303 | | |
| | | 593 | 5.87 | - | 1 | 100 | 1379 | | |

HMX solution 5.19 $mg.ml^{-1}$
Internal standard solution 10.35 $mg.ml^{-1}$
QC solution of HMX 10.20 $mg.ml^{-1}$

TABLE 34 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 29.58 | Stand-ards | 594 | 5.00 | 5 | 1 | 100 | | | |
| | | 595 | 5.00 | 5 | 1 | 100 | | | |
| | | 596 | 5.00 | 5 | 1 | 100 | | | |
| 31.63 | QC | 597 | 5.00 | 5 | 1 | 100 | 31.58 | | |
| | | 598 | 5.00 | 5 | 1 | 100 | 31.86 | 31.74 | 0.3 |
| | | 599 | 5.00 | 5 | 1 | 100 | 31.79 | | |
| 6385 | 4♂ | 600 | 6.32 | - | 1 | 105 | 6356 | | |
| | | 601 | 5.65 | - | 1 | 105 | 6642 | 6437 | 0.8 |
| | | 602 | 5.84 | - | 1 | 105 | 6313 | | |
| 3499 | 4♀ | 603 | 5.82 | - | 1 | 105 | 3550 | | |
| | | 604 | 5.37 | - | 1 | 105 | 3547 | 3553 | 1.5 |
| | | 605 | 4.91 | - | 1 | 105 | 3561 | | |
| 7678 | 5♀ | 606 | 4.81 | - | 1 | 105 | 7714 | | |
| | | 607 | 5.18 | - | 1 | 105 | 7489 | 7617 | 0.8 |
| | | 608 | 5.21 | - | 1 | 105 | 7647 | | |

HMX solution 5.915 $mg.ml^{-1}$
Internal standard solution 31.63 $mg.ml^{-1}$
QC solution of HMX 6.325 $mg.ml^{-1}$

TABLE 34 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 44.9 | Stand-ards | 609 | 2.50 | 10 | 1 | 100 | | | |
| | | 610 | 2.50 | 10 | 1 | 100 | | | |
| | | 611 | 2.50 | 10 | 1 | 100 | | | |
| 43.6 | QC | 612 | 2.50 | 10 | 1 | 100 | 43.82 | | |
| | | 613 | 2.50 | 10 | 1 | 100 | 43.87 | 43.89 | 0.7 |
| | | 614 | 2.50 | 10 | 1 | 100 | 43.98 | | |
| 18942 | 5♂ | 615 | 2.52 | - | 1 | 110 | 18833 | | |
| | | 616 | 2.30 | - | 1 | 110 | 17537 | 18744 | 1.0 |
| | | 617 | 2.46 | - | 1 | 110 | 19863 | | |
| 17520 | 6♀ | 618 | 2.76 | - | 1 | 110 | 17576 | | |
| | | 619 | 3.27 | - | 1 | 110 | 17794 | 17730 | 1.2 |
| | | 620 | 2.36 | - | 1 | 110 | 17819 | | |

HMX solution 4.49 $mg.ml^{-1}$
Internal standard solution 51.27 $mg.ml^{-1}$
QC solution of HMX 4.36 $mg.ml^{-1}$

TABLE 34 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 142.0 | Stand-ards | 621 | 2.27 | 25 | 1 | 75 | | | |
| | | 622 | 2.50 | 25 | 1 | 75 | | | |
| | | 623 | 2.50 | 25 | 1 | 75 | | | |
| 155.4 | QC | 624 | 2.50 | 25 | 1 | 75 | 153.1 | | |
| | | 625 | 2.50 | 25 | 1 | 75 | 154.2 | 153.5 | 1.2 |
| | | 626 | 2.50 | 25 | 1 | 75 | 153.3 | | |
| 54105 | ♂ | 627 | 2.27 | - | 1 | 100 | 55136 | | |
| | | 628 | 2.35 | - | 1 | 100 | 55265 | 55405 | 2.4 |
| | | 629 | 2.71 | - | 1 | 100 | 55815 | | |

HMX solution 5.68 $mg.ml^{-1}$
Internal standard solution 129.0 $mg.ml^{-1}$
QC solution of HMX 6.216 $mg.ml^{-1}$

TABLE 35

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 16 January 1981 (commencement)
Date of Analysis: 18 February 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.28 | Standards | 1 | 5.27 | 1 | 1 | 50 | | | |
| | | 2 | 5.50 | 1 | 1 | 50 | | | |
| | | 3 | 5.54 | 1 | 1 | 50 | | | |
| 0.24 | QC | 4 | 4.62 | 1 | 1 | 50 | 0.36 | | |
| | | 5 | 5.43 | 1 | 1 | 50 | 0.30 | 0.30 | 24.8 |
| | | 6 | 5.12 | 1 | 1 | 50 | 0.25 | | |
| 0 | 1 + 7 ♂♀ | 7 | 6.94 | - | 1 | 50 | 0 | | |
| | | 8 | 5.40 | - | 1 | 50 | 0 | 0 | 0 |
| | | 9 | 5.12 | - | 1 | 50 | 0 | | |
| 14 | 2♂ | 10 | 5.00 | - | 1 | 50 | 14 | | |
| | | 11 | 5.59 | - | 1 | 50 | 14 | 14 | 0 |
| | | 12 | 5.75 | - | 1 | 50 | 14 | | |

HMX solution 0.28 $mg.ml^{-1}$
Internal standard solution 0.31 $mg.ml^{-1}$
QC solution of HMX 0.24 $mg.ml^{-1}$

TABLE 35 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 23 | 2♀ | 13R | 5.93 | - | 1 | 50 | 32 | 28 | 21.7 |
| | | 14R | 5.57 | - | 1 | 50 | 28 | | |
| | | 15R | 4.99 | - | 1 | 50 | 24 | | |
| 35 | 3♂ | 16 | 5.76 | - | 1 | 50 | 35 | 32 | 8.6 |
| | | 17 | 5.47 | - | 1 | 50 | 30 | | |
| | | 18 | 6.53 | - | 1 | 50 | 30 | | |
| 68 | 3♀ | 19 | 5.25 | - | 1 | 50 | 61 | 62 | 8.8 |
| | | 20 | 5.62 | - | 1 | 50 | 62 | | |
| | | 21 | 5.15 | - | 1 | 50 | 63 | | |
| 84 | 4♂ | 22 | 5.16 | - | 1 | 50 | 76 | 79 | 6.0 |
| | | 23 | 6.53 | - | 1 | 50 | 83 | | |
| | | 24 | 4.67 | - | 1 | 50 | 77 | | |

HMX solution 0.28 $mg.ml^{-1}$
Internal standard solution 0.31 $mg.ml^{-1}$
QC solution of HMX 0.24 $mg.ml^{-1}$

TABLE 35 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 2.83 | Standards | 25 | 5.51 | 1 | 1 | 50 | | | |
| | | 26 | 4.92 | 1 | 1 | 50 | | | |
| | | 27 | 5.46 | 1 | 1 | 50 | | | |
| 2.73 | QC | 28 | 4.77 | 1 | 1 | 50 | 2.88 | 2.87 | 5.1 |
| | | 29 | 5.49 | 1 | 1 | 50 | 2.83 | | |
| | | 30 | 4.87 | 1 | 1 | 50 | 2.89 | | |
| 204 | 4♀ | 31 | 5.40 | - | 1 | 50 | 188 | 195 | 4.4 |
| | | 32 | 5.22 | - | 1 | 50 | 196 | | |
| | | 33 | 5.36 | - | 1 | 50 | 202 | | |

HMX solution 2.83 $mg.ml^{-1}$
Internal standard solution 2.06 $mg.ml^{-1}$
QC solution of HMX 2.73 $mg.ml^{-1}$

AD-A171 595 HMX: ANALYSIS OF DOSING FORMULATIONS USED IN ACUTE SUB-ACUTE AND SUB-CHRO..(U) INVERESK RESEARCH INTERNATIONAL LTD MUSSELBURGH (SCOTLAND) M S HENDERSON 2/2

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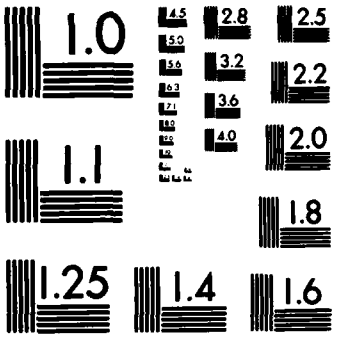


TABLE 35 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 225 | 5♂ | 34 | 4.79 | - | 1 | 50 | 222 | 231 | 2.7 |
| | | 35 | 4.64 | - | 1 | 50 | 223 | | |
| | | 36 | 5.73 | - | 1 | 50 | 250 | | |
| 578 | 5♀ | 37 | 5.01 | - | 1 | 50 | 575 | 595 | 2.9 |
| | | 38 | 4.90 | - | 1 | 50 | 594 | | |
| | | 39 | 5.52 | - | 1 | 50 | 616 | | |
| 662 | 6♂ | 40 | 5.19 | - | 1 | 50 | 664 | 657 | 0.8 |
| | | 41 | 4.92 | - | 1 | 50 | 648 | | |
| | | 42 | 5.18 | - | 1 | 50 | 659 | | |

HMX solution 2.83 $mg.ml^{-1}$
Internal standard solution 2.06 $mg.ml^{-1}$
QC solution of HMX 2.73 $mg.ml^{-1}$

TABLE 35 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 9.22 | Standards | 43 | 4.47 | 2 | 1 | 50 | | | |
| | | 44 | 4.78 | 2 | 1 | 50 | | | |
| | | 45 | 4.82 | 2 | 1 | 50 | | | |
| 11.32 | QC | 46 | 5.21 | 2 | 1 | 50 | 11.75 | | |
| | | 47 | 4.55 | 2 | 1 | 50 | 11.77 | 11.79 | 4.2 |
| | | 48 | 5.54 | 2 | 1 | 50 | 11.84 | | |
| 2146 | 6♀ | 49 | 5.11 | - | 1 | 52 | 2138 | | |
| | | 50 | 6.14 | - | 1 | 52 | 2063 | 2109 | 1.7 |
| | | 51 | 5.38 | - | 1 | 52 | 2125 | | |

HMX solution 4.61 $mg.ml^{-1}$
Internal standard solution 22.17 $mg.ml^{-1}$
QC solution of HMX 5.66 $mg.ml^{-1}$

TABLE 36

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 23 January 1981 (Week 1)
Date of Analysis: 28 January 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.38 | Standards | 52 | 5.43 | 1 | 1 | 50 | | | |
| | | 53 | 5.50 | 1 | 1 | 50 | | | |
| | | 54 | 5.14 | 1 | 1 | 50 | | | |
| 0.28 | QC | 55 | 5.42 | 1 | 1 | 50 | 0.28 | | |
| | | 56 | 5.89 | 1 | 1 | 50 | 0.29 | 0.29 | 3.6 |
| | | 57 | 5.97 | 1 | 1 | 50 | 0.29 | | |
| 0 | 1 + 7 ♂♀ | 58 | 5.75 | - | 1 | 50 | 0 | | |
| | | 59 | 5.39 | - | 1 | 50 | 0 | 0 | 0 |
| | | 60 | 5.20 | - | 1 | 50 | 0 | | |
| 16 | 2♂ | 61 | 5.96 | - | 1 | 50 | 15 | | |
| | | 62 | 4.83 | - | 1 | 50 | 17 | 16 | 0 |
| | | 63 | 5.52 | - | 1 | 50 | 16 | | |

HMX solution 0.38 $mg.ml^{-1}$
Internal standard solution 0.32 $mg.ml^{-1}$
QC solution of HMX 0.28 $mg.ml^{-1}$

TABLE 36 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 25 | 2♀ | 64 | 5.17 | - | 1 | 50 | 26 | 25 | 0 |
| | | 65 | 5.87 | - | 1 | 50 | 27 | | |
| | | 66 | 4.73 | - | 1 | 50 | 23 | | |
| 39 | 3♂ | 67 | 5.01 | - | 1 | 50 | 41 | 39 | 0 |
| | | 68 | 5.26 | - | 1 | 50 | 37 | | |
| | | 69 | 5.34 | - | 1 | 50 | 38 | | |
| 78 | 3♀ | 70 | 4.29 | - | 1 | 50 | 80 | 79 | 1 |
| | | 71 | 4.72 | - | 1 | 50 | 78 | | |
| | | 72 | 5.30 | - | 1 | 50 | 78 | | |
| 113 | 4♂ | 73 | 5.07 | - | 1 | 50 | 112 | 113 | 0 |
| | | 74 | 5.25 | - | 1 | 50 | 114 | | |
| | | 75 | 4.75 | - | 1 | 50 | 113 | | |

HMX solution 0.38 $mg.ml^{-1}$
Internal standard solution 0.32 $mg.ml^{-1}$
QC solution of HMX 0.28 $mg.ml^{-1}$

TABLE 36 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 2.71 | Standards | 76 | 4.47 | 1 | 1 | 50 | | | |
| | | 77 | 5.52 | 1 | 1 | 50 | | | |
| | | 78 | 4.89 | 1 | 1 | 50 | | | |
| 3.10 | QC | 79 | 4.89 | 1 | 1 | 50 | 3.18 | | |
| | | 80 | 5.44 | 1 | 1 | 50 | 3.17 | 3.19 | 2.9 |
| | | 81 | 5.84 | 1 | 1 | 50 | 3.21 | | |
| 201 | 4♀ | 82 | 4.79 | - | 1 | 50 | 208 | | |
| | | 83 | 5.33 | - | 1 | 50 | 204 | 205 | 2.0 |
| | | 84 | 5.28 | - | 1 | 50 | 203 | | |

HMX solution 2.71 $mg.ml^{-1}$
Internal standard solution 2.25 $mg.ml^{-1}$
QC solution of HMX 3.10 $mg.ml^{-1}$

TABLE 36 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 257 | 5♂ | 85 | 4.71 | - | 1 | 50 | 285 | 274 | 6.6 |
| | | 86 | 5.21 | - | 1 | 50 | 263 | | |
| | | 87 | 4.94 | - | 1 | 50 | 274 | | |
| 575 | 5♀ | 88 | 5.16 | - | 1 | 50 | 577 | 569 | 1.0 |
| | | 89 | 6.88 | - | 1 | 50 | 587 | | |
| | | 90 | 5.89 | - | 1 | 50 | 543 | | |
| 688 | 6♂ | 91 | 5.00 | - | 1 | 50 | 716 | 708 | 2.9 |
| | | 92 | 4.58 | - | 1 | 50 | 701 | | |
| | | 93 | 4.92 | - | 1 | 50 | 708 | | |

HMX solution 2.71 $mg.ml^{-1}$
Internal standard solution 2.25 $mg.ml^{-1}$
QC solution of HMX 3.10 $mg.ml^{-1}$

TABLE 36 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 10.56 | Stand-ards | 94 | 5.32 | 2 | 1 | 50 | | | |
| | | 95 | 5.80 | 2 | 1 | 50 | | | |
| | | 96 | 6.08 | 2 | 1 | 50 | | | |
| 12.16 | QC | 97 | 4.90 | 2 | 1 | 50 | 12.17 | 12.24 | 0.7 |
| | | 98 | 5.37 | 2 | 1 | 50 | 12.32 | | |
| | | 99 | 5.60 | 2 | 1 | 50 | 12.24 | | |
| 1753 | 6♀ | 100 | 5.20 | - | 1 | 52 | 1729 | 1726 | 1.5 |
| | | 101 | 4.76 | - | 1 | 52 | 1712 | | |
| | | 102 | 5.38 | - | 1 | 52 | 1736 | | |

HMX solution 5.28 $mg.ml^{-1}$
Internal standard solution 10.36 $mg.ml^{-1}$
QC solution of HMX 6.08 $mg.ml^{-1}$

TABLE 37

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 30 January 1981 (Week 2)
Date of Analysis: 17 February 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.37 | Standards | 103 | 4.75 | 1 | 1 | 50 | | | |
| | | 104 | 5.25 | 1 | 1 | 50 | | | |
| | | 105 | 5.63 | 1 | 1 | 50 | | | |
| 0.32 | QC | 106 | 5.49 | 1 | 1 | 50 | 0.34 | | |
| | | 107 | 4.95 | 1 | 1 | 50 | 0.32 | 0.33 | 3.1 |
| | | 108 | 4.80 | 1 | 1 | 50 | 0.32 | | |
| 0 | 1 + 7 ♂♀ | 109 | 5.55 | - | 1 | 50 | 0 | | |
| | | 110 | 5.66 | - | 1 | 50 | 0 | 0 | 0 |
| | | 111 | 5.05 | - | 1 | 50 | 0 | | |
| 15 | 2♂ | 112 | 5.01 | - | 1 | 50 | 18 | | |
| | | 113 | 5.97 | - | 1 | 50 | 39 | Analysis Repeated | |
| | | 114 | 4.90 | - | 1 | 50 | 18 | (see p. 103) | |

HMX solution 0.37 $mg.ml^{-1}$
Internal standard solution 0.37 $mg.ml^{-1}$
QC solution of HMX 0.32 $mg.ml^{-1}$

TABLE 37 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 26 | 2♀ | 115 | 6.86 | - | 1 | 50 | 26 | 28 | 7.7 |
| | | 116 | 5.20 | - | 1 | 50 | 31 | | |
| | | 117 | 6.19 | - | 1 | 50 | 28 | | |
| 38 | 3♂ | 118 | 5.47 | - | 1 | 50 | 38 | 39 | 2.6 |
| | | 119 | 5.46 | - | 1 | 50 | 39 | | |
| | | 120 | 4.44 | - | 1 | 50 | 40 | | |
| 90 | 3♀ | 121 | 5.71 | - | 1 | 50 | 96 | 92 | 2.2 |
| | | 122 | 5.10 | - | 1 | 50 | 86 | | |
| | | 123 | 5.36 | - | 1 | 50 | 93 | | |
| 105 | 4♂ | 124 | 6.14 | - | 1 | 50 | 103 | 102 | 2.9 |
| | | 125 | 4.76 | - | 1 | 50 | 100 | | |
| | | 126 | 5.27 | - | 1 | 50 | 103 | | |

HMX solution 0.37 $mg.ml^{-1}$
Internal standard solution 0.37 $mg.ml^{-1}$
QC solution of HMX 0.32 $mg.ml^{-1}$

TABLE 37 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 2.48 | Stand-ards | 127 | 4.90 | 1 | 1 | 50 | | | |
| | | 128 | 4.87 | 1 | 1 | 50 | | | |
| | | 129 | 4.82 | 1 | 1 | 50 | | | |
| 2.76 | QC | 130 | 5.10 | 1 | 1 | 50 | 2.73 | 2.78 | 0.7 |
| | | 131 | 4.87 | 1 | 1 | 50 | 2.80 | | |
| | | 132 | 5.65 | 1 | 1 | 50 | 2.80 | | |
| 258 | 4♀ | 133 | 5.24 | - | 1 | 50 | 249 | 257 | 0.4 |
| | | 134 | 5.49 | - | 1 | 50 | 271 | | |
| | | 135 | 4.79 | - | 1 | 50 | 252 | | |

HMX solution 2.48 $mg.ml^{-1}$
Internal standard solution 2.57 $mg.ml^{-1}$
QC solution of HMX 2.76 $mg.ml^{-1}$

TABLE 37 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 280 | 5♂ | 136 | 5.34 | - | 1 | 50 | 277 | 276 | 1.4 |
| | | 137 | 4.85 | - | 1 | 50 | 278 | | |
| | | 138 | 4.92 | - | 1 | 50 | 273 | | |
| 754 | 5♀ | 139 | 5.63 | - | 1 | 50 | 744 | 750 | 0.5 |
| | | 140 | 5.48 | - | 1 | 50 | 742 | | |
| | | 141 | 5.11 | - | 1 | 50 | 764 | | |
| 756 | 6♂ | 142 | 4.80 | - | 1 | 50 | 767 | 754 | 0.5 |
| | | 143 | 6.30 | - | 1 | 50 | 761 | | |
| | | 144 | 6.05 | - | 1 | 50 | 735 | | |

HMX solution 2.48 $mg.ml^{-1}$
Internal standard solution 2.57 $mg.ml^{-1}$
QC solution of HMX 2.76 $mg.ml^{-1}$

TABLE 37 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 11.70 | Stand-ards | 145 | 5.92 | 3 | 1 | 50 | | | |
| | | 146 | 4.56 | 3 | 1 | 50 | | | |
| | | 147 | 5.96 | 3 | 1 | 50 | | | |
| 15.09 | QC | 148 | 6.38 | 3 | 1 | 50 | 15.06 | 14.89 | 1.3 |
| | | 149 | 5.61 | 3 | 1 | 50 | 14.50 | | |
| | | 150 | 5.70 | 3 | 1 | 50 | 15.12 | | |
| 2650 | 6♀ | 151 | 5.39 | - | 1 | 53 | 2264 | Analysis Repeated (see p. 103) | |
| | | 152 | 4.73 | - | 1 | 53 | 2354 | | |
| | | 153 | 5.75 | - | 1 | 53 | 2434 | | |

HMX solution 3.90 mg.ml$^{-1}$
Internal standard solution 12.05 mg.ml$^{-1}$
QC solution of HMX 5.03 mg.ml$^{-1}$

TABLE 37 (continued)

Repeat Analyses

| Nominal Value | Group | Analytical Sample No. | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|
| 15 | 2♂ | 112R<br>113R<br>114R | 14<br>15<br>14 | 14 | 6.7 |
| 2650 | 6♀ | 151R<br>152R<br>153R | 2884<br>2811<br>2647 | 2781 | 4.9 |

TABLE 38

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 6 February 1981 (Week 3)
Date of Analysis: 17 February 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.125 | Standards | 154 | 5.27 | 1 | 1 | 50 | | | |
| | | 155 | 6.38 | 1 | 1 | 50 | | | |
| | | 156 | 5.38 | 1 | 1 | 50 | | | |
| 0.130 | QC | 157 | 5.38 | 1 | 1 | 50 | 0.125 | | |
| | | 158 | 5.43 | 1 | 1 | 50 | 0.127 | 0.126 | 3.1 |
| | | 159 | 5.32 | 1 | 1 | 50 | 0.126 | | |
| 0 | 1 + 7 ♂♀ | 160 | 7.02 | - | 1 | 50 | 0 | | |
| | | 161 | 5.08 | - | 1 | 50 | 0 | 0 | 0 |
| | | 162 | 5.05 | - | 1 | 50 | 0 | | |

HMX solution 0.125 $mg.ml^{-1}$
Internal standard solution 0.17 $mg.ml^{-1}$
QC solution of HMX 0.130 $mg.ml^{-1}$

TABLE 38 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 16 | 2♂ | 163 | 5.55 | - | 1 | 50 | 16 | 17 | 6.3 |
| | | 164 | 5.23 | - | 1 | 50 | 18 | | |
| | | 165 | 5.16 | - | 1 | 50 | 16 | | |
| 27 | 2♀ | 166 | 5.14 | - | 1 | 50 | 26 | 25 | 7.4 |
| | | 167 | 5.38 | - | 1 | 50 | 26 | | |
| | | 168 | 5.90 | - | 1 | 50 | 23 | | |
| 37 | 3♂ | 169 | 5.50 | - | 1 | 50 | 37 | 37 | 0 |
| | | 170 | 6.23 | - | 1 | 50 | 38 | | |
| | | 171 | 5.33 | - | 1 | 50 | 35 | | |

HMX solution 0.125 $mg.ml^{-1}$
Internal standard solution 0.17 $mg.ml^{-1}$
QC solution of HMX 0.130 $mg.ml^{-1}$

TABLE 38 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.48 | Stand-ards | 172 | 6.67 | 1 | 1 | 50 | | | |
| | | 173 | 5.33 | 1 | 1 | 50 | | | |
| | | 174 | 6.10 | 1 | 1 | 50 | | | |
| 0.51 | QC | 175 | 5.70 | 1 | 1 | 50 | 0.548 | | |
| | | 176 | 5.41 | 1 | 1 | 50 | 0.548 | 0.546 | 7.1 |
| | | 177 | 5.15 | 1 | 1 | 50 | 0.543 | | |
| 85 | 3♀ | 178 | 6.16 | - | 1 | 50 | 92 | | |
| | | 179 | 5.64 | - | 1 | 50 | 85 | 87 | 2.3 |
| | | 180 | 5.44 | - | 1 | 50 | 84 | | |
| 102 | 4♂ | 181 | 5.91 | - | 1 | 50 | 110 | | |
| | | 182 | 6.32 | - | 1 | 50 | 111 | 111 | 8.8 |
| | | 183 | 5.26 | - | 1 | 50 | 111 | | |

HMX solution 0.48 $mg.ml^{-1}$

*Internal standard solution* 0.46 $mg.ml^{-1}$

QC solution of HMX 0.51 $mg.ml^{-1}$

TABLE 38 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1.46 | Stand-ards | 184 | 5.12 | 1 | 1 | 50 | | | |
| | | 185 | 5.93 | 1 | 1 | 50 | | | |
| | | 186 | 6.55 | 1 | 1 | 50 | | | |
| 1.650 | QC | 187 | 5.44 | 1 | 1 | 50 | 1.669 | | |
| | | 188 | 6.17 | 1 | 1 | 50 | 1.675 | 1.684 | 2.1 |
| | | 189 | 6.82 | 1 | 1 | 50 | 1.707 | | |
| 247 | 4♀ | 190 | 5.26 | - | 1 | 50 | 237 | | |
| | | 191 | 5.23 | - | 1 | 50 | 247 | 243 | 1.6 |
| | | 192 | 5.40 | - | 1 | 50 | 245 | | |
| 282 | 5♂ | 193 | 5.69 | - | 1 | 50 | 267 | | |
| | | 194 | 6.04 | - | 1 | 50 | 263 | 270 | 4.3 |
| | | 195 | 5.16 | - | 1 | 50 | 281 | | |

HMX solution 1.46 $mg.ml^{-1}$
Internal standard solution 1.60 $mg.ml^{-1}$
QC solution of HMX 1.650 $mg.ml^{-1}$

TABLE 38 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 3.67 | Stand-ards | 196 | 5.89 | 1 | 1 | 50 | | | |
| | | 197 | 5.21 | 1 | 1 | 50 | | | |
| | | 198 | 5.68 | 1 | 1 | 50 | | | |
| 3.630 | QC | 199 | 4.99 | 1 | 1 | 50 | 3.614 | 3.625 | 0.1 |
| | | 200 | 5.80 | 1 | 1 | 50 | 3.576 | | |
| | | 201 | 5.64 | 1 | 1 | 50 | 3.684 | | |
| 642 | 5♀ | 202 | 5.73 | - | 1 | 50 | 635 | 622 | 3.1 |
| | | 203 | 5.12 | - | 1 | 50 | 641 | | |
| | | 204 | 5.14 | - | 1 | 50 | 591 | | |
| 747 | 6♂ | 205 | 5.62 | - | 1 | 50 | 730 | 719 | 3.7 |
| | | 206 | 5.80 | - | 1 | 50 | 708 | | |
| | | 207 | 5.27 | - | 1 | 50 | 718 | | |

HMX solution 3.67 $mg.ml^{-1}$
Internal standard solution 3.46 $mg.ml^{-1}$
QC solution of HMX 3.630 $mg.ml^{-1}$

TABLE 38 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 5.54 | Stand-ards | 208 | 5.19 | 2 | 1 | 50 | | | |
| | | 209 | 5.81 | 2 | 1 | 50 | | | |
| | | 210 | 6.07 | 2 | 1 | 50 | | | |
| 12.68 | QC | 211 | 5.61 | 2 | 1 | 50 | 12.68 | 12.63 | 0.4 |
| | | 212 | 5.57 | 2 | 1 | 50 | 12.65 | | |
| | | 213 | 6.54 | 2 | 1 | 50 | 12.56 | | |
| 2228 | 6♀ | 214 | 5.30 | - | 1 | 52 | 2258 | 2279 | 2.3 |
| | | 215 | 5.13 | - | 1 | 52 | 2394 | | |
| | | 216 | 5.09 | - | 1 | 52 | 2185 | | |

HMX solution 5.54 $mg.ml^{-1}$
Internal standard solution 12.355 $mg.ml^{-1}$
QC solution of HMX 6.34 $mg.ml^{-1}$

TABLE 39

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 27 February 1981 (Week 6)
Date of Analysis: 11 March 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.174 | Stand-ards | 217 | 5.00 | 1 | 1 | 50 | | | |
| | | 218 | 5.39 | 1 | 1 | 50 | | | |
| | | 219 | 5.19 | 1 | 1 | 50 | | | |
| 0.115 | QC | 220 | 5.09 | 1 | 1 | 50 | 0.130 | | |
| | | 221 | 4.97 | 1 | 1 | 50 | 0.129 | 0.129 | 12.2 |
| | | 222 | 5.34 | 1 | 1 | 50 | 0.127 | | |
| 0 | 1 + 7 ♂♀ | 223 | 5.00 | - | 1 | 50 | 0 | | |
| | | 224 | 5.15 | - | 1 | 50 | 0 | 0 | 0 |
| | | 225 | 5.26 | - | 1 | 50 | 0 | | |

HMX solution 0.174 $mg.ml^{-1}$
Internal standard solution 0.165 $mg.ml^{-1}$
QC solution of HMX 0.115 $mg.ml^{-1}$

TABLE 39 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 13 | 2♂ | 226 | 5.18 | - | 1 | 50 | 14 | 13 | 0 |
| | | 227 | 5.00 | - | 1 | 50 | 13 | | |
| | | 228 | 5.18 | - | 1 | 50 | 12 | | |
| 24 | 2♀ | 229 | 5.07 | - | 1 | 50 | 24 | 27 | 12.5 |
| | | 230 | 5.03 | - | 1 | 50 | 26 | | |
| | | 231 | 5.04 | - | 1 | 50 | 31 | | |
| 31 | 3♂ | 232 | 5.08 | - | 1 | 50 | 36 | 35 | 12.9 |
| | | 233 | 5.28 | - | 1 | 50 | 34 | | |
| | | 234 | 5.08 | - | 1 | 50 | 35 | | |

HMX solution 0.174 $mg.ml^{-1}$
Internal standard solution 0.165 $mg.ml^{-1}$
QC solution of HMX 0.115 $mg.ml^{-1}$

TABLE 39 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.506 | Stand-ards | 235 | 4.99 | 1 | 1 | 50 | | | |
| | | 236 | 5.00 | 1 | 1 | 50 | | | |
| | | 237 | 5.09 | 1 | 1 | 50 | | | |
| 0.630 | QC | 238 | 5.03 | 1 | 1 | 50 | 0.639 | | |
| | | 239 | 5.39 | 1 | 1 | 50 | 0.628 | 0.636 | 1.0 |
| | | 240 | 5.00 | 1 | 1 | 50 | 0.641 | | |
| 70 | 3♀ | 241 | 5.03 | - | 1 | 50 | 66 | | |
| | | 242 | 5.07 | - | 1 | 50 | 76 | 70 | 0 |
| | | 243 | 5.17 | - | 1 | 50 | 68 | | |
| 76 | 4♂ | 244 | 5.01 | - | 1 | 50 | 81 | | |
| | | 245 | 5.08 | - | 1 | 50 | 77 | 78 | 2.6 |
| | | 246 | 5.14 | - | 1 | 50 | 75 | | |

HMX solution 0.506 $mg.ml^{-1}$
Internal standard solution 0.51 $mg.ml^{-1}$
QC solution of HMX 0.630 $mg.ml^{-1}$

TABLE 39 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1.260 | Stand-ards | 247 | 5.00 | 1 | 1 | 50 | | | |
| | | 248 | 5.09 | 1 | 1 | 50 | | | |
| | | 249 | 4.99 | 1 | 1 | 50 | | | |
| 1.470 | QC | 250 | 5.15 | 1 | 1 | 50 | 1.413 | 1.429 | 2.8 |
| | | 251 | 5.13 | 1 | 1 | 50 | 1.444 | | |
| | | 252 | 5.25 | 1 | 1 | 50 | 1.431 | | |
| 204 | 4♀ | 253 | 5.23 | - | 1 | 50 | 220 | 208 | 2.0 |
| | | 254 | 5.26 | - | 1 | 50 | 197 | | |
| | | 255 | 5.13 | - | 1 | 50 | 206 | | |
| 248 | 5♂ | 256 | 5.03 | - | 1 | 50 | 247 | 250 | 0.8 |
| | | 257 | 5.04 | - | 1 | 50 | 253 | | |
| | | 258 | 5.00 | - | 1 | 50 | 250 | | |

HMX solution 1.260 $mg.ml^{-1}$
Internal standard solution 1.495 $mg.ml^{-1}$
QC solution of HMX 1.470 $mg.ml^{-1}$

TABLE 39 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 3.90 | Stand-ards | 259 | 5.06 | 1 | 1 | 50 | | | |
| | | 260 | 5.06 | 1 | 1 | 50 | | | |
| | | 261 | 5.02 | 1 | 1 | 50 | | | |
| 3.44 | QC | 262 | 5.15 | 1 | 1 | 50 | 3.43 | | |
| | | 263 | 4.94 | 1 | 1 | 50 | 3.45 | 3.46 | 0.6 |
| | | 264 | 5.38 | 1 | 1 | 50 | 3.51 | | |
| 613 | 5♀ | 265 | 5.50 | - | 1 | 50 | 602 | | |
| | | 266 | 5.23 | - | 1 | 50 | 647 | 624 | 1.8 |
| | | 267 | 5.04 | - | 1 | 50 | 622 | | |
| 645 | 6♂ | 268 | 5.00 | - | 1 | 50 | 639 | | |
| | | 269 | 5.14 | - | 1 | 50 | 621 | 623 | 3.4 |
| | | 270 | 5.36 | - | 1 | 50 | 608 | | |

HMX solution 3.90 $mg.ml^{-1}$
Internal standard solution 3.68 $mg.ml^{-1}$
QC solution of HMX 3.44 $mg.ml^{-1}$

TABLE 39 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 12.56 | Standards | 271 | 5.00 | 2 | 1 | 50 | | | |
| | | 272 | 4.99 | 2 | 1 | 50 | | | |
| | | 273 | 5.44 | 2 | 1 | 50 | | | |
| 13.06 | QC | 274 | 5.36 | 2 | 1 | 50 | 13.57 | | |
| | | 275 | 5.00 | 2 | 1 | 50 | 13.20 | 13.35 | 2.2 |
| | | 276 | 5.02 | 2 | 1 | 50 | 13.29 | | |
| 1789 | 6♀ | 277 | 5.06 | - | 1 | 52 | 1794 | | |
| | | 278 | 5.87 | - | 1 | 52 | 1783 | 1788 | 0 |
| | | 279 | 5.34 | - | 1 | 52 | 1786 | | |

HMX solution 6.28 $mg.ml^{-1}$
Internal standard solution 12.33 $mg.ml^{-1}$
QC solution of HMX 6.53 $mg.ml^{-1}$

TABLE 40

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 20 March 1981 (Week 9)
Date of Analysis: 30 March 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.134 | Standards | 280 | 5.60 | 1 | 1 | 50 | | | |
| | | 281 | 5.62 | 1 | 1 | 50 | | | |
| | | 282 | 5.55 | 1 | 1 | 50 | | | |
| 0.140 | QC | 283 | 5.31 | 1 | 1 | 50 | 0.138 | | |
| | | 284 | 5.34 | 1 | 1 | 50 | 0.143 | 0.141 | 0.7 |
| | | 285 | 5.02 | 1 | 1 | 50 | 0.143 | | |
| 0 | 1 + 7 ♂♀ | 286 | 5.12 | - | 1 | 50 | 0 | | |
| | | 287 | 5.14 | - | 1 | 50 | 0 | 0 | 0 |
| | | 288 | 5.13 | - | 1 | 50 | 0 | | |

HMX solution 0.134 $mg.ml^{-1}$
Internal standard solution 0.126 $mg.ml^{-1}$
QC solution of HMX 0.140 $mg.ml^{-1}$

TABLE 40 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 15 | 2♂ | 289 | 5.75 | - | 1 | 50 | 17 | 18 | 20.0 |
| | | 290 | 6.08 | - | 1 | 50 | 19 | | |
| | | 291 | 4.75 | - | 1 | 50 | 17 | | |
| 27 | 2♀ | 292 | 5.03 | - | 1 | 50 | 27 | 26 | 3.7 |
| | | 293 | 5.08 | - | 1 | 50 | 25 | | |
| | | 294 | 6.35 | - | 1 | 50 | 27 | | |
| 35 | 3♂ | 295 | 5.51 | - | 1 | 50 | 37 | 37 | 5.7 |
| | | 296 | 5.49 | - | 1 | 50 | 36 | | |
| | | 297 | 5.36 | - | 1 | 50 | 37 | | |

HMX solution 0.134 $mg.ml^{-1}$
Internal standard solution 0.126 $mg.ml^{-1}$
QC solution of HMX 0.140 $mg.ml^{-1}$

TABLE 40 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.518 | Standards | 298 | 5.02 | 1 | 1 | 50 | | | |
| | | 299 | 5.49 | 1 | 1 | 50 | | | |
| | | 300 | 5.49 | 1 | 1 | 50 | | | |
| 0.398 | QC | 301 | 5.74 | 1 | 1 | 50 | 0.395 | | |
| | | 302 | 4.78 | 1 | 1 | 50 | 0.401 | 0.397 | 0.3 |
| | | 303 | 5.04 | 1 | 1 | 50 | 0.394 | | |
| 79 | 3♀ | 304 | 5.42 | - | 1 | 50 | 84 | | |
| | | 305 | 4.42 | - | 1 | 50 | 84 | 84 | 6.3 |
| | | 306 | 5.67 | - | 1 | 50 | 83 | | |
| 89 | 4♂ | 307 | 5.13 | - | 1 | 50 | 96 | | |
| | | 308 | 5.00 | - | 1 | 50 | 96 | 94 | 5.6 |
| | | 309 | 5.10 | - | 1 | 50 | 91 | | |

HMX solution 0.518 $mg.ml^{-1}$
Internal standard solution 0.47 $mg.ml^{-1}$
QC solution of HMX 0.398 $mg.ml^{-1}$

TABLE 40 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1.46 | Stand-ards | 310 | 4.93 | 1 | 1 | 50 | | | |
| | | 311 | 5.66 | 1 | 1 | 50 | | | |
| | | 312 | 5.58 | 1 | 1 | 50 | | | |
| 1.22 | QC | 313 | 5.96 | 1 | 1 | 50 | 1.14 | 1.19 | 2.5 |
| | | 314 | 5.55 | 1 | 1 | 50 | 1.21 | | |
| | | 315 | 4.86 | 1 | 1 | 50 | 1.21 | | |
| 238 | 4♀ | 316 | 4.56 | - | 1 | 50 | 237 | 231 | 2.9 |
| | | 317 | 5.23 | - | 1 | 50 | 236 | | |
| | | 318 | 5.48 | - | 1 | 50 | 220 | | |
| 254 | 5♂ | 319 | 4.88 | - | 1 | 50 | 290 | 273 | 7.5 |
| | | 320 | 5.33 | - | 1 | 50 | 259 | | |
| | | 321 | 5.28 | - | 1 | 50 | 270 | | |

HMX solution 1.46 $mg.ml^{-1}$

Internal standard solution 1.36 $mg.ml^{-1}$

QC solution of HMX 1.22 $mg.ml^{-1}$

TABLE 40 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 4.35 | Stand-ards | 322 | 6.23 | 1 | 1 | 50 | | | |
| | | 323 | 6.82 | 1 | 1 | 50 | | | |
| | | 324 | 6.09 | 1 | 1 | 50 | | | |
| 3.21 | QC | 325 | 6.16 | 1 | 1 | 50 | 3.25 | | |
| | | 326 | 5.08 | 1 | 1 | 50 | 3.27 | 3.24 | 0.9 |
| | | 327 | 5.77 | 1 | 1 | 50 | 3.20 | | |
| 685 | 5♀ | 328 | 5.64 | - | 1 | 50 | 696 | | |
| | | 329 | 5.62 | - | 1 | 50 | 698 | 693 | 1.2 |
| | | 330 | 5.29 | - | 1 | 50 | 685 | | |
| 661 | 6♂ | 331 | 4.47 | - | 1 | 50 | 678 | | |
| | | 332 | 5.45 | - | 1 | 50 | 673 | 679 | 2.7 |
| | | 333 | 4.05 | - | 1 | 50 | 685 | | |

HMX solution 4.35 $mg.ml^{-1}$
internal standard solution 3.615 $mg.ml^{-1}$
QC solution of HMX 3.21 $mg.ml^{-1}$

TABLE 40 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 10.78 | Stand-ards | 334 | 5.34 | 2 | 1 | 50 | | | |
| | | 335 | 5.34 | 2 | 1 | 50 | | | |
| | | 336 | 5.39 | 2 | 1 | 50 | | | |
| 11.34 | QC | 337 | 4.68 | 2 | 1 | 50 | 11.22 | | |
| | | 338 | 4.82 | 2 | 1 | 50 | 11.15 | 11.24 | 0.9 |
| | | 339 | 4.76 | 2 | 1 | 50 | 11.34 | | |
| 2204 | 6♀ | 340 | 5.04 | - | 1 | 52 | 2240 | | |
| | | 341 | 4.79 | - | 1 | 52 | 2134 | 2190 | 0.6 |
| | | 342 | 6.34 | - | 1 | 52 | 2197 | | |

HMX solution 5.39 $mg.ml^{-1}$
Internal standard solution 11.55 $mg.ml^{-1}$
QC solution of HMX 5.67 $mg.ml^{-1}$

TABLE 41

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 10 April 1981 (termination)
Date of Analysis: 24 April 1981

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.128 | Standards | 343 | 4.95 | 1 | 1 | 50 | | | |
| | | 344 | 6.00 | 1 | 1 | 50 | | | |
| | | 345 | 5.66 | 1 | 1 | 50 | | | |
| 0.137 | QC | 346 | 5.43 | 1 | 1 | 50 | 0.133 | | |
| | | 347 | 5.40 | 1 | 1 | 50 | 0.125 | 0.128 | 6.6 |
| | | 348 | 4.60 | 1 | 1 | 50 | 0.127 | | |
| 0 | 1 + 7 ♂♀ | 349 | 5.47 | - | 1 | 50 | 0 | | |
| | | 350 | 7.16 | - | 1 | 50 | 0 | 0 | 0 |
| | | 351 | 4.77 | - | 1 | 50 | 0 | | |

HMX solution 0.128 $mg.ml^{-1}$
Internal standard solution 0.113 $mg.ml^{-1}$
QC solution of HMX 0.137 $mg.ml^{-1}$

TABLE 41 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 16 | 2♂ | 352 | 7.83 | - | 1 | 50 | 16 | 16 | 0 |
| | | 353 | 5.32 | - | 1 | 50 | 16 | | |
| | | 354 | 5.80 | - | 1 | 50 | 15 | | |
| 27 | 2♀ | 355 | 6.14 | - | 1 | 50 | 36 35 36 | 31 | 14.8 ++ |
| | | 356 | 4.70 | - | 1 | 50 | 24 35 24 | | |
| | | 357 | 6.04 | - | 1 | 50 | 28 28 | | |
| 41 | 3♂ | 358 | 5.24 | - | 1 | 50 | 38 | 38 | 7.3 |
| | | 359 | 4.82 | - | 1 | 50 | 38 | | |
| | | 360 | 5.34 | - | 1 | 50 | 37 | | |

HMX solution 0.128 $mg.ml^{-1}$

Internal standard solution 0.113 $mg.ml^{-1}$

QC solution of HMX 0.137 $mg.ml^{-1}$

++ = Analysis repeated since original values were outwith $\pm$ 5 ppm

TABLE 41 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.546 | Standards | 361 | 4.79 | 1 | 1 | 50 | | | |
| | | 362 | 5.42 | 1 | 1 | 50 | | | |
| | | 363 | 5.18 | 1 | 1 | 50 | | | |
| 0.408 | QC | 364 | 5.99 | 1 | 1 | 50 | 0.356 | | |
| | | 365 | 6.12 | 1 | 1 | 50 | 0.428 | 0.410 | 0.5 |
| | | 366 | 5.22 | 1 | 1 | 50 | 0.445 | | |
| 82 | 3♀ | 367 | 5.46 | - | 1 | 50 | 89 78 | | ++ |
| | | 368 | 5.06 | - | 1 | 50 | 97 75 | 82 | 0 |
| | | 369 | 5.86 | - | 1 | 50 | 95 92 | | |
| 105 | 4♂ | 370 | 6.79 | - | 1 | 50 | 125 122 | | ++ |
| | | 371 | 6.32 | - | 1 | 50 | 130 105 | 109 | 3.8 |
| | | 372 | 5.63 | - | 1 | 50 | 114 101 | | |

HMX solution 0.546 $mg.ml^{-1}$
Internal standard solution 0.570 $mg.ml^{-1}$
QC solution of HMX 0.408 $mg.ml^{-1}$
++ = Analysis repeated since original values were outwith 10%
Mean concentrations are those from the repeat analyses

TABLE 41 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1.18 | Standards | 373 | 5.23 | 1 | 1 | 50 | | | |
| | | 374 | 5.17 | 1 | 1 | 50 | | | |
| | | 375 | 5.14 | 1 | 1 | 50 | | | |
| 1.03 | QC | 376 | 5.27 | 1 | 1 | 50 | 1.05 | 1.05 | 1.9 |
| | | 377 | 4.65 | 1 | 1 | 50 | 1.11 | | |
| | | 378 | 5.43 | 1 | 1 | 50 | 0.99 | | |
| 245 | 4♀ | 379 | 5.91 | - | 1 | 50 | 240 | 236 | 3.7 |
| | | 380 | 7.05 | - | 1 | 50 | 235 | | |
| | | 381 | 6.08 | - | 1 | 50 | 232 | | |
| 302 | 5♂ | 382 | 5.68 | - | 1 | 50 | 303 | 301 | 0.3 |
| | | 383 | 6.20 | - | 1 | 50 | 301 | | |
| | | 384 | 5.50 | - | 1 | 50 | 298 | | |

HMX solution 1.18 $mg.ml^{-1}$
Internal standard solution 1.585 $mg.ml^{-1}$
QC solution of HMX 1.03 $mg.ml^{-1}$

TABLE 41 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 4.60 | Stand-ards | 385 | 5.67 | 1 | 1 | 50 | | | |
| | | 386 | 6.05 | 1 | 1 | 50 | | | |
| | | 387 | 5.49 | 1 | 1 | 50 | | | |
| 3.26 | QC | 388 | 6.06 | 1 | 1 | 50 | 3.35 | 3.41 | 4.6 |
| | | 389 | 5.80 | 1 | 1 | 50 | 3.49 | | |
| | | 390 | 5.80 | 1 | 1 | 50 | 3.39 | | |
| 728 | 5♀ | 391 | 5.40 | - | 1 | 50 | 755 | 749 | 2.9 |
| | | 392 | 5.16 | - | 1 | 50 | 750 | | |
| | | 393 | 5.36 | - | 1 | 50 | 743 | | |
| 830 | 6♂ | 394 | 5.25 | - | 1 | 50 | 871 | 874 | 5.3 |
| | | 395 | 6.15 | - | 1 | 50 | 869 | | |
| | | 396 | 5.63 | - | 1 | 50 | 881 | | |

HMX solution 4.60 $mg.ml^{-1}$
Internal standard solution 3.47 $mg.ml^{-1}$
QC solution of HMX 3.26 $mg.ml^{-1}$

TABLE 41 (continued)

| Nominal Value | Group | Analytical Sample No. | Weight Diet Used (g) | Volume HMX Solution Added (ml) | Volume Internal Standard Solution (ml) | Volume Extracting Solvent (ml) | Value Found | Mean Value Found | Deviation of Mean Value (%) |
|---|---|---|---|---|---|---|---|---|---|
| 11.14 | Stand-ards | 397 | 6.34 | 2 | 1 | 50 | | | |
| | | 398 | 7.34 | 2 | 1 | 50 | | | |
| | | 399 | 5.21 | 2 | 1 | 50 | | | |
| 11.34 | QC | 400 | 5.38 | 2 | 1 | 50 | 11.26 | 11.27 | 0.6 |
| | | 401 | 5.03 | 2 | 1 | 50 | 11.24 | | |
| | | 402 | 5.92 | 2 | 1 | 50 | 11.30 | | |
| 2550 | 6? | 403 | 5.33 | - | 1 | 52 | 2494 | 2531 | 0.7 |
| | | 404 | 5.58 | - | 1 | 52 | 2520 | | |
| | | 405 | 6.05 | - | 1 | 52 | 2578 | | |

HMX solution 5.57 $mg.ml^{-1}$
Internal standard solution 12.04 $mg.ml^{-1}$
QC solution of HMX 5.67 $mg.ml^{-1}$

FIGURE 1

Ultraviolet Spectrum of HMX

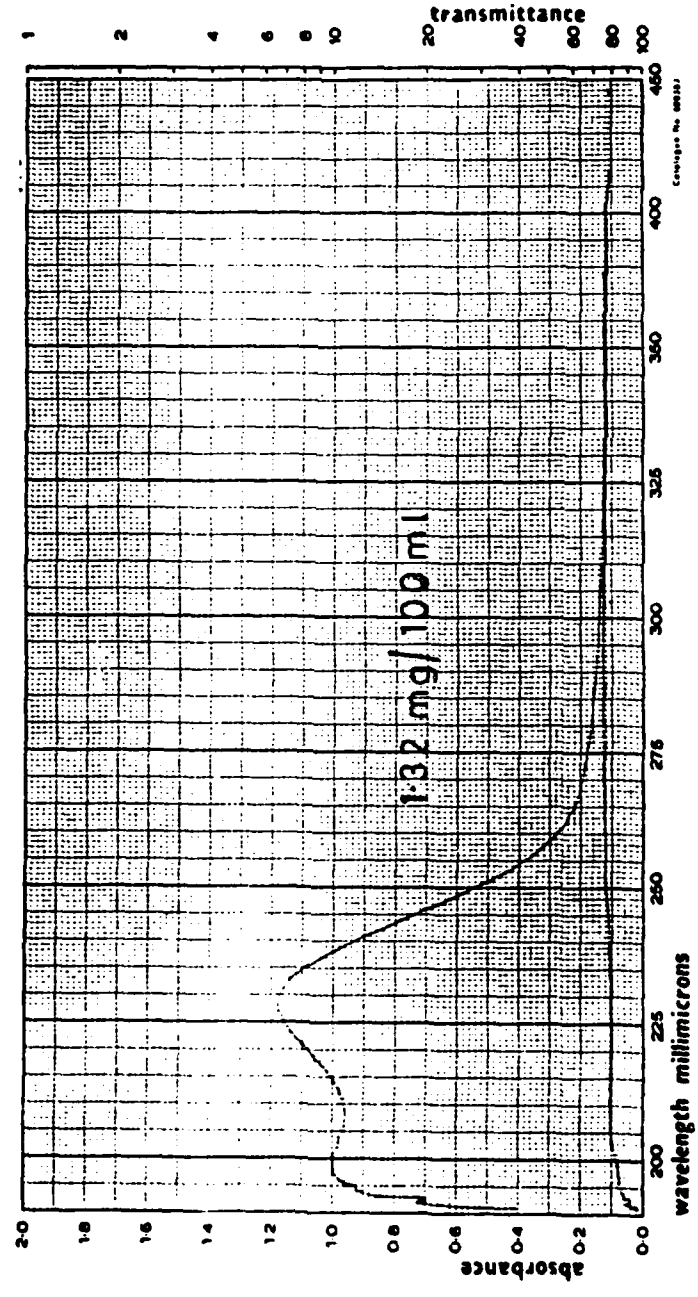


FIGURE 2

HPLC of HMX and Related Explosives (Solvent 1)

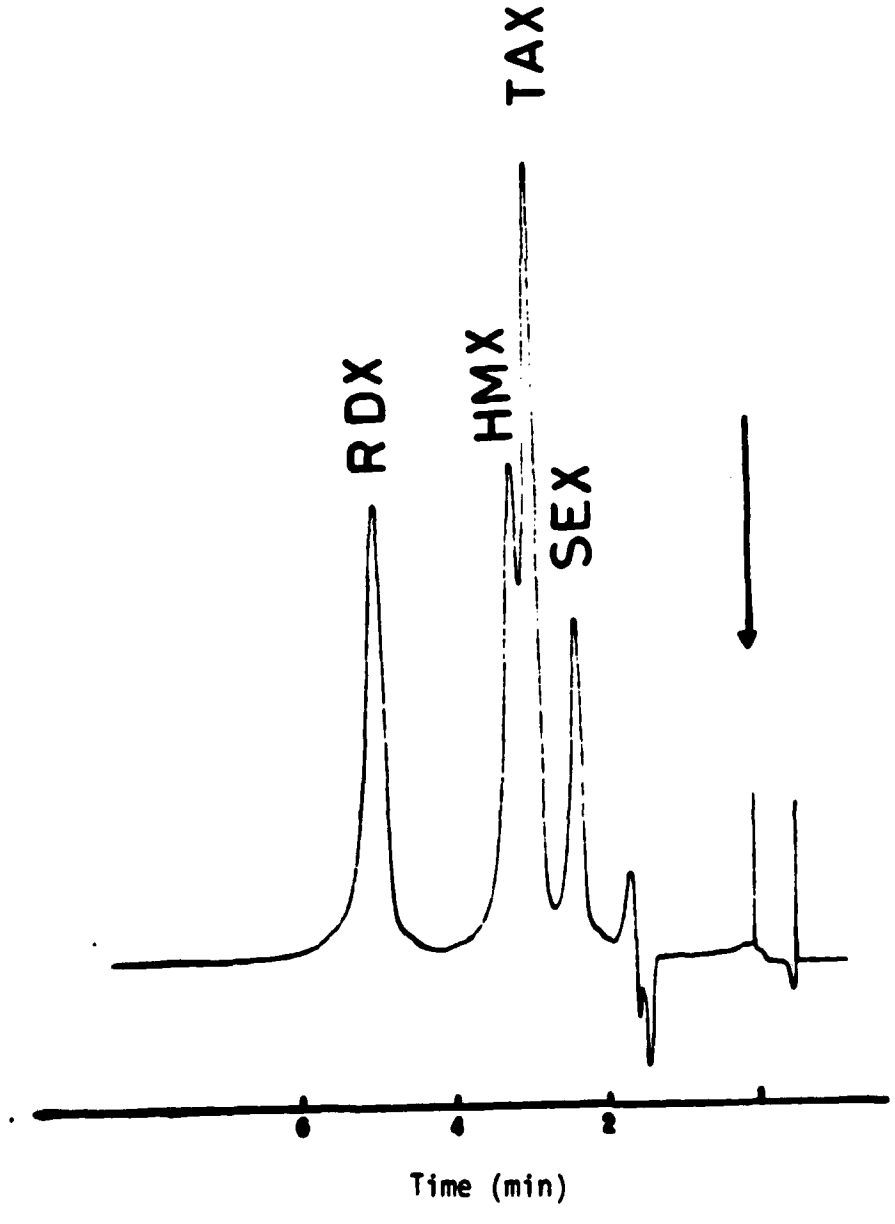


FIGURE 3

HPLC of HMX and RDX (Solvent 2)

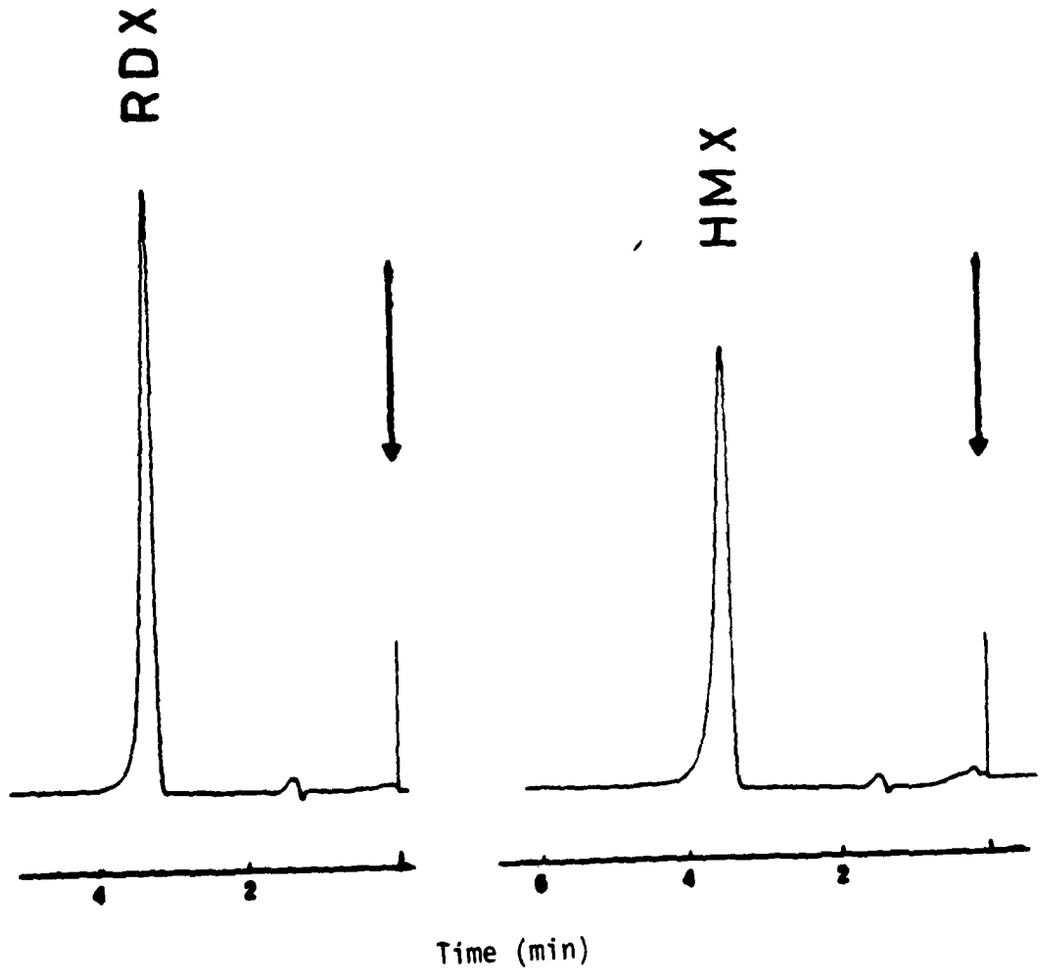


FIGURE 4

HPLC of HMX and RDX (Solvent 2)

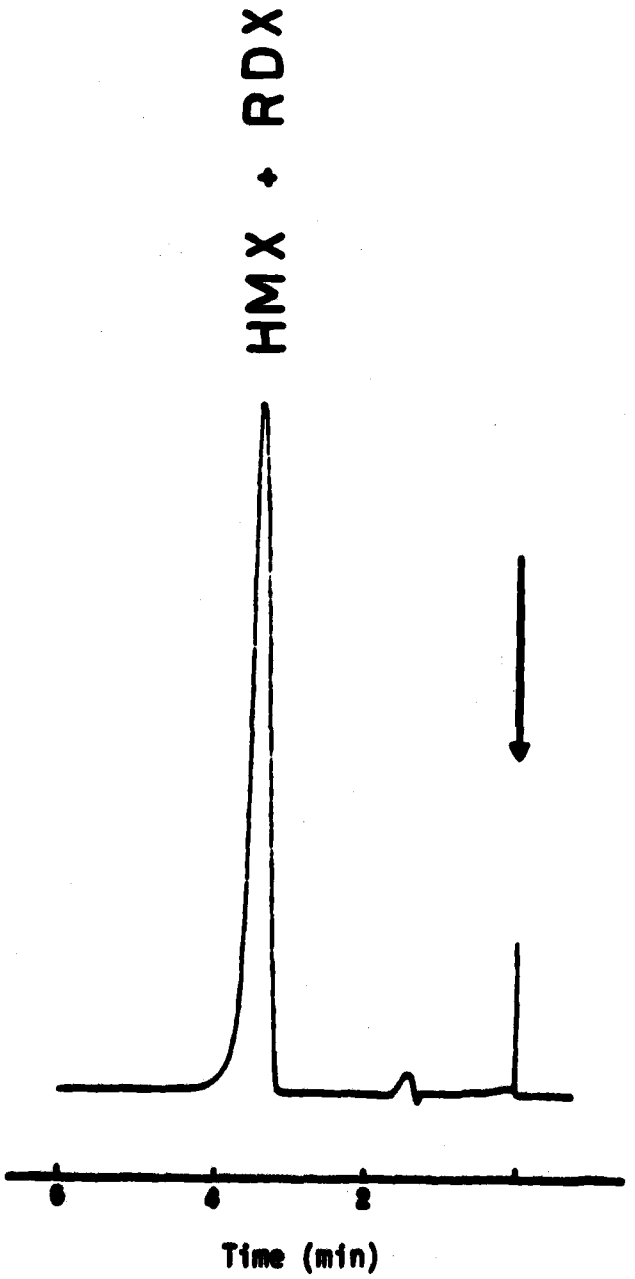


FIGURE 5

HPLC of HMX and Related Explosives (Solvent 2)

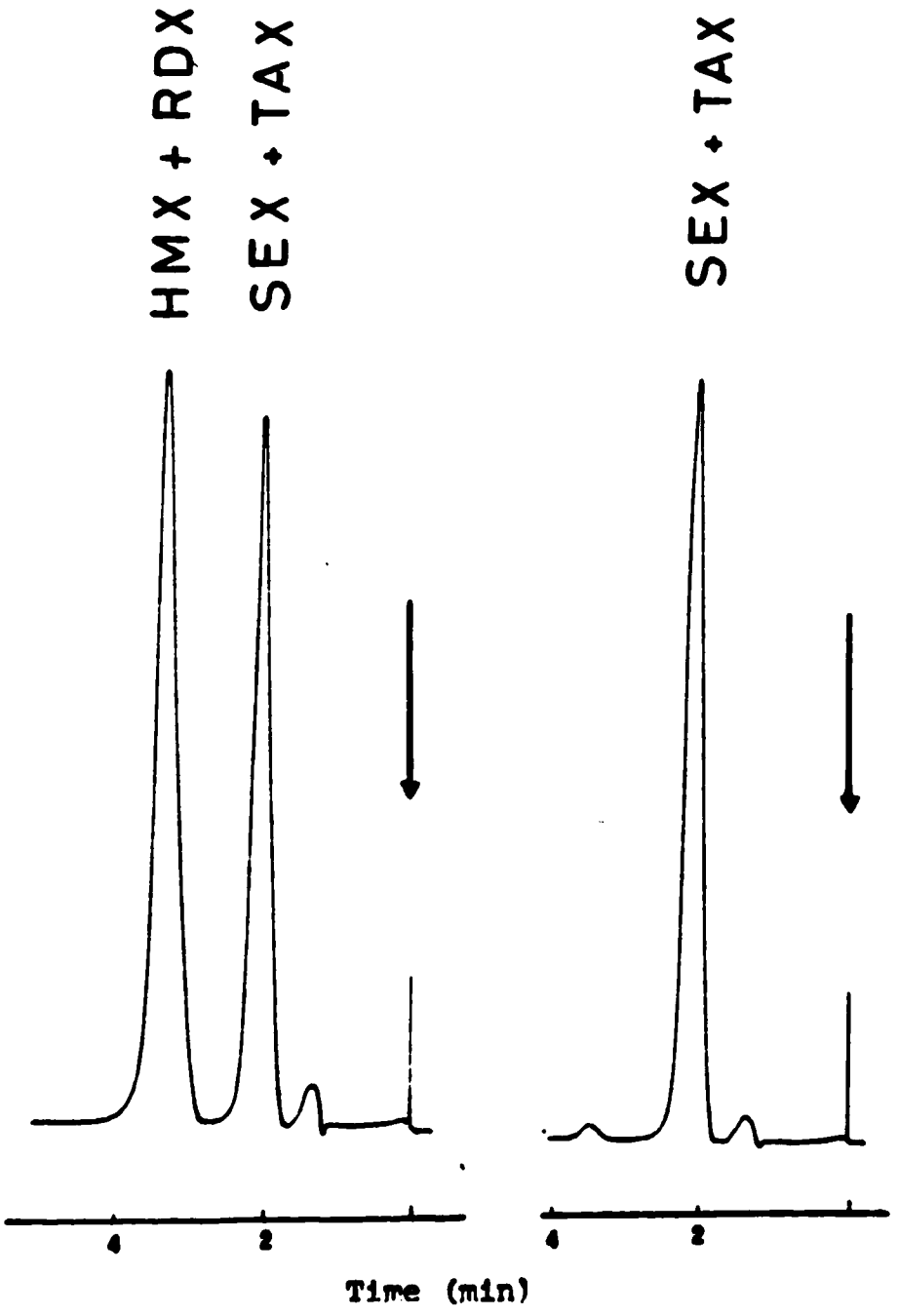


FIGURE 6

HPLC of HMX and Tetryl (Solvent 2)

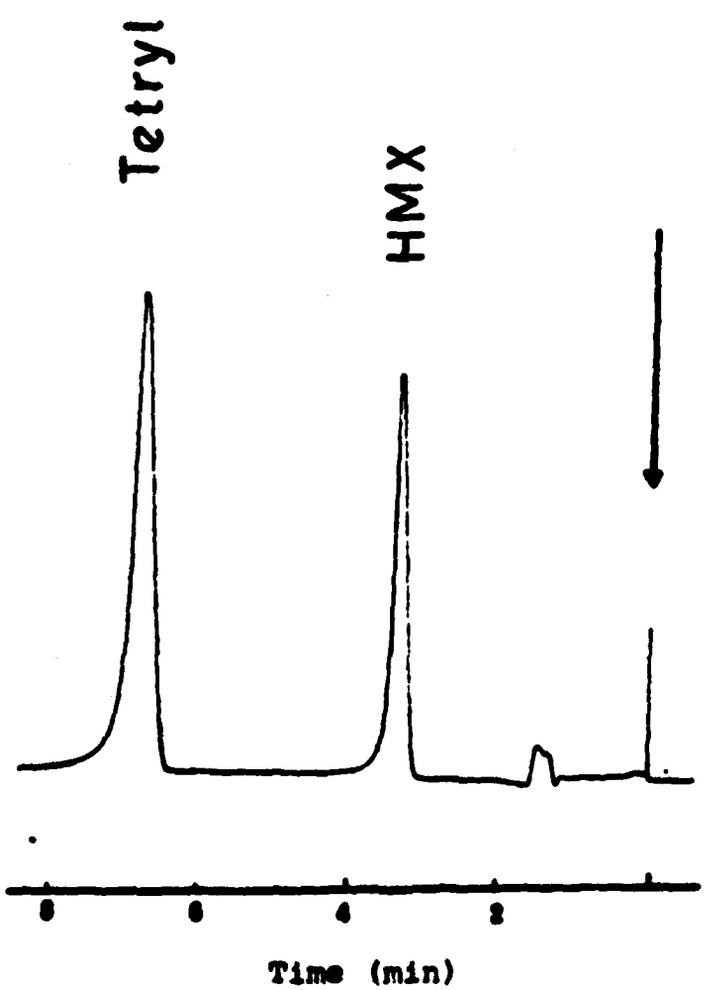


FIGURE 7

HPLC if HMX and 1,3-Dinitrobenzene (DNB)

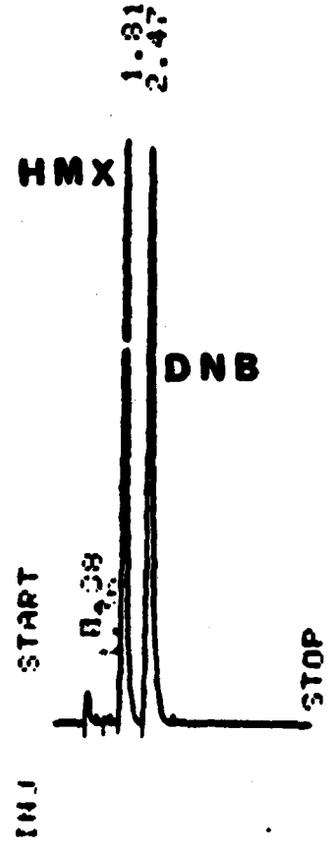
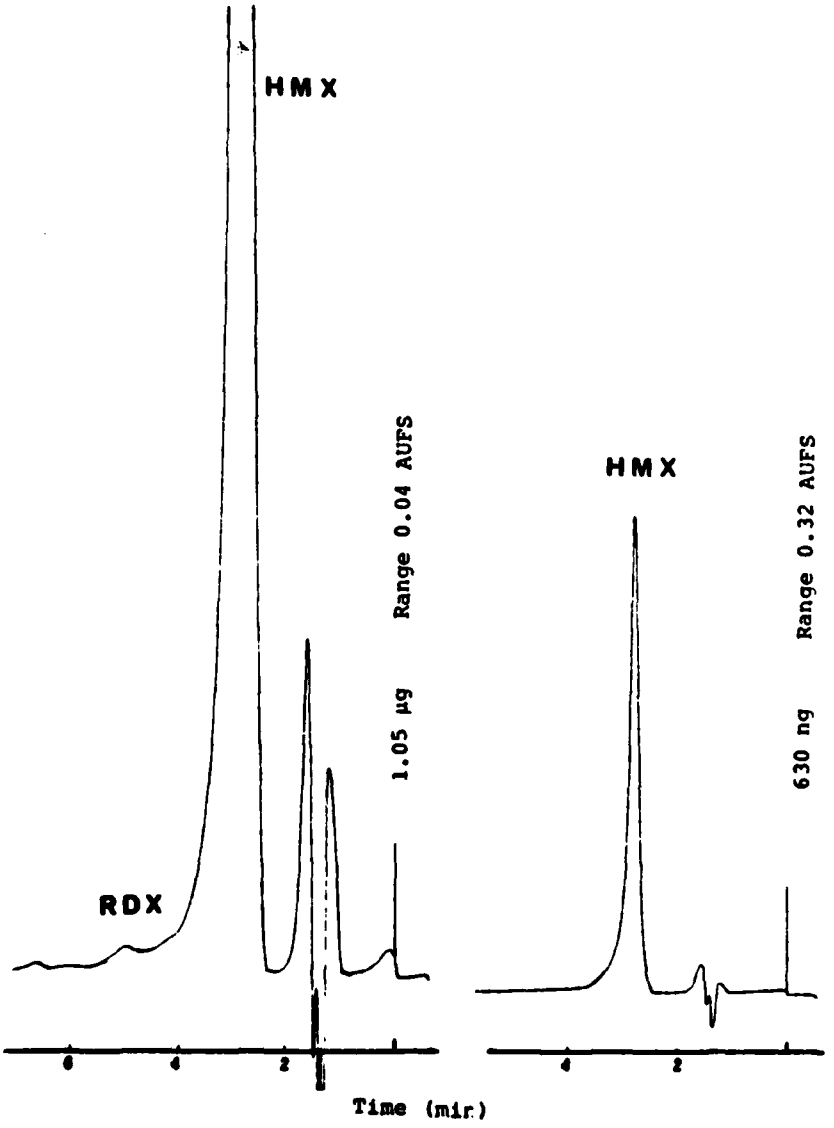


FIGURE 8

HPLC of HMX Solutions (Solvent 1)



# APPENDIX 1

## HMX: Analysis of Dosing Formulations Used in Acute, Sub-acute and Sub-chronic Toxicity Studies
## Diet Analysis

**B.P. NUTRITION (U.K.) LTD.**
**SPECIAL QUALITY CONTROL OF LABORATORY ANIMAL DIETS**

**CERTIFICATE OF ANALYSIS**

PRODUCT: RAT & MOUSE NO.1 (MODIFIED) EXPANDED FINE GROUND

BATCH NO: 919 PREMIX BATCH NO: P110

DATE OF MANUFACTURE: 15TH AUGUST 1980

| Nutrient | Found Analysis | | Contaminant | Found Analysis | | Limit of Detection |
|---|---|---|---|---|---|---|
| Moisture | 7.1 | % | Fluorine | 7.6 | mg/kg | 10.0 mg/kg |
| Crude Fat | 3.5* | % | Nitrate as $NaNO_3$ | 11.0 | mg/kg | 1.0 mg/kg |
| Crude Protein | 14.9 | % | Nitrite as $NaNO_2$ | $< 1.0$ | mg/kg | 1.0 mg/kg |
| Crude Fibre | 2.2 | % | Lead | $< 1.0$ | mg/kg | 1.0 mg/kg |
| Ash | 4.8 | % | Arsenic | 0.23 | mg/kg | 0.2 mg/kg |
| Calcium | 0.69 | % | Cadmium | 0.13 | mg/kg | 0.2 mg/kg |
| Phosphorus | 0.53 | % | Mercury | $< 0.01$ | mg/kg | 0.01 mg/kg |
| Sodium | 0.22 | % | Selenium | 0.12 | mg/kg | 0.02 mg/kg |
| Chlorine | 0.34 | % | | | | |
| Potassium | 1.10 | % | | | | |
| Magnesium | 0.13 | % | Total Aflatoxins | NONE DETECTED | ug/kg | 1 ug/kg each of B1,B2,G1,G2 |
| Iron | 231 | mg/kg | | | | |
| Copper | 7 | mg/kg | | | | |
| Manganese | 55 | mg/kg | | | | |
| Zinc | 40 | mg/kg | Total P.C.B. | NONE DETECTED | mg/kg | 0.001 mg/kg |
| | | | Total D.D.T. | 0.003 | mg/kg | 0.001 mg/kg |
| | | | Dieldrin | 0.001 | mg/kg | 0.001 mg/kg |
| | | | Lindane | 0.005 | mg/kg | 0.001 mg/kg |
| | | | Heptachlor | NONE DETECTED | mg/kg | 0.001 mg/kg |
| | | | Malathion | NONE DETECTED | mg/kg | 0.02 mg/kg |
| Vitamin A | 7000 | iu/kg | Total Viable Organisms | $1.13 \times 10^3$ | per grm | 1000/g |
| Vitamin E | 60 | mg/kg | | | | |
| Vitamin C | | mg/kg | Mesophilic Spores | $17.5 \times 10^2$ | per grm | 100/g |
| | | | Salmonellae Species | NONE DETECTED | per grm | Absent in 20 grm |
| | | | Presumptive E. Coli | NONE DETECTED | per grm | Absent in 10 grm |
| | | | E. Coli Type 1 | NONE DETECTED | per grm | Absent in 10 grm |
| | | | Fungal Units | NONE DETECTED | per grm | Absent in 10 grm |
| | | | Antibiotic Activity | | | |

*repeat 3.4

RECEIVED
17 SEP 1980

Signed C R Popplestone

Dated 10th September 1980

C. R. POPPLESTONE M.Sc., Ph.D., C.Chem., M.R.I.C.
Quality Control Manager

B.P. Nutrition (U.K.) Limited
Stoptield,
Witham,
Essex, CM8 3AB
Telephone: (0376) 513651

## PERSONNEL INVOLVED IN PROJECTS 415669 AND 416877

| | |
|---|---|
| Principal Investigator: | A.B. Wilson, B.V.Sc., M.R.C.V.S., D.A.B.T. |
| Department Manager: (Analytical Chemistry) | J.D. Gilbert, B.Sc., Ph.D. C.Chem., F.R.S.C. |
| Senior Analyst: | M.S. Henderson, B.Sc., Ph.D. |
| Analysts: | D.W. Carmichael, B.Sc.<br>S. Souter |
| Project Leader (Acute Toxicology): | S.M.A. Carr |
| Test Substance Formulator: | A. Soden |
| Dispensary Assistant: | A. Trench |
| Quality Assurance: | E.M. Baxendine, B.Sc.,<br>A.W. Waddell, B.Sc., Ph.D.<br>F.M. Cunningham, M.Sc., M.I.Biol. |

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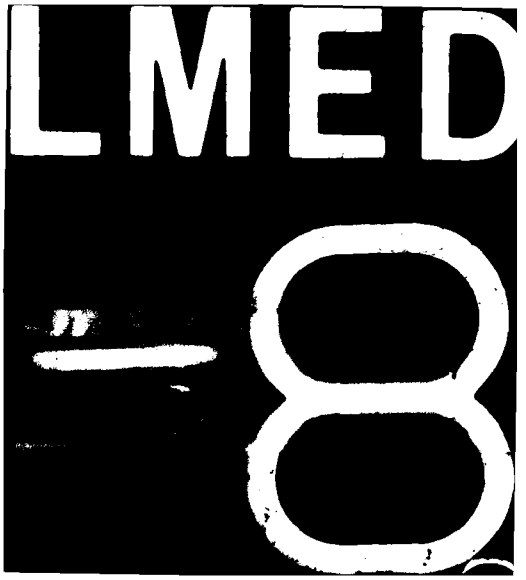
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