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An Acute Inhalation Toxicological Evaluation of Combustion Products From Fire Retarded and Non-Fire Retarded Flexible Polyurethane Foam and Polyester

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Fire Research
Washington, DC 20234

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Final Report

Sponsored in part by:
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Bethesda, MD 20207

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AN ACUTE INHALATION TOXICOLOGICAL EVALUATION OF COMBUSTION PRODUCTS FROM FIRE RETARDED AND NON-FIRE RETARDED FLEXIBLE POLYURETHANE FOAM AND POLYESTER

Barbara C. Levin, Maya Paabo, Mary Lou Fultz,
Cheryl Bailey, Way Yin, Steven E. Harris

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, ***Secretary***
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, ***Director***

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An Acute Inhalation Toxicological Evaluation of Combustion Products from Fire Retarded and Non-Fire Retarded Flexible Polyurethane Foam and Polyester

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Abstract

The acute inhalation toxicity of the combustion products from selected upholstered furniture filling materials with and without fire retardants was evaluated by the toxicity test method developed by the National Bureau of Standards. The fire materials that were evaluated consisted of two different formulations of flexible polyurethane foam (each formulation was supplied in both a fire retarded and non-fire retarded form) and a polyester fiberfill (not fire retarded). Atmospheric concentrations of carbon monoxide, carbon dioxide, oxygen, and hydrogen cyanide in the exposure chamber, were monitored throughout the thermal decomposition of the materials. In addition, time-to-ignition, rate of heat release, hydrogen cyanide generation via a two phase decomposition procedure and x-ray fluorescence measurements were performed. The LC_{50} (30 minute and 14 day) values did not distinguish any of the materials as being significantly more toxic than the others. In the worst case, one of the fire retarded polyurethane foams was only a factor of two more toxic than its untreated counterpart. Only the fire retarded foams and the polyester caused deaths of the Fischer 344 rats during the 30 minute exposures. Extensive weight loss and post-exposure deaths occurred after exposure to the combustion products from all the materials. Animal deaths during or following exposure to the polyurethane cannot be attributed to carbon monoxide or hydrogen cyanide alone. Deaths during exposure to the polyester combustion products appear to be more directly related to the concentrations of carbon monoxide.

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An Acute Inhalation Toxicological Evaluation of Combustion Products from Fire Retarded and Non-Fire Retarded Flexible Polyurethane Foam and Polyester

1.0 INTRODUCTION

The largest number of U.S. fire deaths occur in one and two family residences [1][1]. The most frequent cause of residential fires is heating and cooking, while the major number of fire deaths and injuries stem from the cigarette ignition of upholstered furniture and bedding [1]. This information has led to much interest in producing fire resistant and thermally stable materials for upholstered furniture.

Most of the work in the fire retardant field has been directed toward meeting requirements in flammability regulations and voluntary standards. In 1974, however, the potential toxicological effects of combustion products from materials containing fire retardants were dramatically pointed out by the work of Petajan et al. [2]. Their study showed that the combustion products from a trimethylolpropane-based rigid urethane foam containing a reactive phosphate fire retardant caused grand mal seizures and death in rats. The presence of an obscure toxicant, 4-ethyl-1-phospha-2,6,7-trioxabicyclo [2.2.2] octane-1-oxide, was found to be responsible for the deaths in the above study. As this toxicant, under ordinary circumstances, would not have been detected by routine chemical analysis, the need for a biological test system was identified. In addition, toxicity studies on the combustion products of materials containing fire retardants was shown to be necessary.

Herpol has reported that four fire retardant treated materials were more toxic than their untreated counterparts [3]. On the other hand, Hilado showed that the toxicity was reduced in the fire retarded materials that he tested [4]. Other studies have found that the presence of fire

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[1] Numbers in brackets refer to the literature references listed at the end of this report.

retardants do not significantly increase the toxicity or toxic combustion products (e.g., hydrogen cyanide) from materials [5,6,7,8,9,10]. These differing results show that the polymer formulation and the type of fire retardant will influence the toxicity of materials.

The present study was designed to assess the acute inhalation toxicity of selected upholstered furniture filling materials with and without fire retardants using the National Bureau of Standards (NBS) toxicity test method [11]. However, it should be pointed out that it is very difficult to judge the fire safety of a material by the results of only a toxicity test. The NBS toxicity test, like other small-scale laboratory tests, is conducted under specified laboratory conditions*. It was designed to assess the relative intrinsic toxicity of materials under these specified conditions and not to measure the overall toxic hazard which might occur in a real fire situation. Some of the parameters needed to evaluate the toxic hazard to which a material may contribute in a real fire situation include: the quantity of material present, its configuration, the proximity of other combustibles, the volume of the compartments to which the combustion products may spread, the ventilation conditions, the ignition and combustion properties of the material(s) present, the presence of ignition sources, the presence of fire protection systems, and the building occupancy.

In this study, in addition to the NBS toxicity test method data specified by the contract with the U.S. Consumer Product Safety Commission (CPSC), other fire performance criteria such as time-to-ignition, rate of heat release, hydrogen cyanide generation under the conditions of the NBS toxicity test method and via a two phase decomposition procedure were collected and evaluated. Also, x-ray fluorescence measurements were performed on all the materials.

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*Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the equipment or material identified is necessarily the best available for the purpose.

2.0 MATERIALS AND METHODS

2.1 Materials

The five materials evaluated in this study were supplied by CPSC, Washington, DC 20207 and identified as FPU #11, 12, 13, 14 and PE #15. They consisted of two different formulations of flexible polyurethane foam (hereby specified as A and B) and a polyester fiberfill. Information provided by CPSC indicated that FPU #11 and 12 were formulation A and FPU #13 and 14 were formulation B. One sample of each formulation contained a fire retardant. The polyester fiberfill was not fire retarded. Prior to testing, NBS was not informed which samples were fire retarded.

2.2 Methods

2.2.1 NBS Toxicity Test Method

The acute inhalation toxicity of the combustion products was assessed by the NBS toxicity test method [11]. Briefly, the test method consists of three major components: (1) a combustion system, (2) a chemical analysis system, and (3) an animal exposure system (Fig. 1). The combustion system is a closed design in which all the combustion products are generated in a furnace located directly below the 200 liter rectangular exposure chamber (Fig. 2) and are kept within the chamber except for the volume which is transferred for chemical analysis and subsequently returned. The cup furnace is similar to that designed by Potts and Lederer (Fig. 3) [12]. A material is thermally degraded at a furnace temperature 25°C above (flaming mode) or 25°C below (nonflaming mode) its predetermined autoignition temperature. In an actual flaming exposure, ethanol and/or an electric spark was used to ensure immediate flaming.

Before experiments, all test materials were conditioned for at least 48 hours at 40-50 percent relative humidity and a temperature of 22-24°C. One piece samples were tested; larger pieces were rolled and bound with wire.

Carbon monoxide (CO) and carbon dioxide (CO_2) were measured continuously by non-dispersive infrared spectroscopy. Oxygen (O_2) concentrations were measured continuously by a galvanic cell. The CO, CO_2, and O_2 data were recorded by an on-line computer every 15 seconds as were the temperature measurements in the quartz beaker of the combustion system and at animal exposure positions #1, 3, and 6. The hydrogen cyanide (HCN) generated from the polyurethane foam samples was analyzed approximately every three minutes with a gas chromatograph equipped with a thermionic detector [13].

The animals used for these experiments were Fischer 344 male rats weighing 200-300 grams. They were obtained from the Harlan Sprague-Dawley Company (Walkersville, MD) and were allowed to acclimate to our laboratory conditions for at least 10 days prior to experimentation. Animal care and maintenance were performed in accordance with the procedures outlined in the National Institutes of Health's "Guide for the Care and Use of Laboratory Animals" [14]. Each rat was housed individually in suspended stainless steel cages and provided with food and water *ad libitum*. Twelve hours of fluorescent lighting per day were provided using an automatic timer. All animals were weighed daily from the day of arrival until the end of the 14 day post-exposure period. Animals that were still losing weight on day 14 were weighed daily until they died or recovered as indicated by three days of successive weight gain. Control animals from the same batch were weighed daily also.

Animals designated for blood analysis of carboxyhemoglobin (COHb) underwent cannulation 24 hours before experiments. This procedure involves the surgical insertion of a cannula into the animal's femoral artery [15] thereby allowing blood samples to be taken during the exposure. The blood levels of COHb are indicative of the amount of carbon monoxide inhaled by the animals.

Six animals were exposed in each experiment. Each animal was placed in a restrainer (Fig. 4) which was then inserted into one of the six portholes located along the front of the exposure chamber such that only the heads of the animals were exposed to the combustion atmosphere. Exposures were for 30 minutes, during which time blood was taken at 0 time, approximately 15 minutes and just before the end of the experiment from the cannulated animals (one or two animals were cannulated).

The toxicological endpoint was death. The number of animals that died at each mass loading of material was plotted to produce a concentration-response curve from which an LC_{50} (30 minutes and 14 days) was calculated. The LC_{50} in this case is defined as the mass loading of material per unit chamber volume (mg/ℓ) which caused 50% of the animals to die during the 30 minute exposure plus the 14 day post-exposure observation period[2]. The LC_{50}'s, their 95 percent confidence limits, and the slopes of the concentration-response curves and the 95% confidence limits on the slopes were calculated via the statistical method of Litchfield and Wilcoxon [16]. According to the NBS toxicity test method, the highest concentration of material tested was 40 mg/ℓ. Therefore, in some cases the LC_{50} is listed as greater than 40 mg/ℓ.

If the LC_{50} (30 minute and 14 day) values fell between 2 mg/ℓ and 30 mg/ℓ, the animals were exposed for 10 minutes to a 30 mg/ℓ loading. (The 10 minute test was not performed if a 30 minute test at 30 mg/ℓ produced less than 50% death in the animals during the exposure or post-exposure period even though the calculated LC_{50} was less than 30 mg/ℓ.) This 10 minute test was to determine if the material rapidly produces effective concentrations of toxic combustion products. The 10 minute exposure test was run twice at the temperature condition (flaming or non-flaming) which gave the lower LC_{50} value for 30 minutes and 14 days. If 50% or more of the animals from both 10 minute exposure tests died either within the 10 minutes or during a 14 day post-exposure observation period, the material was considered capable of rapidly producing toxicants.

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[2] Animals that were still losing weight on day 14 were kept until they died or recovered as noted above. Animals that died after day 14 were included in the LC_{50} calculation.

2.2.2 X-ray Fluorescence

X-ray fluorescence measurements were performed to characterize all the material samples and to determine the various elements that may have been added as fire retardants. The X-ray energy dispersive system consisted of a tungsten tube in conjunction with titanium and molybdenum secondary emitters and a lithium-drifted silicon detector. This system detects elements with atomic numbers greater than 11 (sodium). These measurements were performed according to the procedure of Pella, et al. [17] except that the samples were analyzed as received.

2.2.3 Combustion Parameters

Ignition delay times and rates of heat release were determined using an oxygen consumption based technique, the NBS cone calorimeter, which has been described by Babrauskas [18]. Samples of each material (#11-15) were exposed to three different radiant heat fluxes: 25, 50, and 75 kW/m^2. The sample size was 100 x 100 x 50 mm thick.

2.2.4 Two Phase Decomposition of Polyurethane Foam

A 20 mg/ℓ sample of each polyurethane foam (#11, 12, 13 and 14) was decomposed at 25°C below its autoignition temperature for 30 minutes. The furnace setting was then increased so that the 30 minute residue was heated at approximately 13°C per minute until the final temperature was 800°C. The time to reach 800°C was 32-34 minutes. These experiments were performed to measure HCN generation under conditions previously determined by NBS to produce increased concentrations of HCN [19]. No animals were exposed in these experiments.

3.0 RESULTS

3.1 X-ray Fluorescence

The major elements detected by the energy-dispersive spectra for the five samples were bromine (sample #11), calcium (sample #12), phosphorus and chlorine (sample #14), and antimony (sample #15). The bromine, phosphorus and chlorine are most likely associated with fire retardants indicating that samples #11 and 14 were the fire retarded samples. The finding of antimony was not surprising, since it is a catalyst used in the polymerization of polyesters. Although samples #11 and 12 were supposed to be the same formulation except for the fire retardant, the detection of a calcium compound in sample #12 was indicative of some differences.

3.2 Rates of Heat Release and Ignition Times

As shown in Table 1, there is no significant difference in the average heat release rate among the four polyurethane foam specimens. There is, however, some difference in ignition times. The two fire retarded samples (#11 and 14) take longer to ignite than the untreated ones (#12 and 13) especially at the lowest heating flux. The polyester batting (#15) did not ignite at 25 kW/m^2 and burned at substantially lower rates at 50 and 75 kW/m^2 than the polyurethane foam samples.

3.3 Autoignition Temperatures

The temperature of the cup furnace which caused the samples to flame spontaneously was determined for all five materials. The autoignition temperature for three polyurethane samples (#11, 12, and 13) was 400°C and that of polyurethane sample #14 was slightly lower - 375°C. Therefore, the differences in material composition or the presence of fire retardants did not affect the autoignition temperatures of the polyurethane foam samples. The non-fire retarded polyester material had a higher autoignition temperature (525°C) than the polyurethane samples.

3.4 Gas Analysis and Temperature Measurements

3.4.1 Carbon Dioxide

Detailed analysis of the CO_2 data was not attempted, since the concentration of CO_2 is dependent upon both the material combustion and the animals' respiration rate and the latter changes with the amount and type of irritants present in the combustion atmospheres. A comparison of the CO_2 values recorded in the analytical (A) and animal (R) experiments in Table A1 (appendix, sample #11) in the non-flaming mode at a mass loading of 20.1 mg/ℓ and in the flaming mode at a mass loading of 19.8 mg/ℓ and Table A2 (sample #12) in the non-flaming mode at a mass loading of 19.9 mg/ℓ (analytical-A) and 20.1 mg/ℓ (animals-R) shows the differences in CO_2 between these experiments with and without animals to be 970, 700 and 2060 ppm, respectively. These increased amounts of CO_2 in the animal experiments are attributed to the animals' respiration.

3.4.2 Carbon Monoxide

The average CO concentrations over the 30 minute exposures at each mass loading/chamber volume are shown in Tables A1 - A5 in the attached appendix. These values have been plotted in Figure 5 which shows that a linear relationship fits between the CO concentrations and the mass loadings/chamber volumes for all the materials when decomposed in either the flaming mode or the non-flaming mode.

The average amount of CO generated from the same mass loading of each material differed as follows (Fig. 5): material #15 produced significantly more CO in both the flaming mode and the non-flaming mode than any of the other materials. The lowest average CO above 20 mg/ℓ for polyester (#15) was higher than all the data from the polyurethanes, indicating a statistically significant difference between the two types of materials [20]. Material #11, which is fire retarded, produced more CO per mass loading in the non-flaming mode than any of the other polyurethane materials. Material #14, however, which is also fire retarded, produced the least amount of CO per mass loading in the non-flaming mode. In the flaming mode, the fire retarded polyurethanes produced more CO than their non-fire retarded counterparts. The same statistical procedure as above [20] showed that the average amount of CO generated by material #14 is significantly different from that of materials #12 and 13.

The generation of CO over time from each of the materials at a mass loading of 30 mg/ℓ is shown in Figure 6. The polyester material decomposed in two stages, first producing CO at a rapid generation rate and then at a slower rate. Unlike the polyurethane materials, the polyester continued to generate CO for about 13 minutes in the flaming mode and throughout the 30 minute exposure in the non-flaming mode. The CO from the polyurethane materials leveled off in the first three minutes in the flaming mode and between 6 and 10 minutes in the non-flaming mode.

The average CO concentrations and carboxyhemoglobin (COHb) values calculated at the LC_{50} concentrations for the 30 minute exposure and 14 day post-exposure period are shown in Table 2. For the polyurethane materials, the CO levels ranged from 750 ppm (#11, non-flaming) to 1400 ppm (#14, flaming). Carboxyhemoglobin (COHb), the molecule which forms in the blood when the animals are exposed to CO, ranged from 10-66%. The polyester produced even higher CO and COHb concentrations at the LC_{50}; the CO concentrations were 2650 and 3000 ppm in the non-flaming and flaming mode, respectively; COHb levels were about 85%.

3.4.3 Hydrogen Cyanide

The results in Table 2 show that the levels of HCN generated at the LC_{50} (30 minutes and 14 day) concentrations for each polyurethane foam in the non-flaming mode were very low, i.e., less than 10 ppm. The flaming mode produced slightly higher HCN concentrations (30-70 ppm). The highest average value (70 ppm) was observed during the decomposition of polyurethane foam #14. The decomposition of 35 mg/ℓ of polyester did not produce any HCN in either the flaming or non-flaming mode. Therefore, HCN was not routinely measured during the polyester experiments. The average HCN concentrations determined for each experiment are given in Tables A1 - A4 in the appendix.

Other studies conducted at NBS have shown that increased concentrations of HCN can be generated from flexible polyurethane foam if the material is decomposed in two phases (procedure is described in Section 2.2.4. Each of the polyurethane materials in this study was decomposed in this two phase manner to determine if increased HCN levels would be generated and the effect of the fire retardants. Heating the charred residues from the 30 minute non-flaming experiments generated increased HCN concentrations for all the polyurethane samples (Fig. 7). Polyurethane foam #12 generated the least HCN - 120 ppm. Polyurethane #11, the fire retarded counterpart to #12, generated 210 ppm HCN, almost double the amount for sample #12. Non-fire retarded #13 peaked at 180 ppm HCN, whereas its fire retarded counterpart, #14, peaked at 220 ppm. The fire retarded materials produced more HCN in this two phase decomposition procedure than their non-fire retarded counterparts.

3.5 Toxicological Results

3.5.1 LC_{50} Results

The lowest LC_{50} values observed for the polyurethane foams were 17 mg/ℓ for #11 (non-flaming conditions) and 28 mg/ℓ for #14 (flaming conditions). All the other polyurethane LC_{50} values either approached or exceeded 40 mg/ℓ (Table 2). The LC_{50} values of the polyester fiberfill material #15 in the flaming and non-flaming modes were 28 and 31 mg/ℓ, respectively. Based on a comparison of the 95% confidence limits, the LC_{50} of material #11 is significantly different from that of materials #12 and 13, but not that of #14 or 15. However, these LC_{50} values were not considered toxicologically different from each other based on the classical toxicological definition which uses a factor of 10 to determine differences in degrees of toxicity [21].

The only within-exposure deaths observed during this study occurred during the flaming combustion of the fire retarded polyurethane foams, #11 and #14, and during the non-flaming and flaming combustion of the polyester fiberfill, #15. The non-fire retarded foams, #12 and #13, caused no within-exposure deaths.

Deaths were observed during the post-exposure period after the rats were exposed to the non-flaming decomposition products from materials #11, 12, 13 and 15 and to the flaming decomposition products from the fire retarded polyurethanes #11 and 14, and from the polyester, #15. Many of the deaths observed in the post-exposure period occurred after the animals lost a great amount of weight and after a number of days had passed. The exceptions were polyurethane foam #14 and polyester #15 decomposed in the flaming mode. In these two cases, all post-exposure deaths occurred on the same day as the experiments.

3.5.2 Post-Exposure Effects

All the animals that survived the exposures and were not cannulated to provide blood during the exposures were kept and weighed daily for at least 14 days. The weight profiles provide one indication of the post-exposure health of the animals. In this study, the following questions regarding the post-exposure weights of the animals were addressed:

A. Did placing the animals in restrainers and exposing them to atmospheres heated by different furnace settings affect the control animals' weight profiles?

B. Was the weight loss experienced by the experimental animals correlated with mass loading of material that was thermally decomposed?

C. Which decomposition mode - flaming or non-flaming - produced the most severe effects on the post-exposure weights of the animals?

D. Did the presence of the fire retardant affect the weight profiles of the animals?

E. In a comparison of all the materials, what differences were seen in the animal weight profiles after exposure to the combustion products from a specified mass loading?

F. How does the individual animals' weight data differ from the mean weight of the surviving animals in an experiment?

Representative graphs are shown in Figures 8-30. In these graphs, the weight of the animals on the day of the experiments have been normalized to 0 to allow a visual comparison of the weight gain or loss due to the different exposure conditions. Standard deviations of the mean weights are shown in some cases to indicate the spread of the results. It should also be noted that occassional rapid increases in the mean weight may be due to the death of the animal with the lowest weight.

3.5.2.1 Effect of Restrainer and Head-Only Exposure

Figure 8 shows the weight profiles for four sets of control animals. In these cases, the furnace temperature was set at 425°C, 550°C, 600°C, and 800°C, and the animals were placed in their restrainers and exposed in the head-only mode to the atmosphere in the closed exposure chamber. No materials were burned. Although the furnace was set at the above temperatures, the average temperatures that the animals experienced ranged from 28°C to 47°C over the 30 minute exposure period. These four control groups show that the physical stress of being placed in the restrainer and being exposed to a non-toxic atmosphere could cause the animals to lose some weight during the first few days of the post-exposure period. The degree of this initial weight loss did not correlate with the furnace temperature setting. In the worst case at 425°C, the animals lost 27 grams the first day but were back to their original weight by day 5.

3.5.2.2 Correlation of Weight Loss and Amount of Material Decomposed

Figure 9 shows the weight loss experenced by the animals following exposure to various concentrations of material #11 which was decomposed in the non-flaming mode. The control animals shown are those (also shown in Fig. 8) which were exposed to an atmosphere where the furnace temperature was set at 425°C. A concentration-response relationship is apparent as the animals lose more weight than the controls at the higher mass loadings (17.5 and 20 mg/ℓ). At the highest mass loading, 30 mg/ℓ, all the animals died between day 1 and day 2. Figure 10 shows the weight profiles and their standard deviations of the controls and the animals exposed to 17.5 mg/ℓ (approximately the LC_{50}) of material #11. After day 5, there is no overlap of the standard deviations indicating the divergence of the two curves.

A similar correlation between weight loss and concentration of combustion products was apparent from the non-flaming weight results from material #12 (Fig. 11). This relationship between weight loss and mass loading/chamber volume was not apparent for materials #13 and 14 in the non-flaming mode, although in both cases, the exposed animals lost more weight than the untreated controls (Fig. 12 and 13). The polyester material #15 also showed a correlation between weight loss and concentration of combustion products in the non-flaming mode (Fig. 14 and 15).

In the flaming mode, the correlation between weight loss and mass loading was not evident for any of the materials. The flaming data for materials #11 and 12 are shown in Figure 16, and that for materials #13, 14 and 15 are in Figures 17, 19, and 20, respectively.

3.5.2.3 Effect of Flaming or NonFlaming Decomposition

Regardless of the material tested, the combustion products from the non-flaming mode produced greater effects on the weight profiles of the animals than those from the flaming mode. This is most clearly illustrated in Figure 17 which shows the obvious difference in weight loss after the animals were exposed to either the flaming or non-flaming combustion products from material #13. In the non-flaming mode, the animals lost approximately 30 grams in the first two days following exposure regardless of the concentration of material decomposed. In the flaming mode, the greatest weight loss was 5.5 grams on day 2. The mean weights and their standard deviations of the animals after exposure to 40 mg/ℓ of material #13 showed no overlap of the standard deviations only on day 2 when the animals exposed in the non-flaming mode lost the most weight (Fig. 18). This indicates that once the animals start to recover, there is no significant difference between the experimental and controls. Results following exposure to the other materials are shown in Figures 19 (material #14), 21 (material #11), 22 (material #12), and 23 (material #15).

3.5.2.4 Effect of the Fire Retardants

Figure 24 compares samples #11 and #12 at comparable concentrations after decomposition in the non-flaming mode. Material #11, the fire retarded material, produced a greater toxicological effect on both post-exposure weights and deaths than #12, the corresponding untreated foam. For example, none of the animals exposed to these concentrations of material #12 died during the post-exposure period. However, all the animals exposed to these concentrations from material #11 did die during the post-exposure period. The higher the concentration, the sooner the deaths occurred. Comparison of mean weights and standard deviations of animals exposed to 20 mg/ℓ of materials #11 and 12 decomposed in the non-flaming mode showed no overlap of the standard deviations between material #11 and the control data (Fig. 25). Material #12 did overlap with the control.

Although the LC_{50} values in Table 2 were the same, the weight profiles of the animals after exposure to samples #11 and #12 in the flaming mode indicate that exposure to material #11 was generally more severe than exposure to #12 (Fig. 16). The differences are not as obvious as in the non-flaming mode.

Figure 26 compares the non-flaming results for materials #13 (non-fire retarded) and #14 (fire retarded). Both materials show the same initial weight loss. The animals exposed to material #13 show an attempt to recover about days 5 and 6 and then again lose weight, after which some recovery is seen. The higher concentrations of material #14 appear to cause the animals to recover more slowly than seen with material #13; however, this may be an artifact due to no data at the time the recovery would have occurred. These results at the highest mass loading/chamber volume tested (40 mg/ℓ) were plotted along with their standard deviations (Fig. 27). The control data is incomplete between days 4-6 but the remaining results indicate that no overlap of the standard deviations occurs on days 2 and 3 following the exposure and that by day 7, there is no difference between the exposed and control animals. The flaming results from materials #13 and #14 did not show any significant differences (Fig. 28).

3.5.2.5 Weight Profiles Following Exposure to All the Materials at One Mass Loading

Figure 29 compares the non-flaming results for materials #11, 12, 13, 14, and 15 at 30 mg/ℓ. The decomposition products from #11 caused all the animals to die on day 1. The other materials all caused about the same initial weight loss for the first two days, after which a period of recovery is noted (except #14) for three days. The animals then either continue to recover (#15) or experienced additional weight losses before starting to gain weight. The only material other than #11 to cause deaths in these illustrated experiments was #13 which caused one death on day 11.

3.5.2.6 Weight Data of Individual Animals

In each experiment, six animals were exposed to the combustion products and all surviving animals, which were not sacrificed for blood, were weighed daily. In all the above cases, the mean weights of the remaining animals were calculated and plotted. However, since individual animals died at various times, Figure 30 illustrates each individual animal's weight loss and day of death after exposure to 20 mg/ℓ of material #11. All the animals reacted in a similar way, i.e., they initially lost weight for two days, appeared to recover through day 6 or 7, and then started losing weight again until they died. In this experiment, the greatest weight loss was 91 grams.

3.5.3 10-Minute Tests

Ten minute tests were performed for polyurethane #11 in the non-flaming mode where the LC_{50} was 17 mg/ℓ, and for polyurethane #14 in the flaming mode where the LC_{50} was 28 mg/ℓ. In both cases, no animals died either during the exposure or during the 14 day post-exposure period. The LC_{50} for the polyester fiberfill in the non-flaming mode was 28 mg/ℓ, but since 30 minute experiments had been carried out with this material at 30 mg/ℓ and 32 mg/ℓ and less than 50% of the animals had died either within or post-exposure, it was concluded that a 10 minute exposure to 30 mg/ℓ would also not produce 50% deaths.

4.0 DISCUSSION

The purpose of this study was to compare upholstered furniture filling materials, in particular two different formulations of flexible polyurethane foam and a polyester fiberfill. Each formulation of polyurethane foam was supplied as two samples, one of which contained a fire retardant. Therefore, a total of five samples were evaluated. These samples were coded as polyurethane #11, 12, 13, and 14 and polyester #15.

The time-to-ignition was examined under three heat fluxes, 25, 50, and 75 kW/m^2, and the results indicated that the two fire retarded polyurethane foam specimens (#11 and 14) take longer to ignite than the untreated ones (#12 and 13), expecially at the lowest heating flux. This also suggests that over a certain range of heating fluxes, the flame spread might be slower over the fire retarded samples than over the untreated samples. This advantage would diminish at higher fluxes. At the lowest heat flux tested, the polyester fiberfill did not ignite and the times-to-ignition were longer at the higher heat fluxes than those noted for any of the polyurethane foams.

The rate of heat release data showed no significant differences among the four polyurethane samples. In other words, once ignited, the burning rates of these samples are almost identical. The polyester fiberfill's rate of heat release was lower than any of the polyurethane samples at any given heat flux. However, it should be noted that the actual packing density of a fiberfill can vary widely and the density of the polyester packing would affect the heat release rate. Nonetheless, it is clear that, in comparison to these polyurethane samples, this polyester is less ignitable and has a lower heat release rate.

The autoignition temperatures which were determined as part of the NBS toxicity test method (400°C for polyurethane foams #11, 12 and 13, 375°C for polyurethane foam #14 and 525°C for the polyester) indicated that the autoignition temperatures were not affected by the presence of the flame retardants. This value is determined under different conditions than the ignition time. The autoignition temperature is found by placing the sample in a quartz cup which has been preheated to different temperatures and determining the temperature which causes the material to flame. The ignition time is determined by exposing a sample to different heat fluxes and measuring the time at which it ignites. Since the autoignition temperatures were similar and little difference was observed in times-to-ignition at high heat fluxes, the cup furnace exposure appears comparable to the higher heat fluxes used in the ignition time tests.

The polyester fiberfill, which was the least ignitable at the high heat flux exposures, had the highest autoignition temperature.

Pure gas studies performed at NBS have shown that the LC_{50} for a 30 minute CO exposure is 5000 ppm (when the animals are exposed gradually, i.e., the CO is introduced into the exposure chamber over a 5 minute period) or 4600 ppm (when the animals are exposed immediately). Carboxyhemoglobin, the molecule that forms in the blood when the animals are exposed to CO, will reach 87-89% before the animals die. For these polyurethanes, the CO concentrations at the LC_{50} ranged from 750 ppm to 1400 ppm and for this polyester, the CO concentrations were 2650 ppm and 3000 ppm at the LC_{50}'s in the non-flaming and flaming modes, respectively. The highest COHb levels noted in this study ranged from 10% to 66% for the polyurethane materials and about 85% for the polyester. These results indicate that the CO and COHb levels generated at the LC_{50} (30 minutes and 14 day) concentrations of the polyurethane foam were not sufficient to account for the deaths of the animals. Although the CO levels generated at the LC_{50} value for the polyester were lower than the lethal levels seen in the pure gas studies, the COHb levels were high enough (85%) to consider CO as the primary toxicant.

Another factor to consider is that most of the deaths observed in this study occurred post-exposure. (Exceptions were polyurethane #14 in the flaming mode in which half the animals died the same day of the experiment and polyester #15 in which most of the deaths occurred during the exposure.) The NBS pure CO studies have shown that if the animals survive the exposure, they will not succumb during the post-exposure period. Therefore, the post-exposure deaths should not be attributed to CO.

HCN can be generated during the thermal decomposition of any nitrogen-containing polymer. Therefore, HCN concentrations were measured during the decomposition of the polyurethane samples, but not the polyester material. Pure gas studies at NBS have indicated that approximately 160 ppm of HCN for 30 minutes are necessary to kill 50% of the animals during exposure to the NBS toxicity test method conditions. Examination of the HCN results presented in Table 2 shows that the HCN levels ranged from less than 10 ppm to 70 ppm and therefore were never high enough to account for the within-exposure deaths. Although CO and HCN are probably contributing to the within-exposure deaths of the animals exposed to polyurethane combustion products, there appear to be additional factors or toxic gases which are not yet understood. Also the reasons for the post-exposure deaths are still unclear.

This study also has shown that increased amounts of HCN can be generated from the polyurethane samples if they are decomposed according to a two phase procedure developed at NBS. This two phase decomposition procedure simulates a real fire scenario, namely, that of a polyurethane upholstered chair which smolders for some unspecified time forming a char and eventually ignites. These results indicated that the fire retarded samples produced more HCN than the untreated specimens. Although no animals were exposed in this series of experiments, it is clear that the LC_{50} (30 min) levels of pure HCN were reached in the case of materials #11, 13, and 14.

The materials examined in this study should not be rank ordered based on the calculated LC_{50} values. Toxicologically, the LC_{50}'s should differ by a factor of 10 before any one is considered significantly more toxic than another [21]. In this study, the LC_{50}'s were not found to be toxicologically different. However, it is interesting to note that the non-fire retarded polyurethane foams (#12 and 13) produced only post-exposure deaths and those only in the non-flaming mode. The fire retarded foams (#11 and 14) produced within-exposure deaths but only in the flaming mode. The post-exposure deaths that resulted from the fire retarded foams were in the non-flaming mode (#11) and flaming mode (#11 and 14). The production of within-exposure deaths from the fire retarded foams only is indicative of a rapidly acting toxicant (within 30 minutes) being generated by the flaming combustion.

The 10 minute tests designed to determine whether these materials rapidly produced effective concentrations of toxicants were performed for the two materials not previously tested for 30 minutes at 30 mg/ℓ. These materials did not rapidly produce (within 10 minutes) lethal levels of toxicants at those concentrations.

The weight profiles of the animals after exposure to the combustion products of the materials were also evaluated in this study. The greater toxicological effects on the animals by the fire retarded material #11 was reflected by increased weight loss and less rapid recovery than seen with material #12 which was untreated. The combustion products from materials #13 and #14 appeared to have about equal effects on the post-exposure weights of the animals.

In addition, the weight profiles indicated that the non-flaming conditions caused greater post-exposure effects than the flaming conditions. After exposure to the non-flaming conditions, the animals lost weight in the first few days, appeared to stabilize or recover slightly and then, about day 5-7, began to lose weight again. Material #15, the polyester, did not produce the second weight loss. It was during this second period of weight loss that most of the deaths from the polyurethanes occurred. The deaths that did occur during the first few days are probably directly related to the insult caused by the toxic exposure. Some of the control data indicates that, in some cases, the initial weight loss may be due to the physical stress of being restrained and not the chemical stress of the toxicants. The weight loss and deaths that occur later, however, are most likely related to the toxic insult which caused physiological damage or increased susceptibility to secondary infection.

5.0 CONCLUSIONS

As a result of this study, the following conclusions can be stated:

- The LC_{50} (30 minute and 14 day) values did not distinguish any of the test materials as being toxicologically different from the the others. The worst case fire retarded material #11 was only a factor of two more toxic than its untreated counterpart.

- The fire retarded polyurethane foam decomposition products caused within-exposure deaths. No such deaths were observed when the non-fire retarded polyurethane samples were burned.

- Exposure to the combustion products of these materials caused extensive weight loss of the animals during the post-exposure period and, in many cases, death. The non-flaming mode produced greater weight losses than the flaming mode. The effect of stress on the animal weights during the early post-exposure period cannot be clearly separated from the effect of the toxicants.

- The physiological explanation for the late-occurring post-exposure deaths is not known.

- The animal deaths from exposure to the combustion products from the polyurethane foams tested cannot be attributed to CO or HCN alone.

- Animal deaths due to the combustion products of the polyester were generally within-exposure and appear to be more directly related to the CO concentrations.

- The greatest concentration of CO was generated by the polyester regardless of the combustion mode (flaming or non-flaming). The fire retarded polyurethanes generated more CO in the flaming mode than the non-fire retarded polyurethanes. This difference was not observed in the non-flaming mode.

- Increased concentrations of HCN can be generated from the polyurethane foams by a two phase decomposition procedure. The fire retarded materials produced even more HCN than the untreated samples.

- The rate of heat release for the polyurethane materials increased with increased heat flux and was comparable for all the foams at a given heat flux. The polyester burned at lower rates when compared to the polyurethane foams at a given heat flux.

- The autoignition temperatures of the polyurethane foams #11 (fire retarded), 12, and 13 were the same. Number 14 (fire retarded) was 25°C lower than the other polyurethane foams. Therefore, fire retardants did not affect the temperature at which the samples flamed. The polyester was 125°C higher indicating lower ignitability.

- The fire retarded polyurethane samples had longer times-to-ignition than the untreated samples at the lowest heat flux tested. At higher heat fluxes, the times were comparable. The polyester had the longest time-to-ignition at all heat fluxes tested.

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Table 1

Ignition Times and Rate of Heat Release

Material	Heating Flux (kW/m^2)	Ignition Time (sec)	Peak Heat Release (kW/m^2)	60 sec. Average Heat Release (kW/m^2)
11	25	39.2	438	276
	50	4.1	1029	456
	75	2.7	1429	545
12	25	5.5	433	278
	50	3.3	1059	443
	75	1.3	1773	501
13	25	5.2	466	272
	50	3.3	876	470
	75	NA	1810	646
14	25	15.0	467	230
	50	4.1	844	428
	75	2.9	1862	561
15	25	∞	0	0
	50	10.2	247	149
	75	9.2	259	129

Table 2

Toxicological and Gas Results from NBS Toxicity Test Method

Material	AIT[1] (°C)	Mode	LC_{50} (30 min + 14 days)		Concentration-Response		COHb[2]		Average Gas Concentrations[3] at LC_{50}		No. Died		Latest Day of Death	10 Min. Test
			(mg/ℓ)	95% Confidence Limits	Slope	95% Confidence Limits	Highest 30 min Value (%)	Conc. Tested (mg/ℓ)	CO (ppm)	HCN (ppm)	No. Not Within Exposure Only	Sacrificed Post Exposure Only		
Poly-urethane # 11	400	NF	17	13-22	1.4	1.1-1.9	45.9	20.1	750[3]	<10	0/29	12/23	16	passed[5]
		F	>40	---	---	---	59.5	40.0	>1350[4]	>70[4]	2/24	2/23	8	NR
Poly-urethane # 12	400	NF	38	37-39	1.0	0.9-1.1	56.5	38.2	1050[4]	<10	0/36	13/26	14	NR
		F	>40	---	---	---	34.5	40.0	> 600[3]	>40[4]	0/18	0/12	NA	NR
Poly-urethane # 13	400	NF	37	30-46	1.4	1.0-2.0	47.0	35.0	300[3]	<10	0/30	10/25	14	NR
		F	>40	---	---	---	46.5	40.0	> 850[4]	>30[4]	0/18	0/16	NA	NR
Poly-urethane # 14	375	NF	>40	---	---	---	10.1	39.9	> 400[4]	<10	0/24	0/23	NA	NR
		F	28	23-33	1.3	1.0-1.8	65.8	28.0	1400[3]	70[3]	5/30	5/24	0	passed[5]
Poly-ester # 15	525	NF	28	22-34	1.7	0.9-3.1	85.6	37.1	2650[3]	ND	12/48	7/41	19	NR
		F	31	28-34	1.3	1.0-1.6	84.8	33.0	3000[3]	ND	21/54	1/48	0	NR

1. Autoignition temperature
2. Carboxyhemoglobin
3. Average concentration for 30 minutes determined at LC_{50} by least squares linear regression analysis
4. If no LC_{50} was determined, then value at highest loading tested is given
5. No animals died

NF: Non-flaming
F : Flaming
NR: Not required
ND: Not determined as material did not contain nitrogen

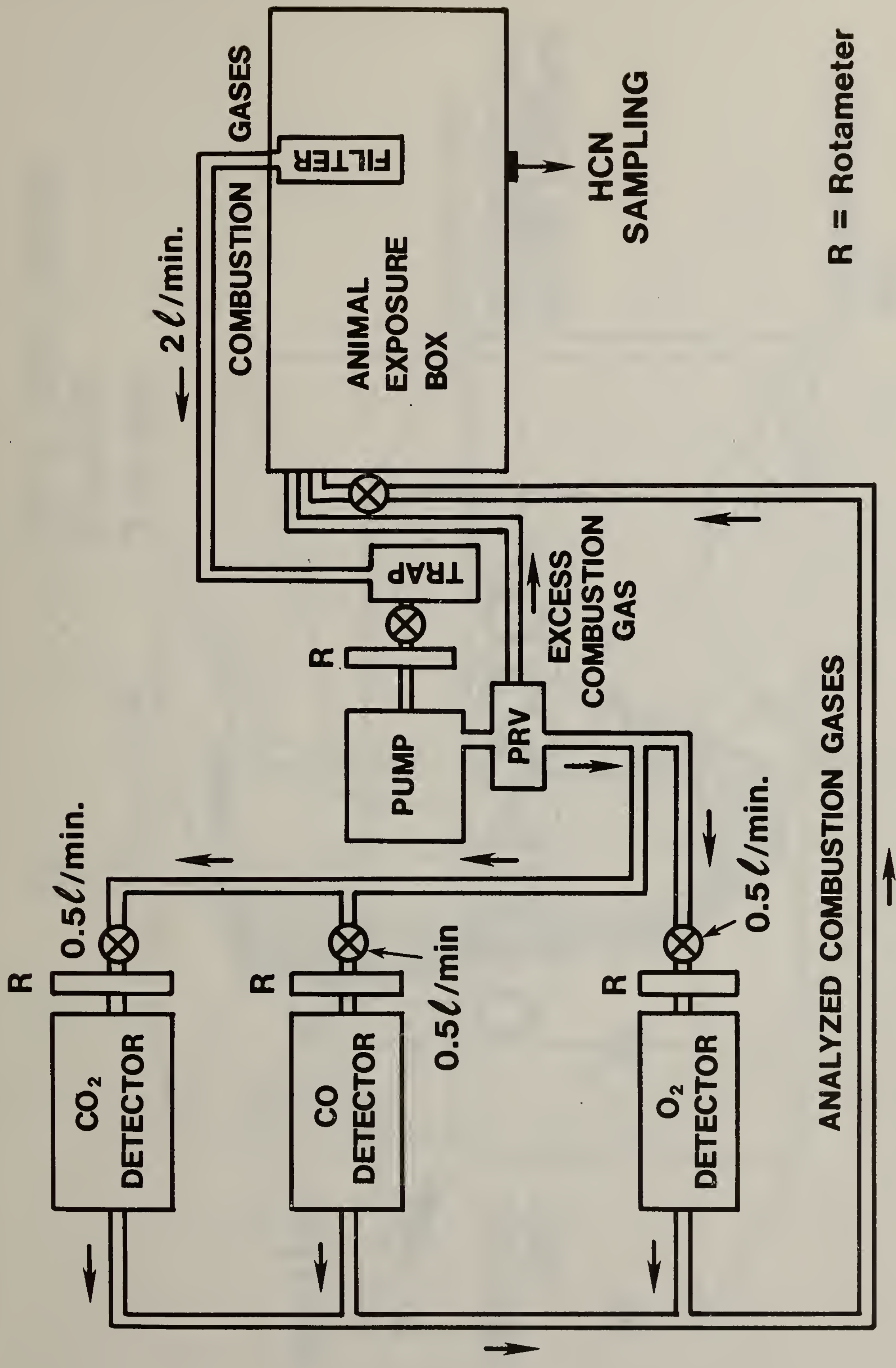

Figure 1. Schematic of NBS gas analysis system.

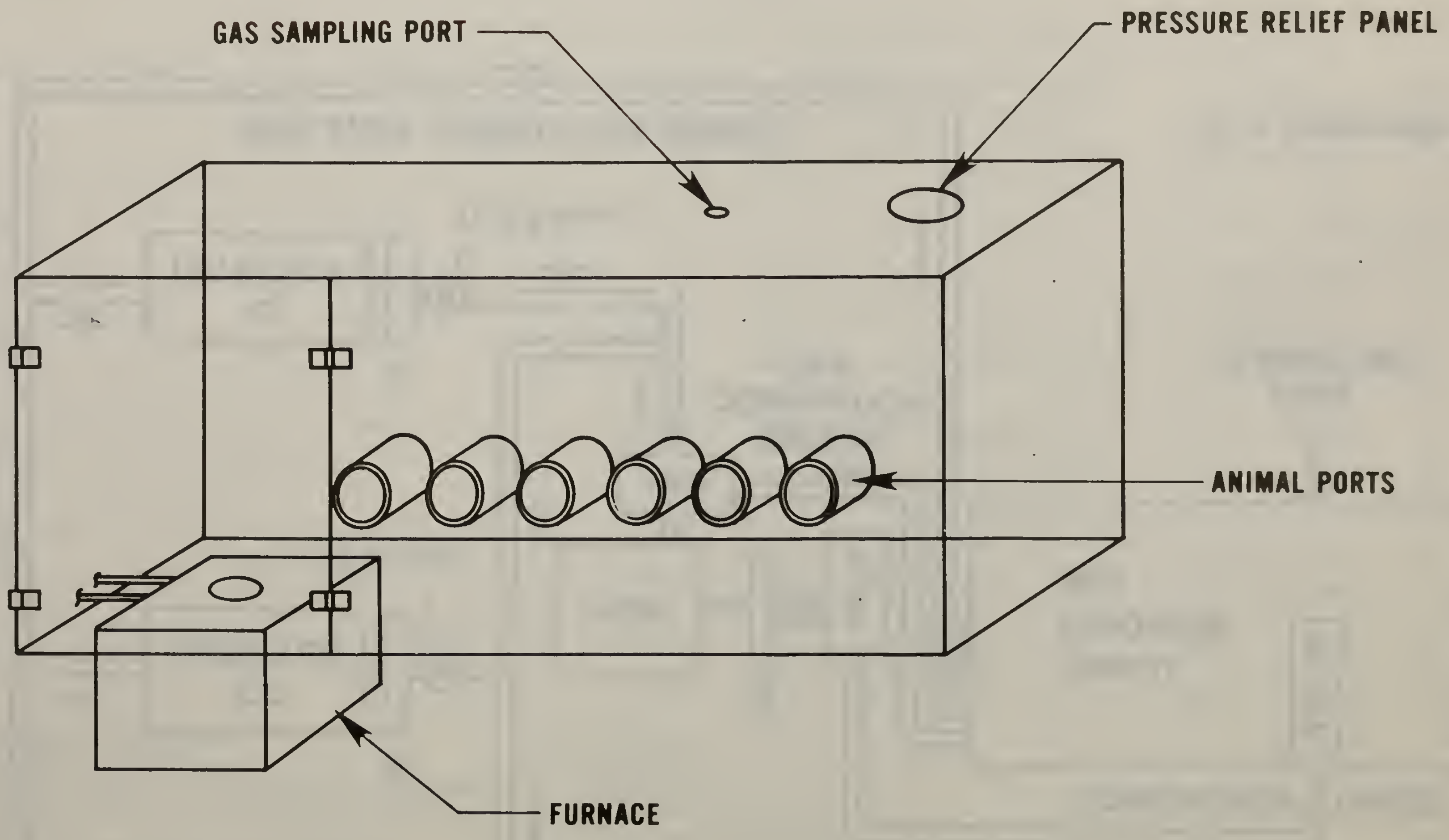

Figure 2. Exposure chamber.

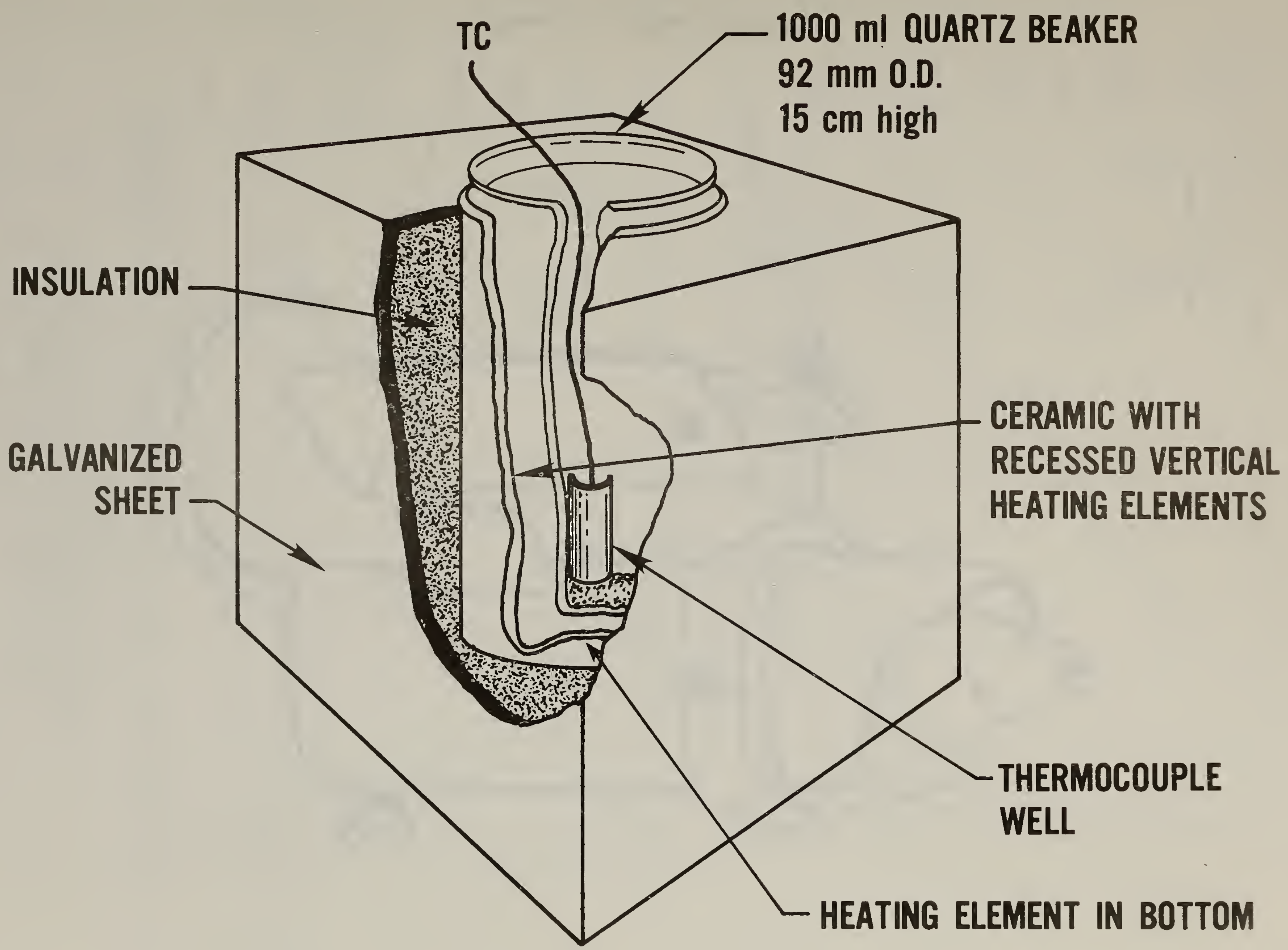

Figure 3. Pyrolysis/combustion furnace.

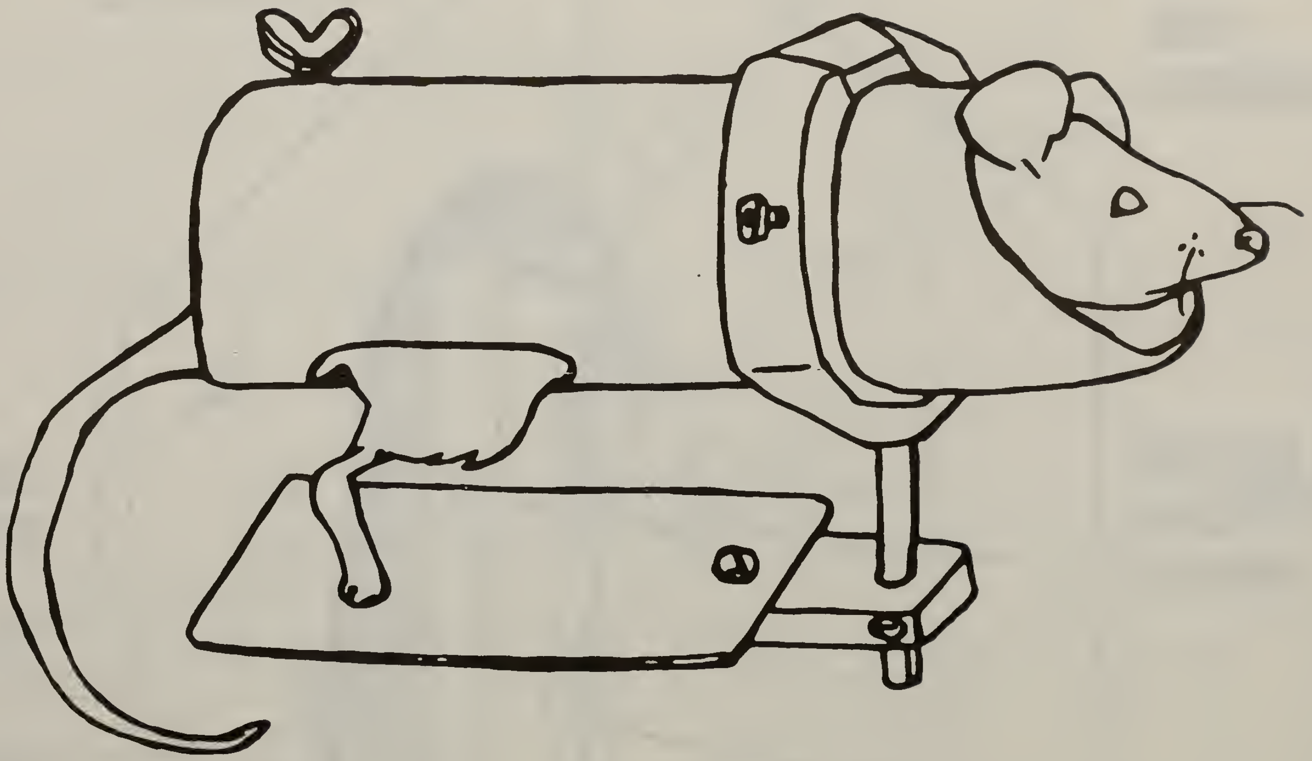

Figure 4. Animal restrainer.

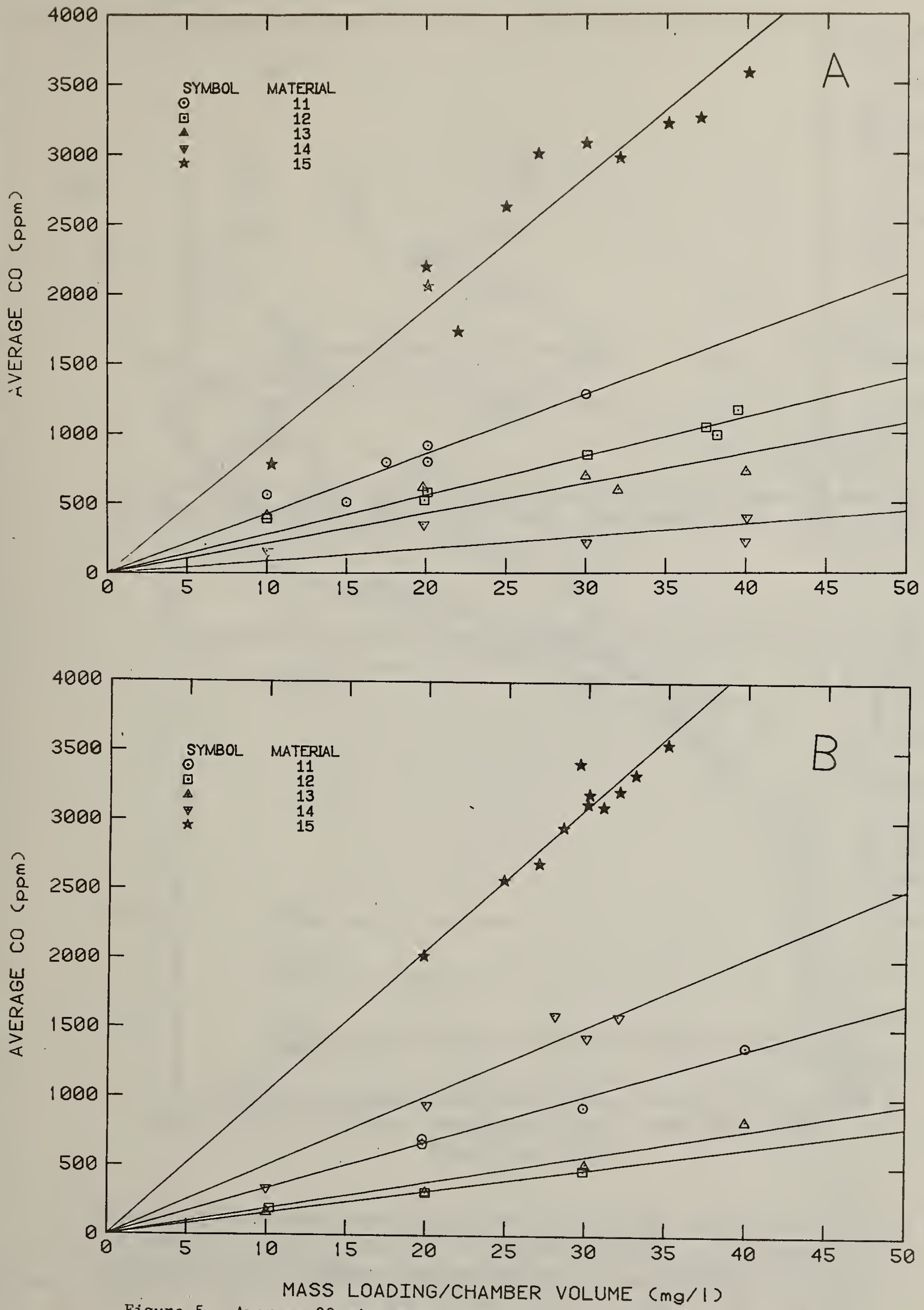

Figure 5. Average 30 minute carbon monoxide concentrations (ppm) as a function of the mass loading/chamber volume (mg/ℓ).
A. - Non-flaming results. B. - Flaming results.

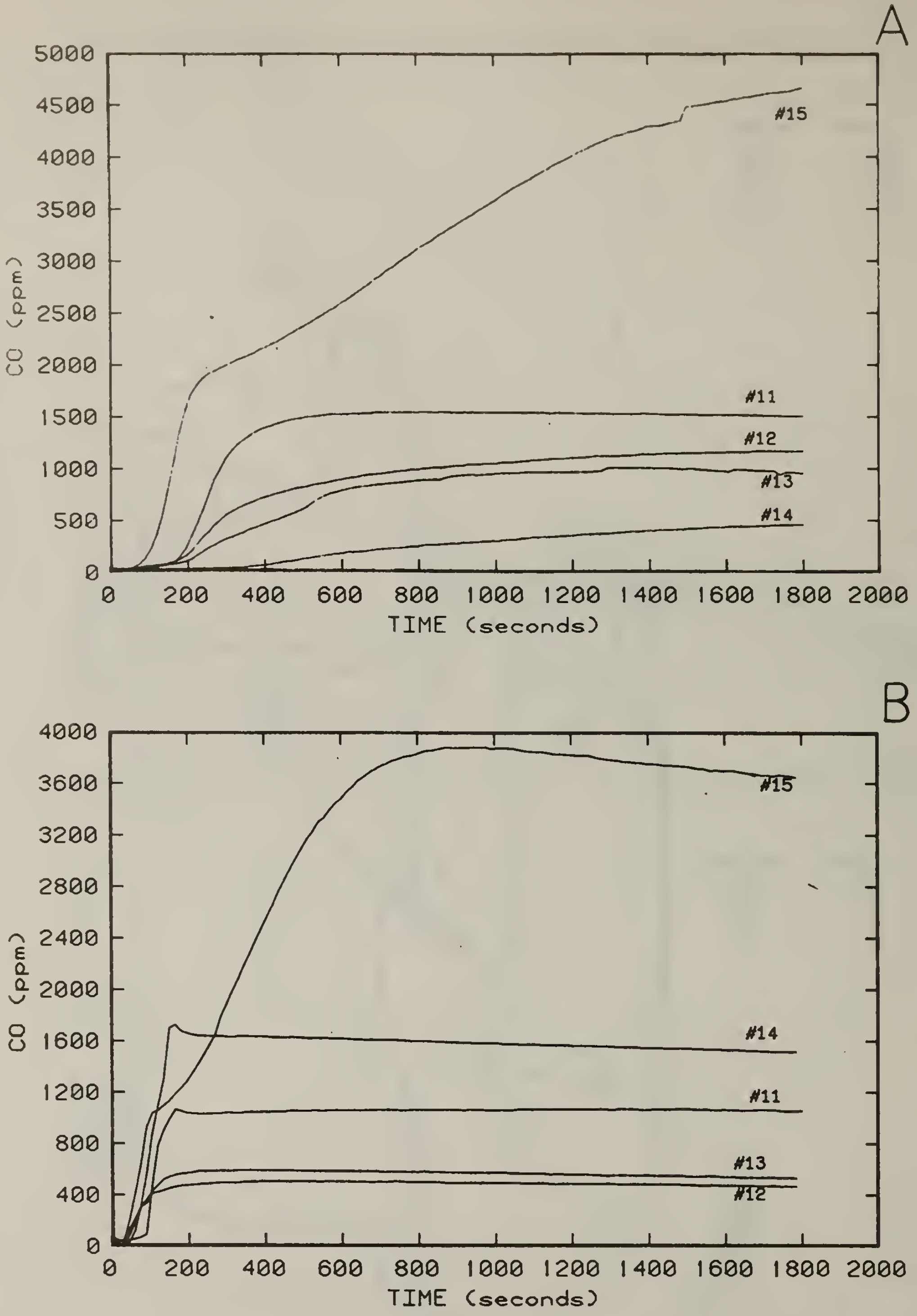

Figure 6. Rate of carbon monoxide generation (ppm) during decomposition of samples 11, 12, 13, 14 and 15 at mass loading/chamber volume of 30 mg/ℓ. A - Non-flaming results. B - Flaming results.

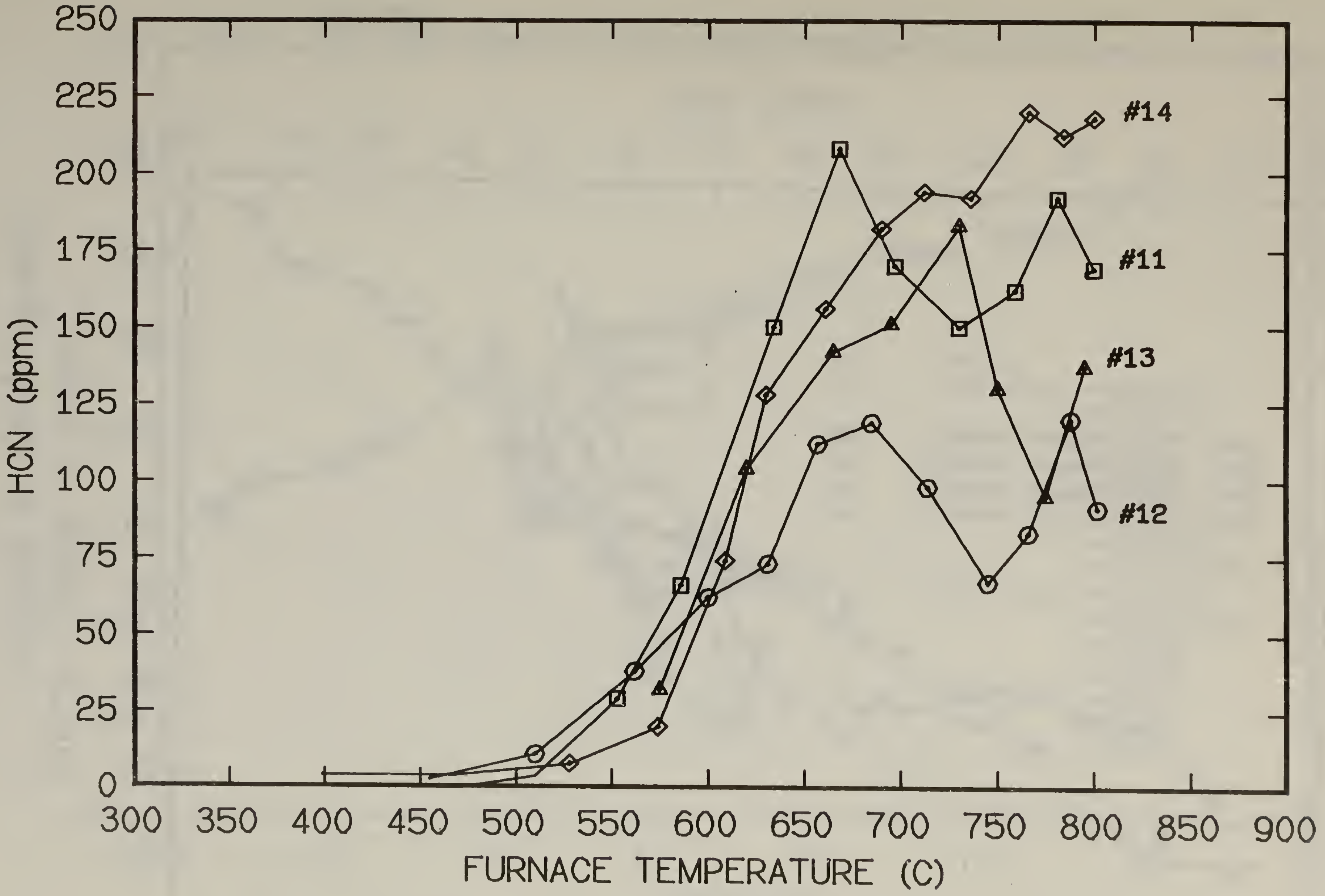

Figure 7. Hydrogen cyanide generation from polyurethane foam charred residues as a function of temperature. The residues resulted from a 30 minute experiment at the non-flaming temperature from a mass loading of 20 mg/ℓ.

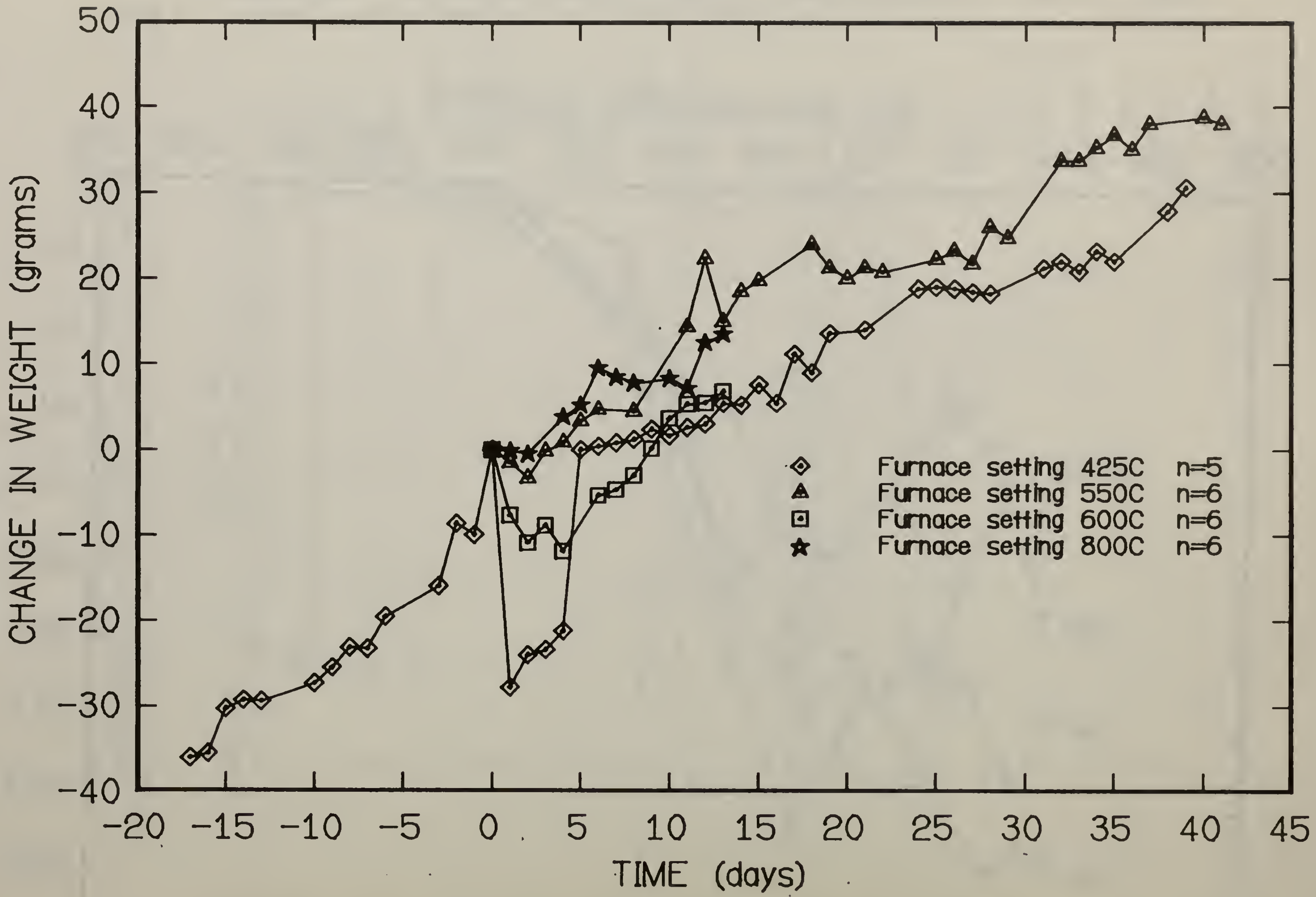

Figure 8. Mean weight profiles of control animals normalized to the initial weight on day of experiment.

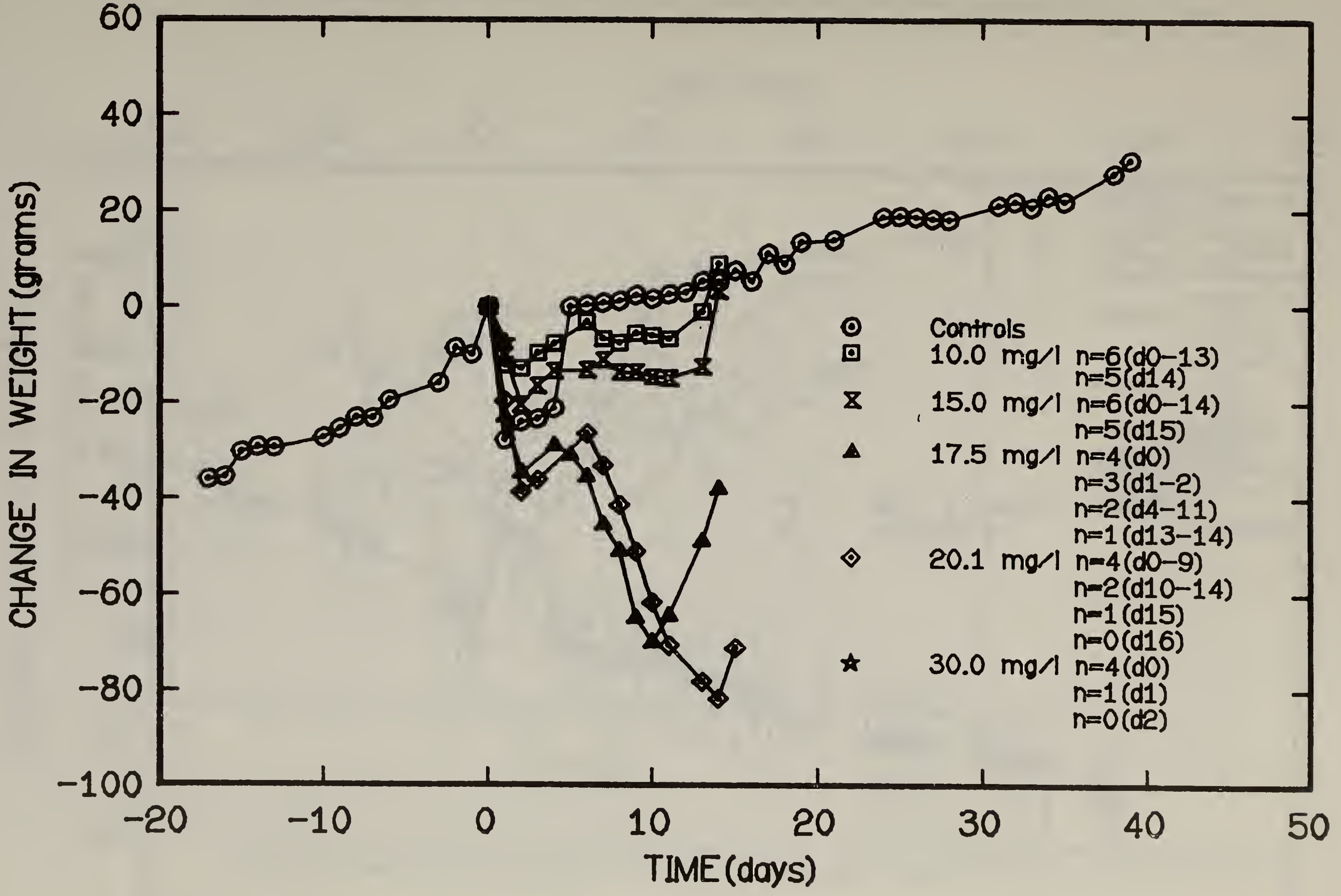

Figure 9. Mean weight profiles of animals exposed to the non-flaming decomposition products of material #11 at various mass loadings/chamber volume (mg/ℓ).

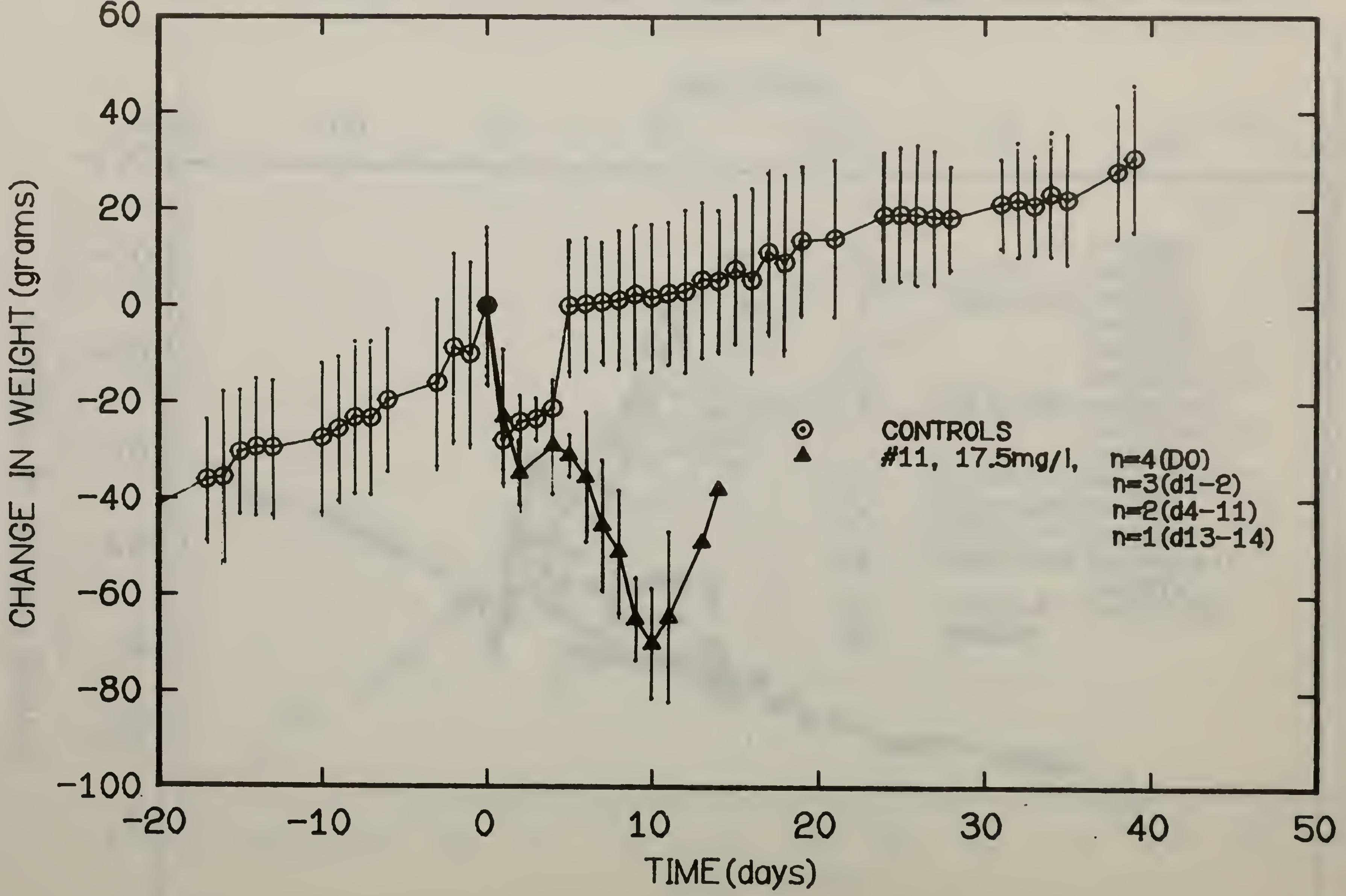

Figure 10. Mean weight profiles and standard deviations of animals exposed to 17.5 mg/ℓ of material #11 decomposed in the non-flaming mode and controls who were restrained and exposed head-only to the chamber atmosphere heated by setting the furnace at 425°C.

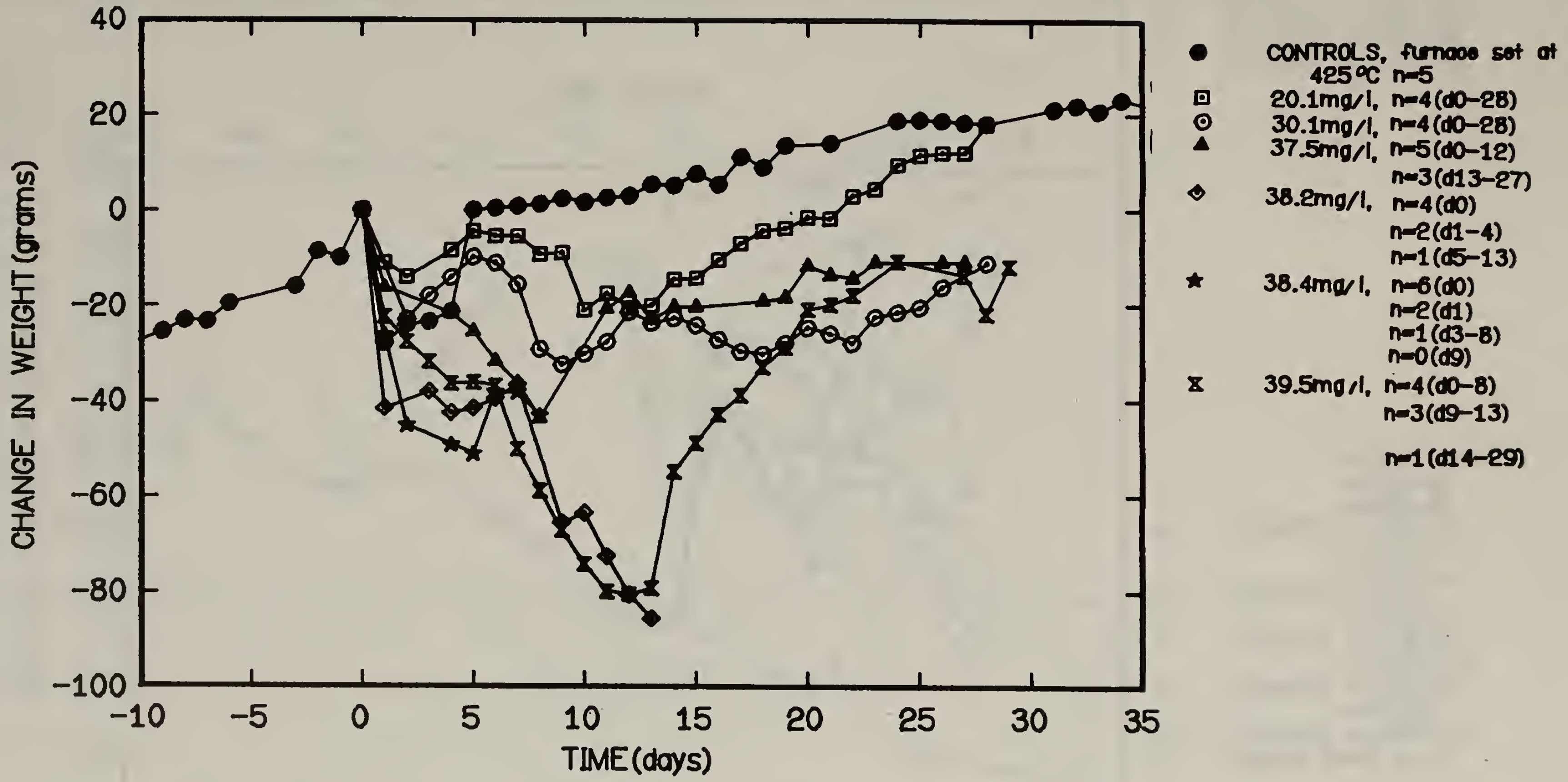

Figure 11. Profiles of the mean weights of animals exposed to the non-flaming thermal decomposition products of sample #12 at various mass loadings/chamber volume (mg/ℓ).

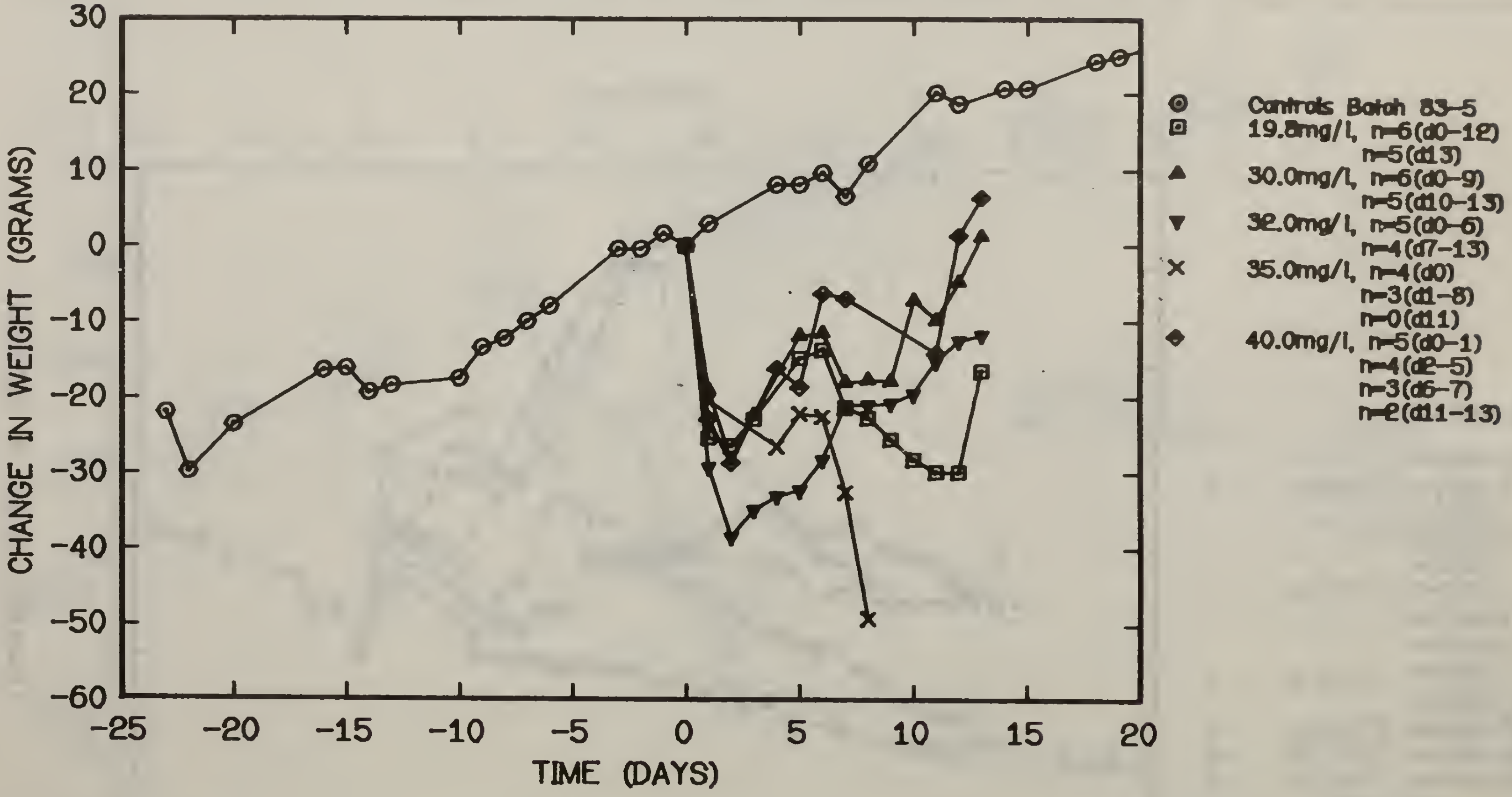

Figure 12. Mean weight profiles of animals exposed to the non-flaming thermal decomposition products of sample #13 at various mass loadings/chamber volume (mg/ℓ).

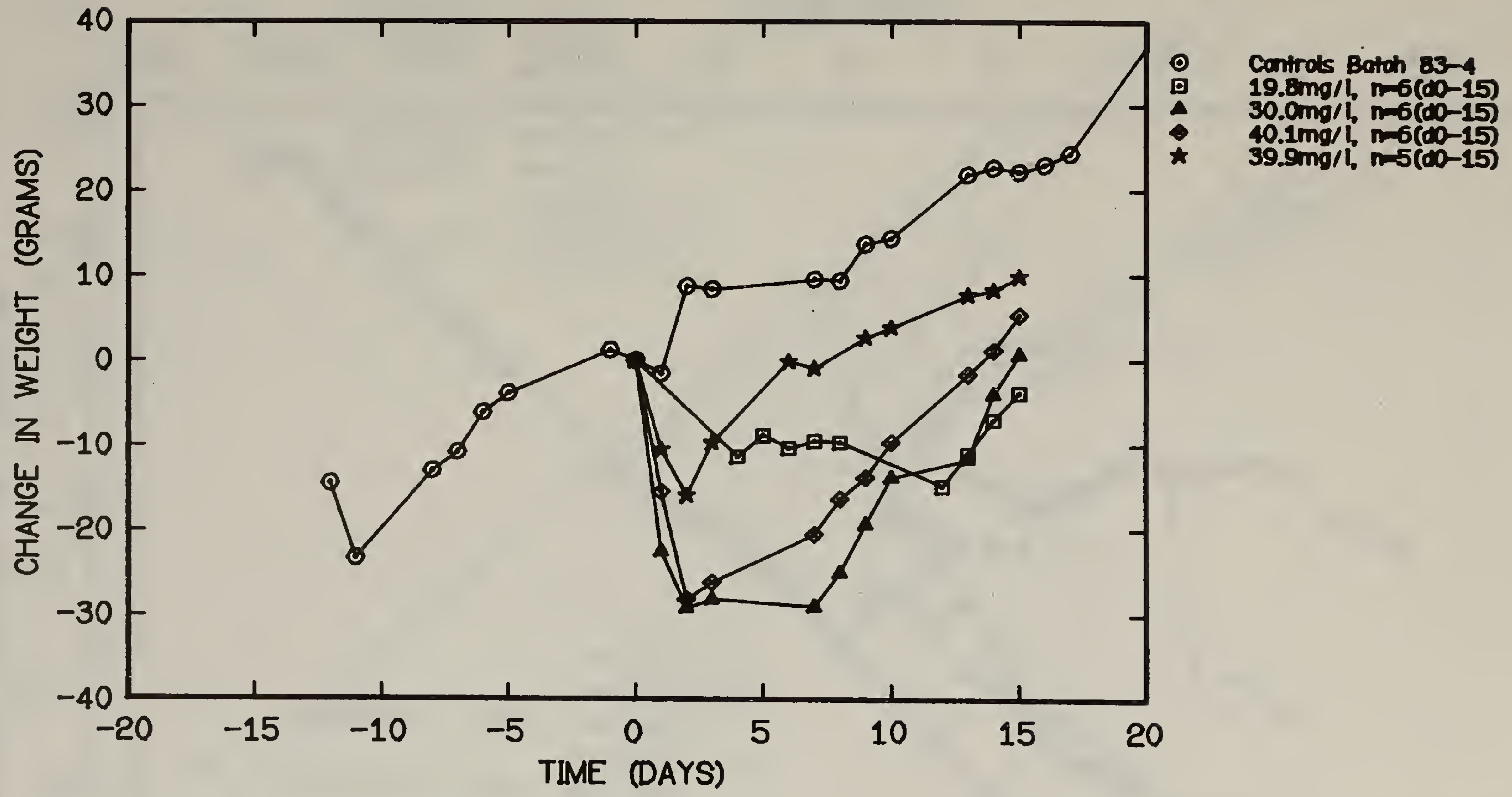

Figure 13. Mean weight profiles of animals exposed to the non-flaming thermal decomposition products from sample #14 at various mass loadings/chamber volume (mg/ℓ).

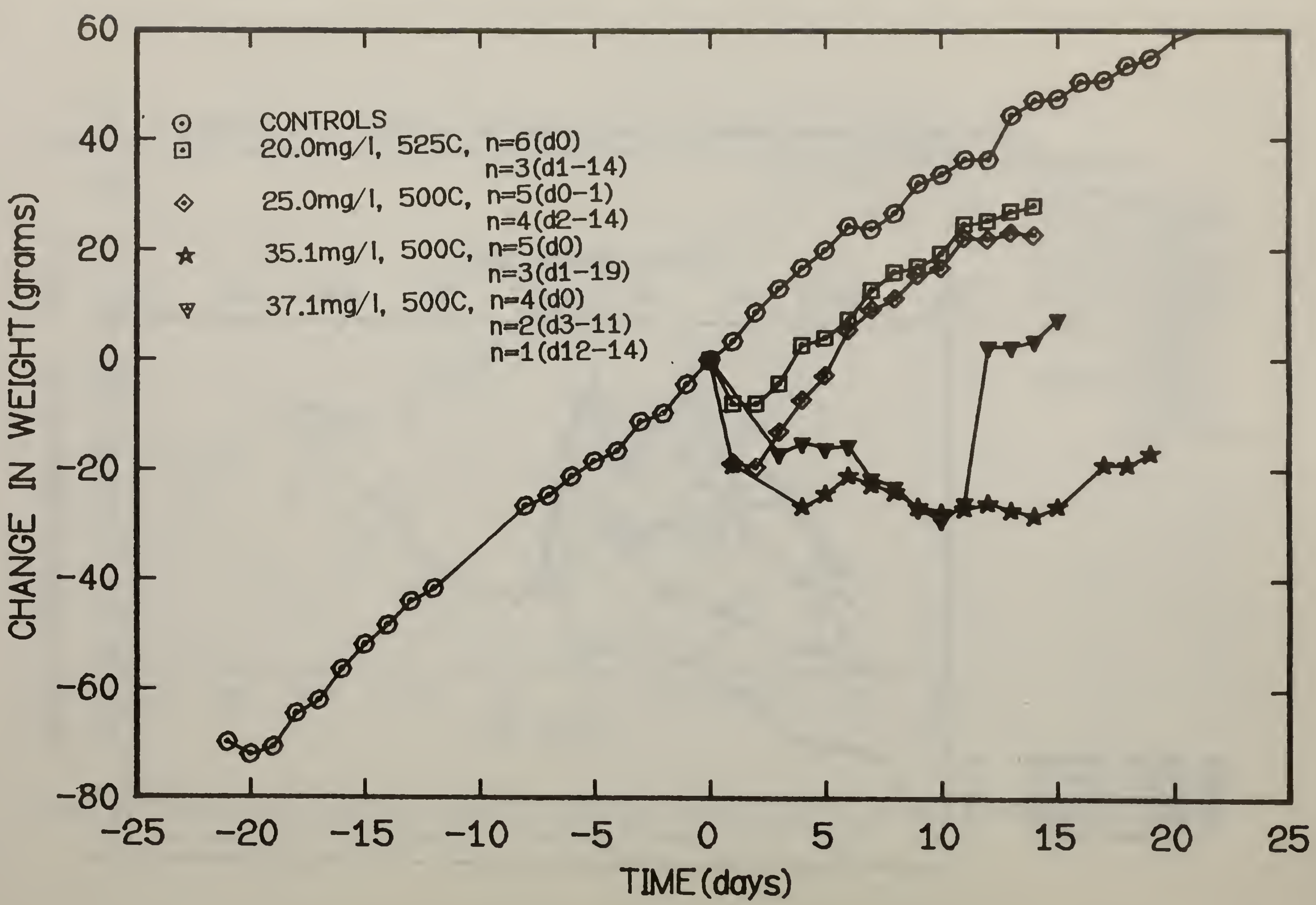

Figure 14. Mean weight profiles after exposure to the non-flaming decomposition of sample #15.

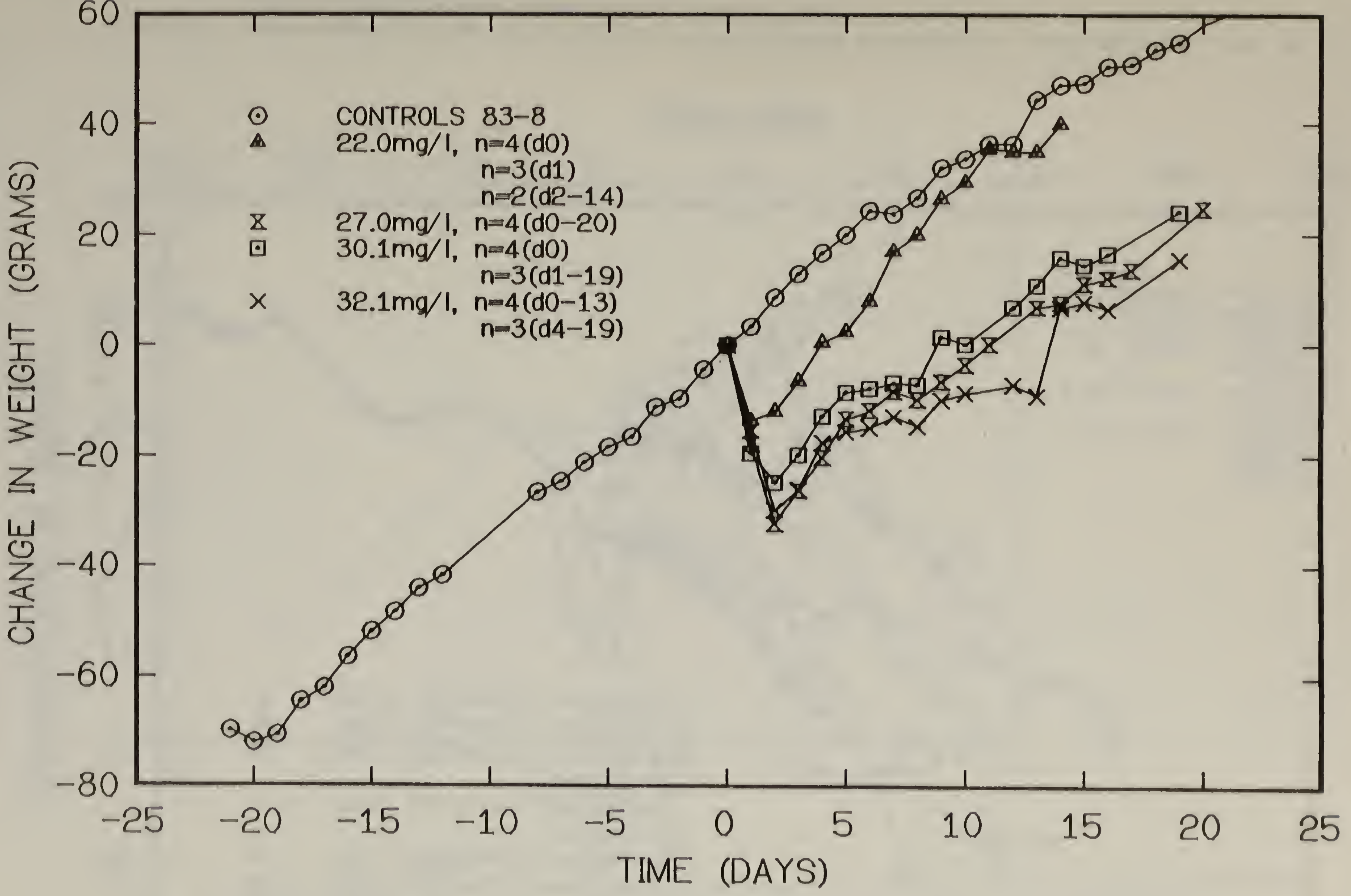

Figure 15. Additional animal mean weight profiles after exposure to the non-flaming decomposition products of material #15.

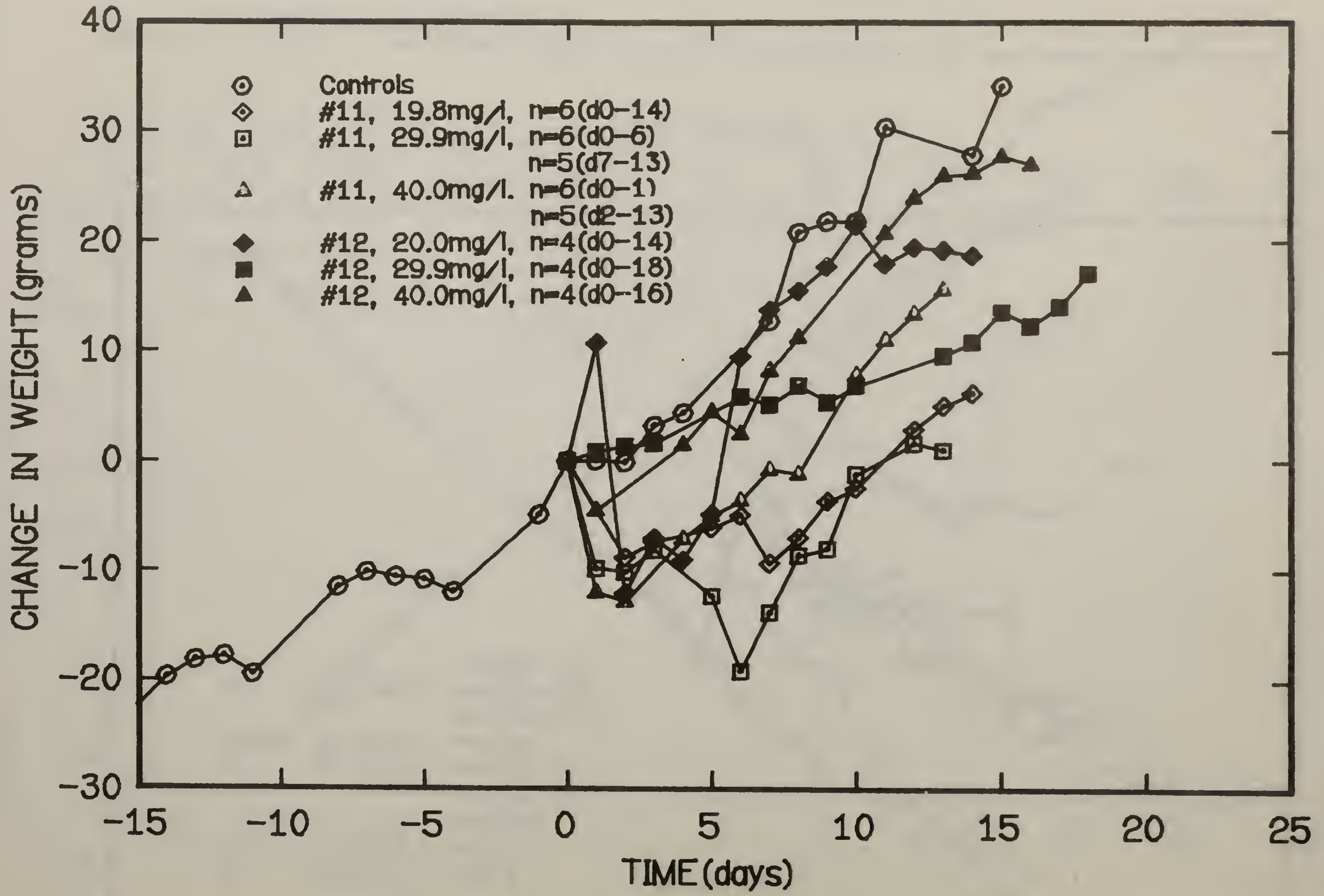

Figure 16. Mean weight profiles after exposure to the flaming decomposition from samples #11 and 12 at mass loadings/chamber volume of 20, 30 and 40 mg/ℓ.

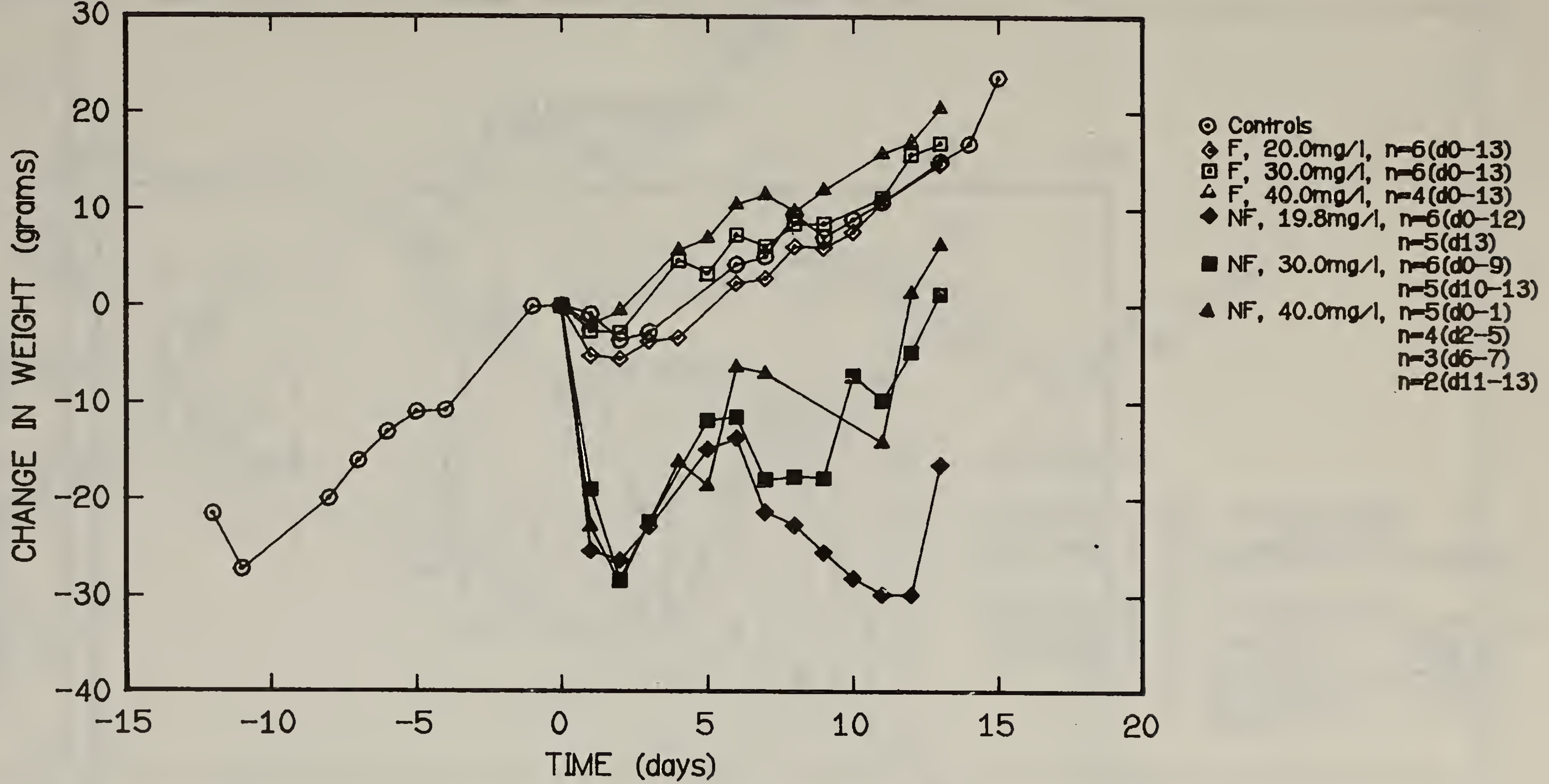

Figure 17. Mean weight profiles of animals exposed to either the flaming or non-flaming decomposition products from sample #13 at mass loadings/chamber volume of 20, 30 and 40 mg/ℓ.

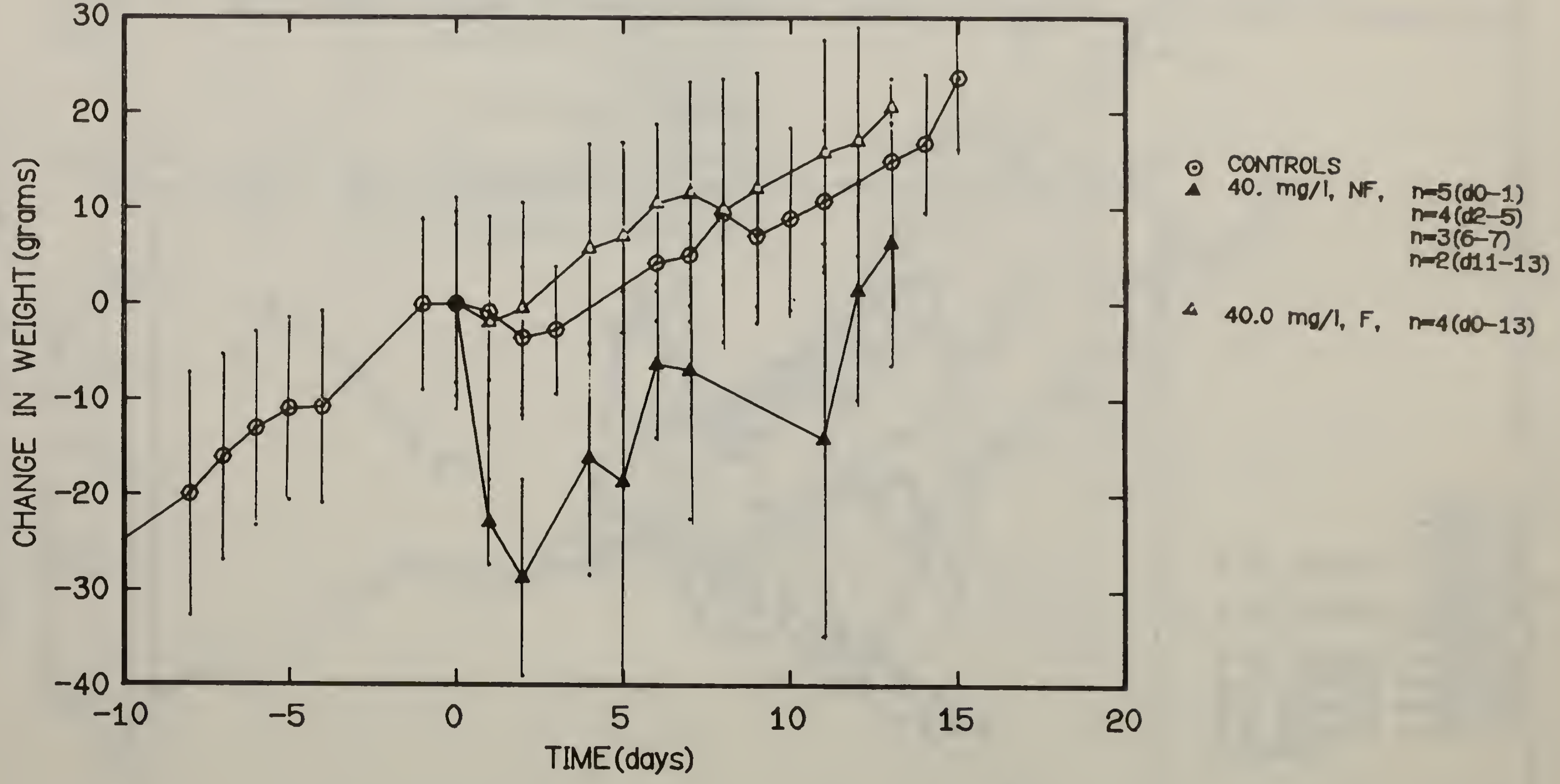

Figure 18. Mean weights and standard deviations of animals exposed to 40 mg/ℓ of material #13 decomposed in the flaming and non-flaming modes.

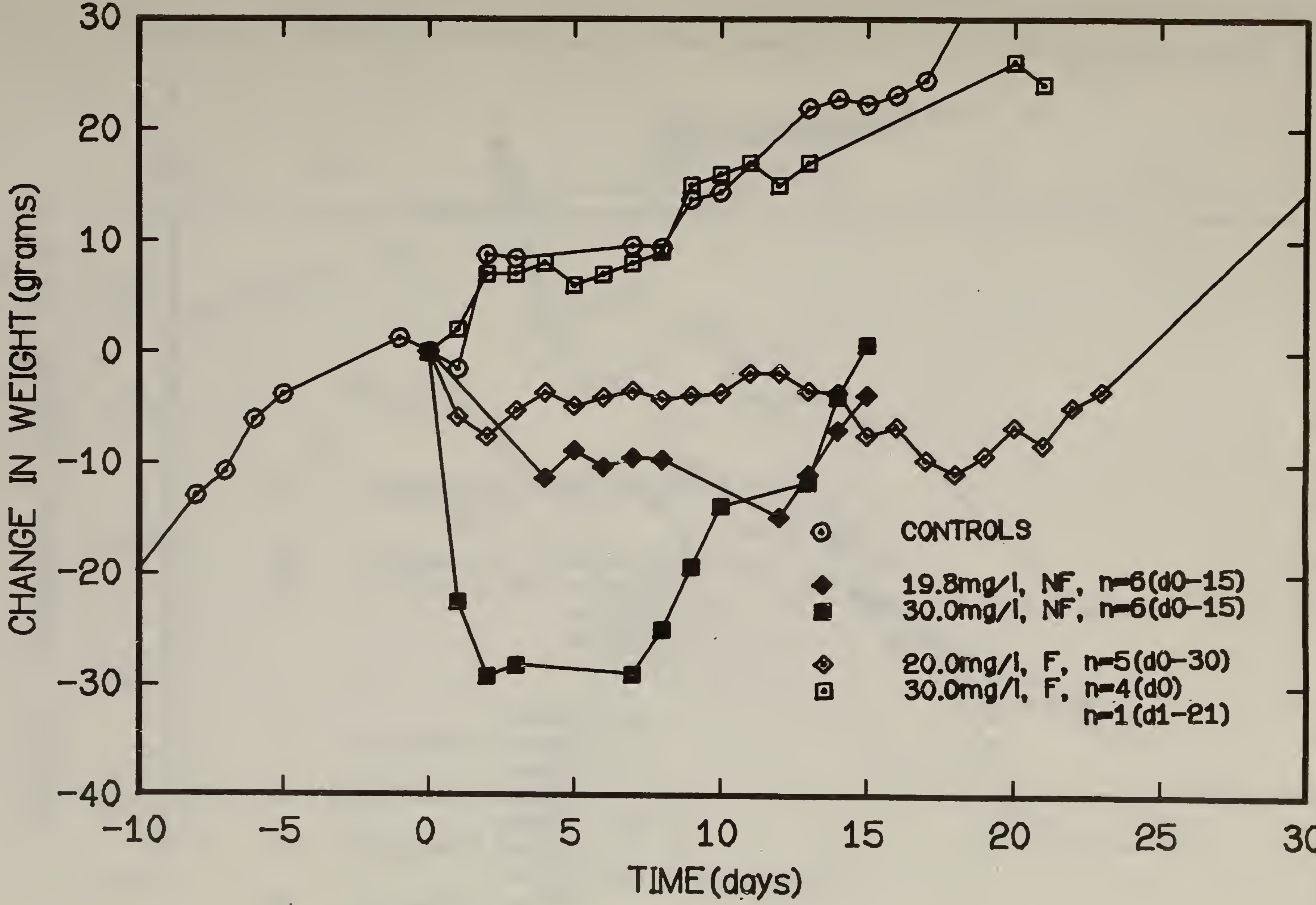

Figure 19. Mean weight profiles of animals exposed to either the flaming or non-flaming decomposition products from sample #14 at mass loadings/chamber volume of 20 and 30 mg/ℓ.

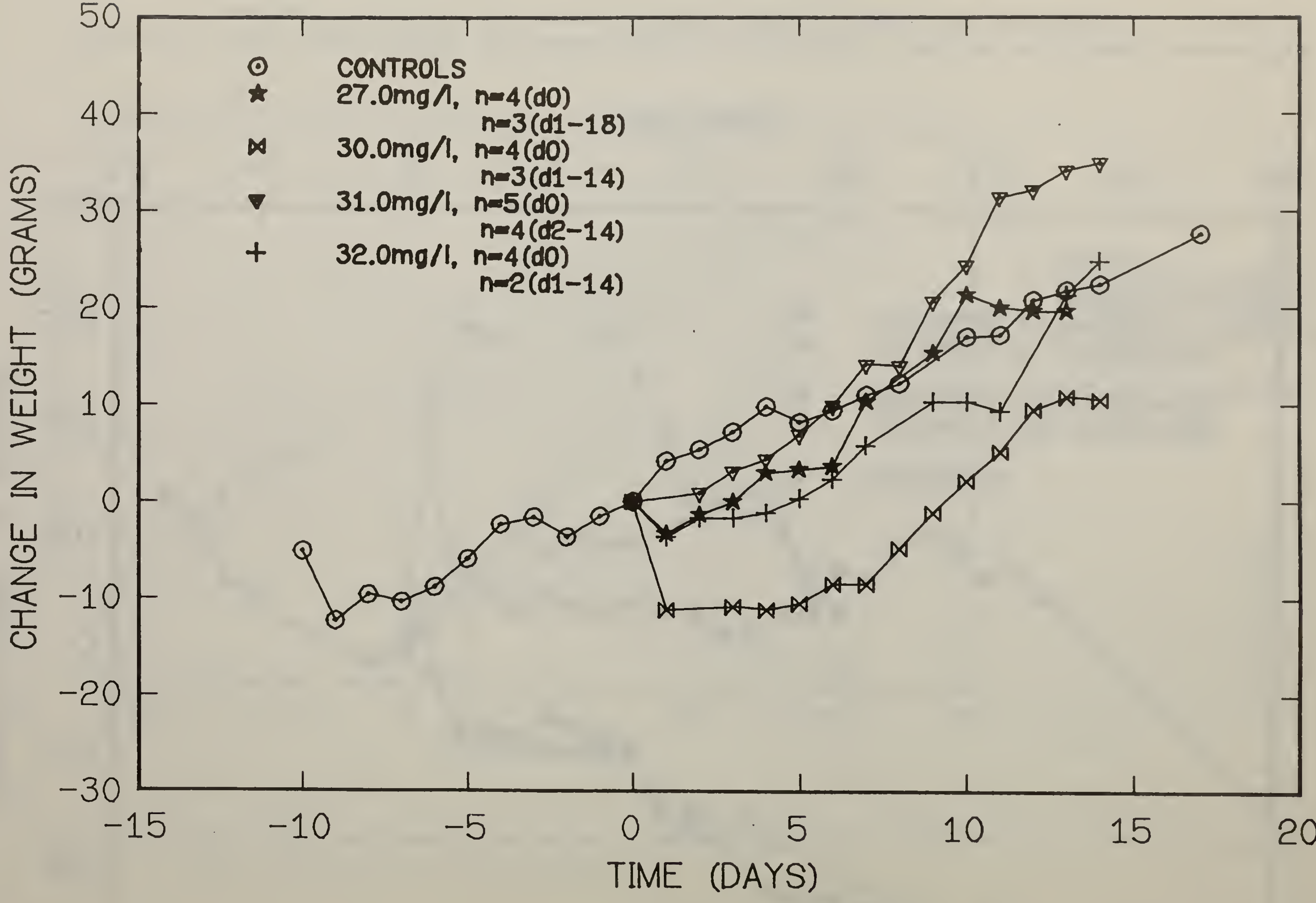

Figure 20. Profiles of mean animal weights after exposure to the flaming decomposition products of material #15.

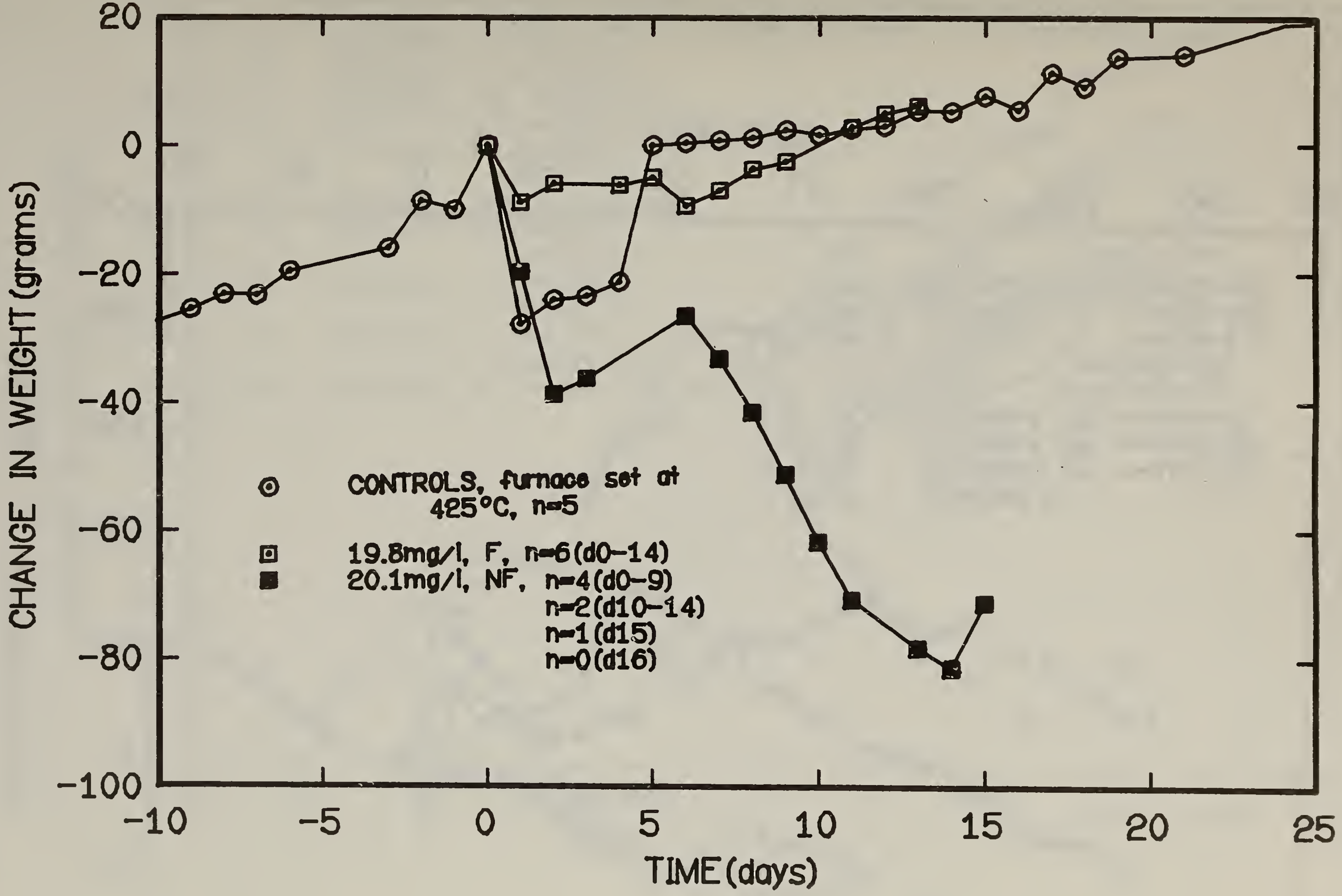

Figure 21. Mean weights of animals after exposure to either the flaming or non-flaming decomposition products from sample #11 at 20 mg/ℓ (mass loading/chamber volume).

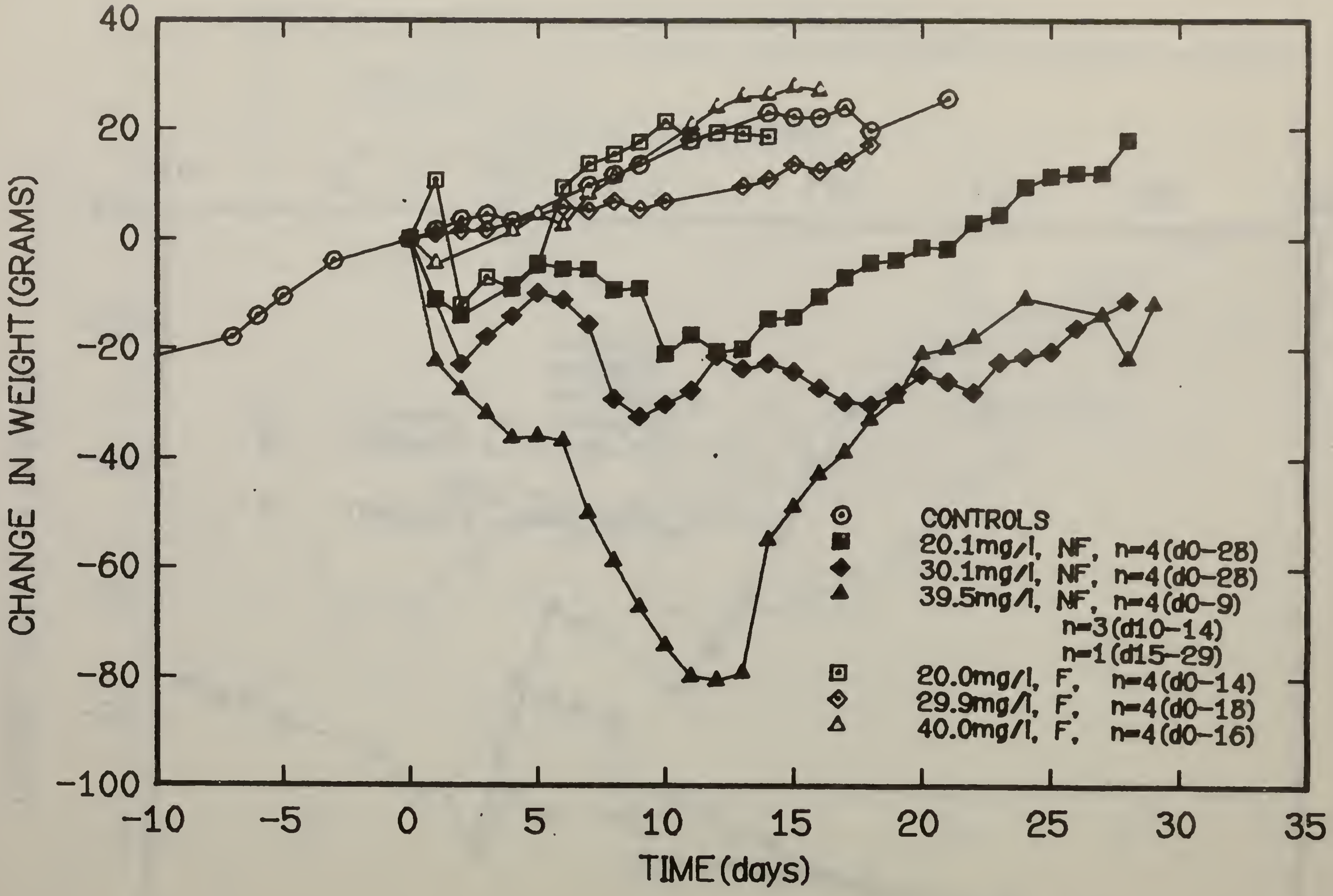

Figure 22. Mean weights of animals exposed to either the flaming or non-flaming decomposition products of sample #12 at different mass loadings/chamber volume (mg/ℓ).

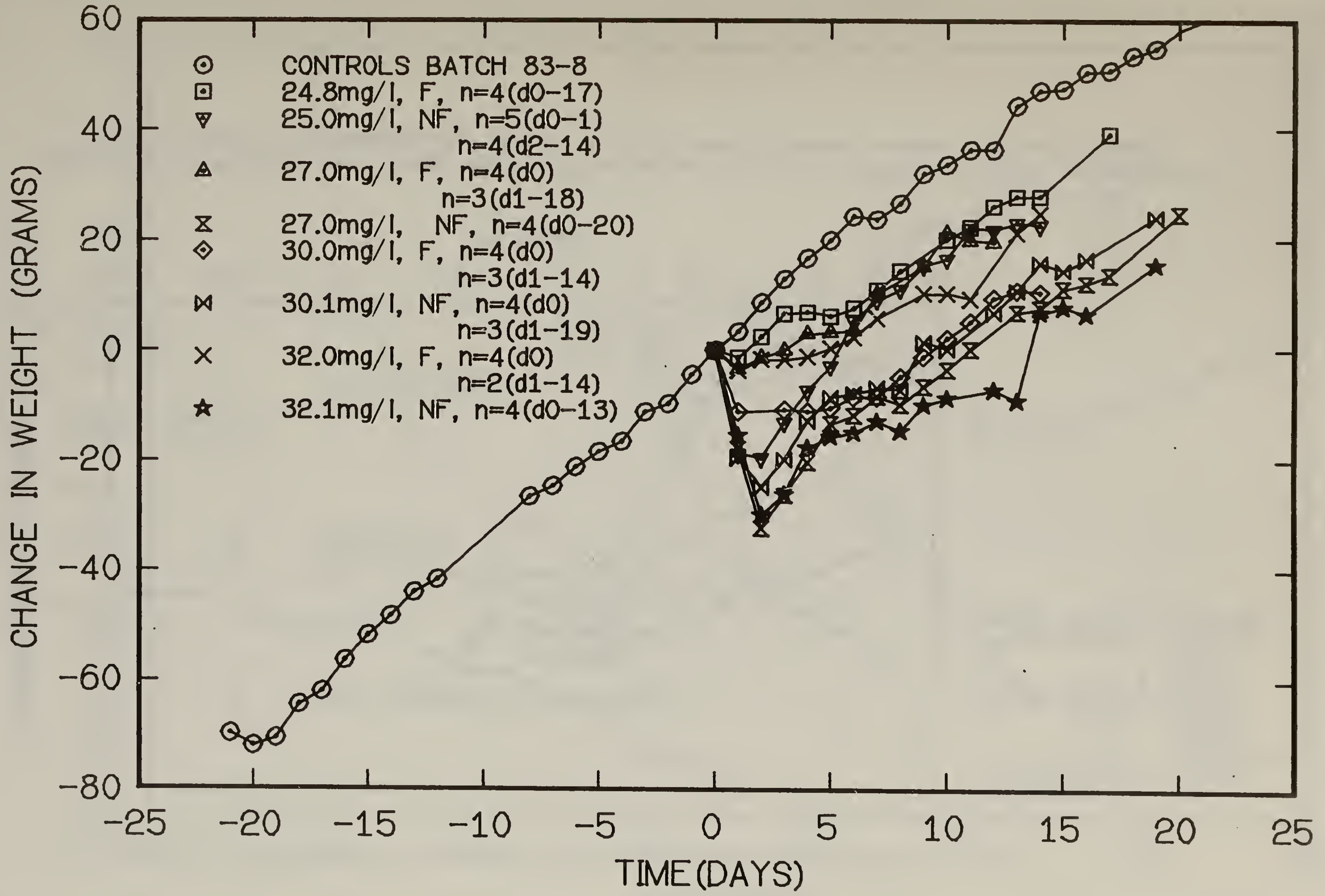

Figure 23. Mean animal weights after exposure to the flaming and non-flaming combustion products from material #14.

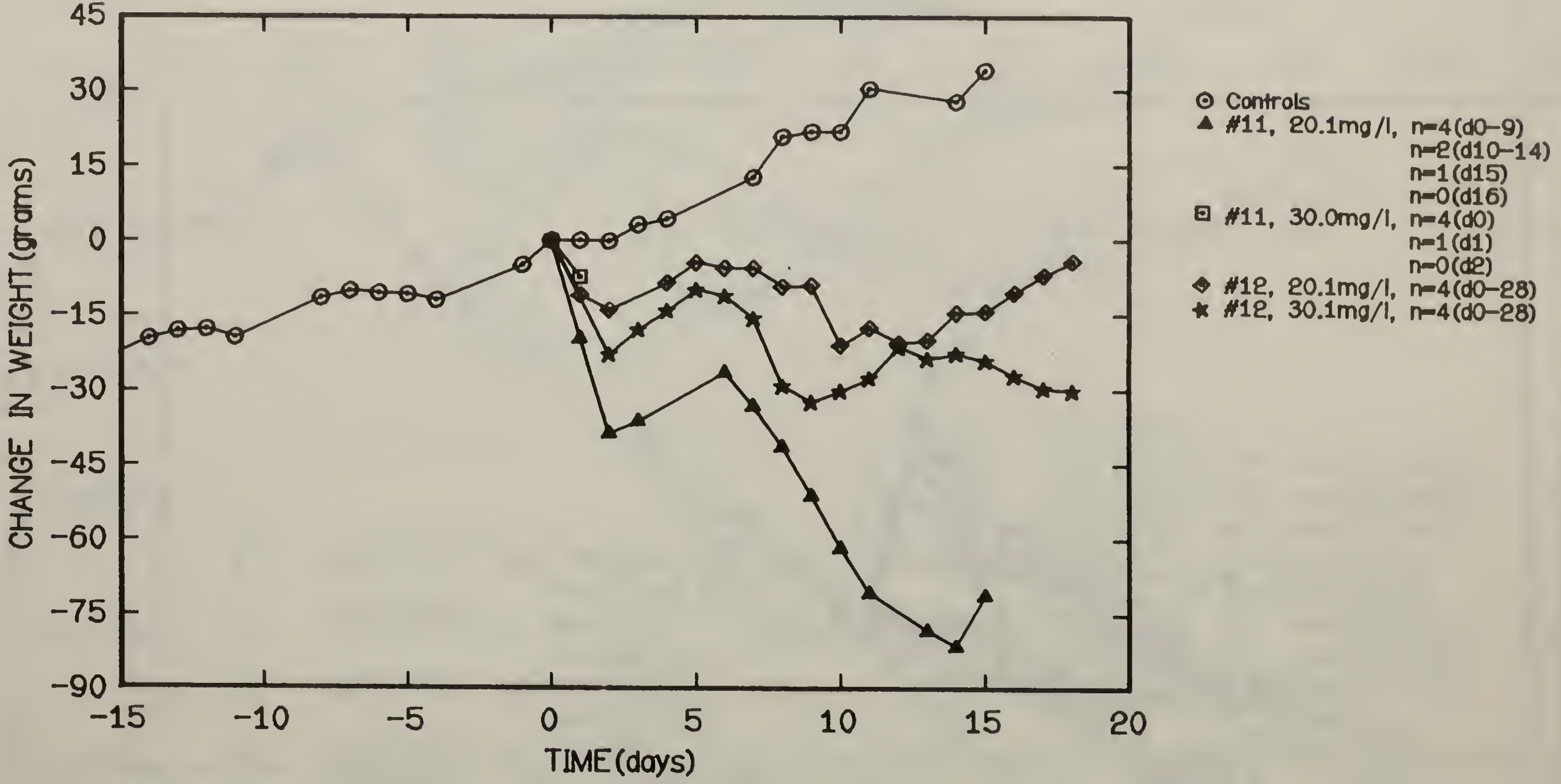

Figure 24. Profiles of mean animal weights after exposure to the non-flaming thermal decomposition products from samples #11 and 12 at mass loadings/chamber volume of 20 and 30 mg/ℓ.

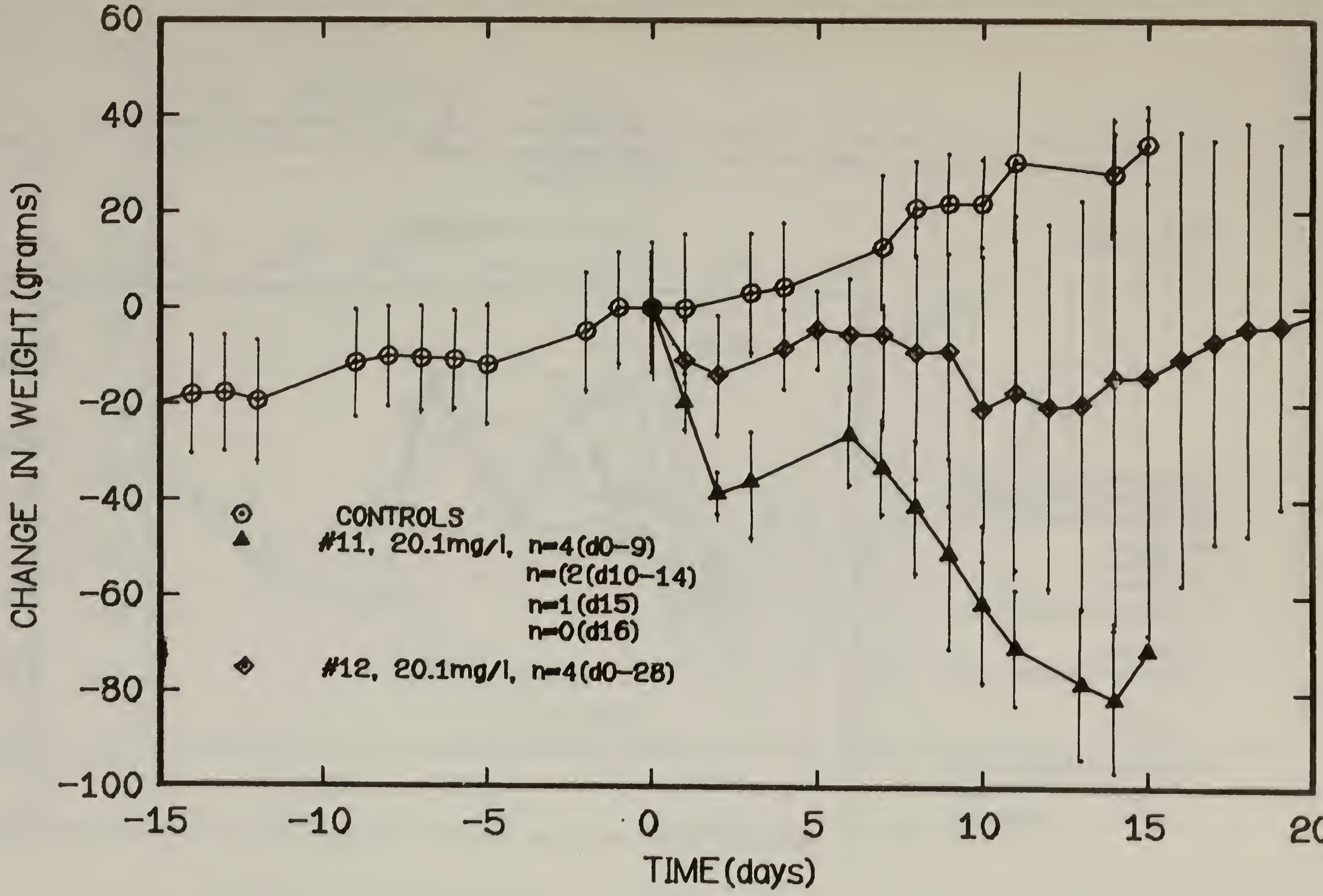

Figure 25. Mean weight profiles and standard deviations of animals exposed to 20 mg/ℓ of materials #11 and 12 decomposed in the non-flaming mode.

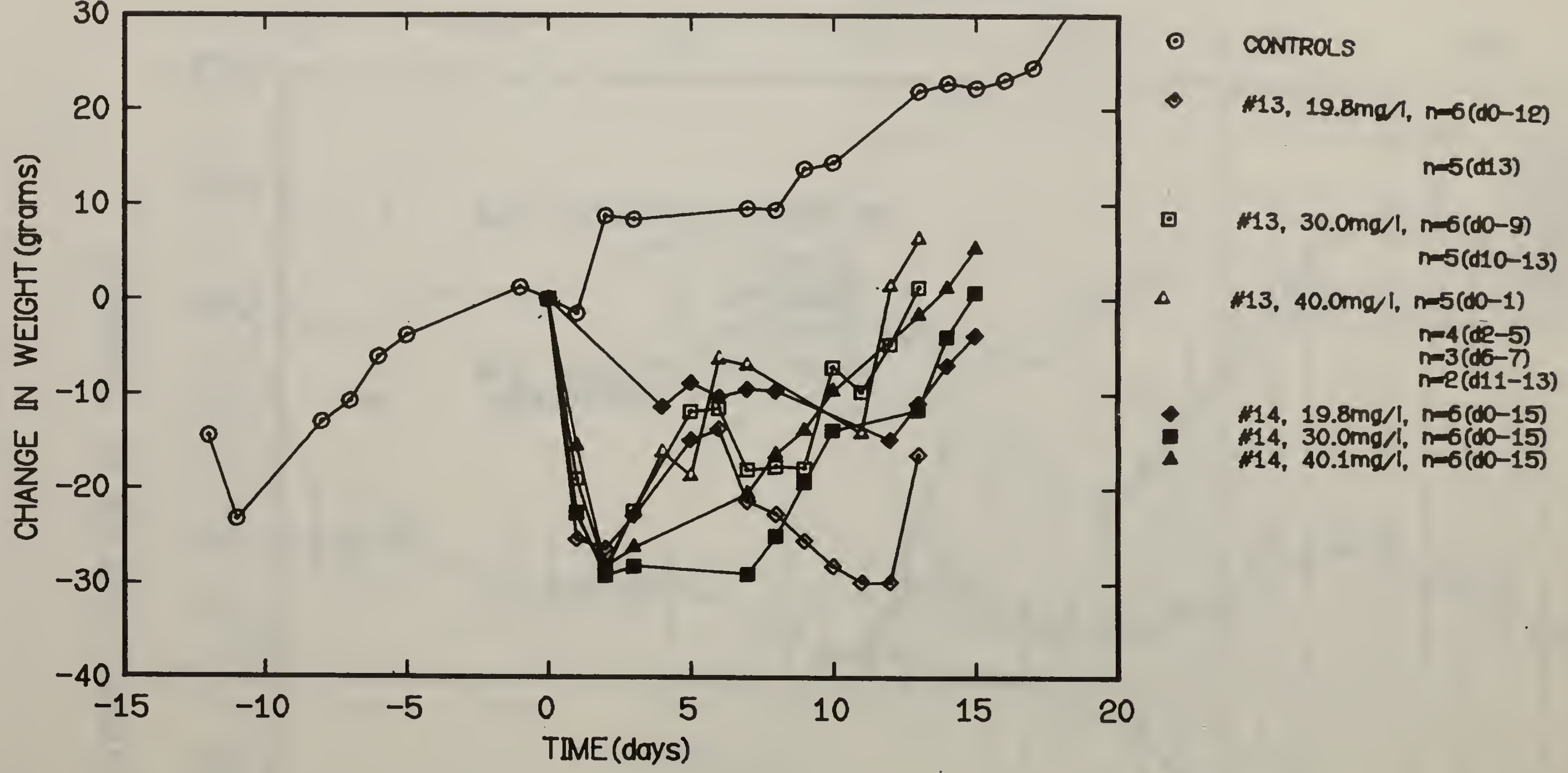

Figure 26. Profiles of mean animal weights after exposure to the non-flaming thermal decomposition products from samples #13 and 14 at mass loadings/chamber volume of 20, 30 and 14 mg/ℓ.

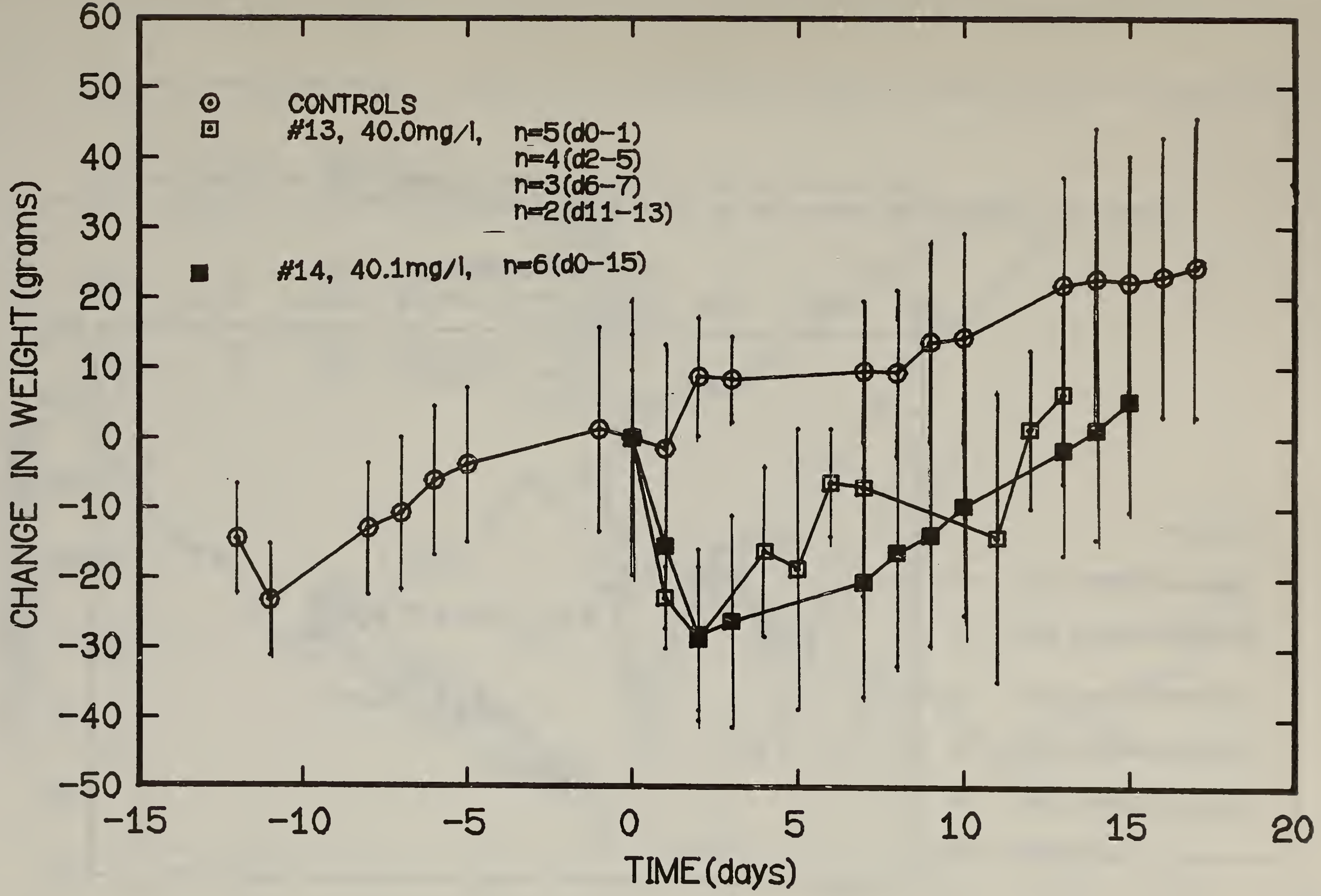

Figure 27. Means and standard deviations of the animal weights after exposure to 40 mg/ℓ of materials #13 and 14 decomposed in the non-flaming mode.

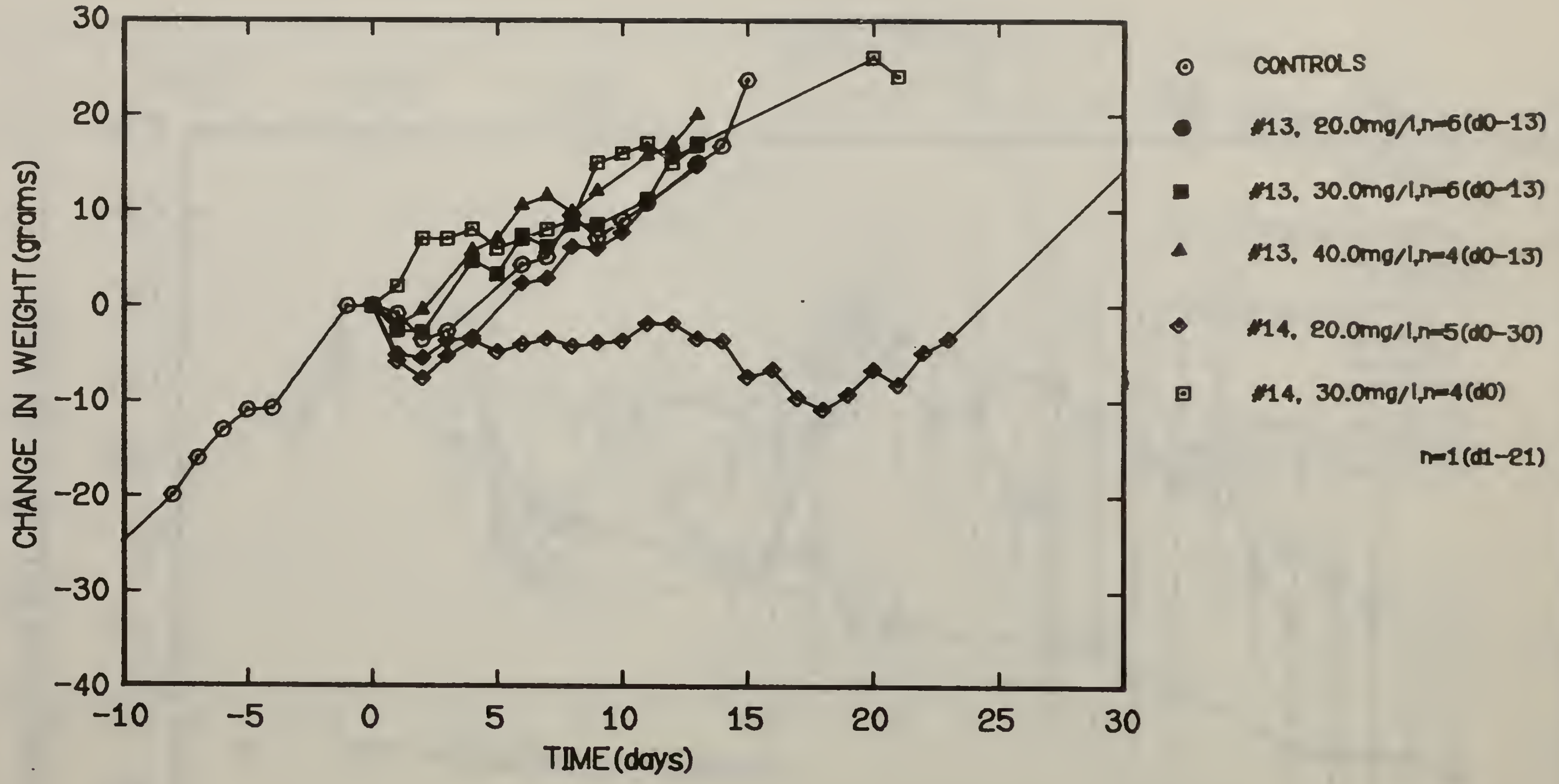

Figure 28. Profiles of mean animal weights after exposure to the flaming decomposition of samples #13 and 14 at mass loadings/chamber volumes of 20, 30 and 40 mg/ℓ.

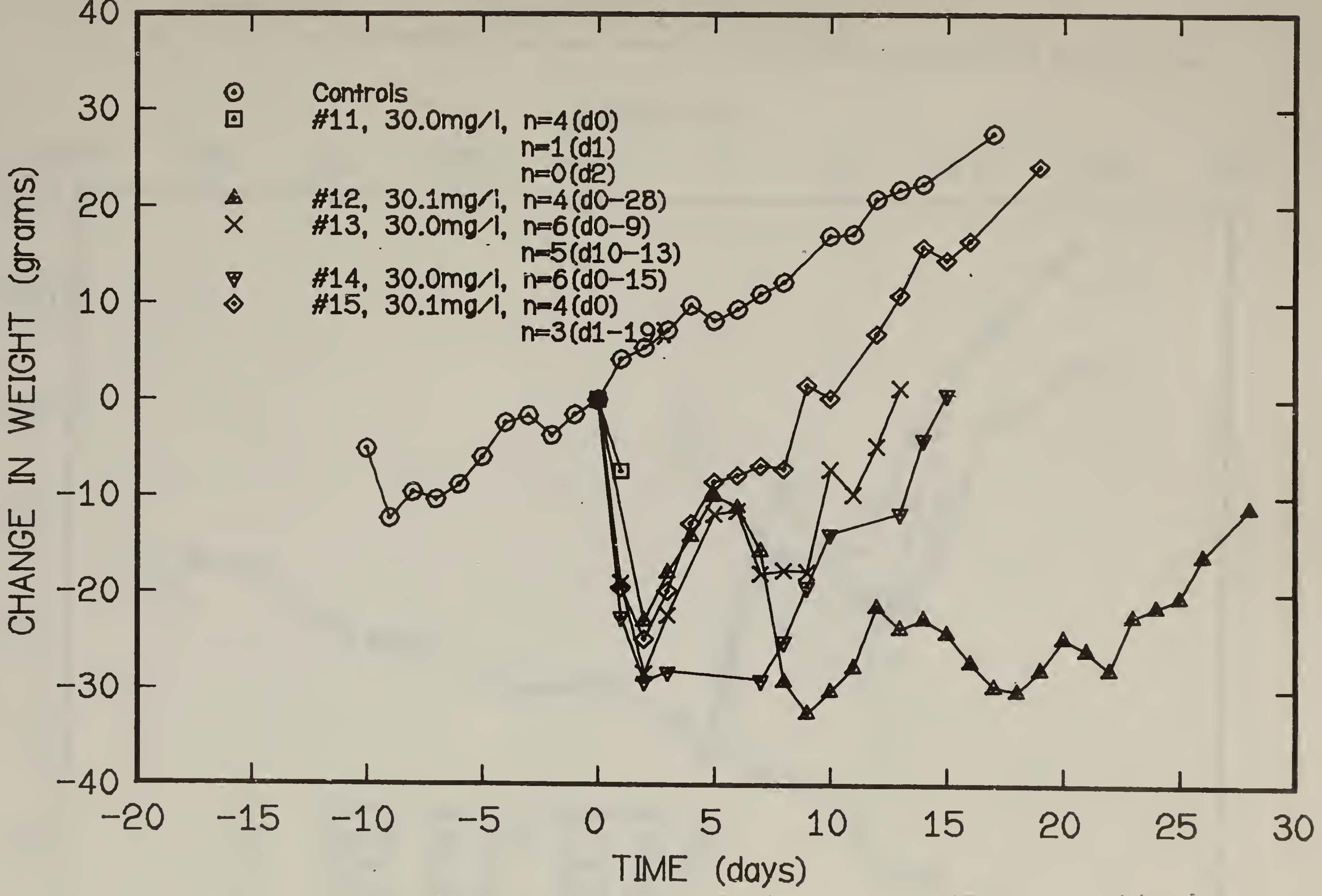

Figure 29. Comparison of mean weights of animals after exposure to non-flaming thermal decomposition products from samples #11, 12, 13, 14 and 15 at a mass loading/chamber volume of 30 mg/ℓ.

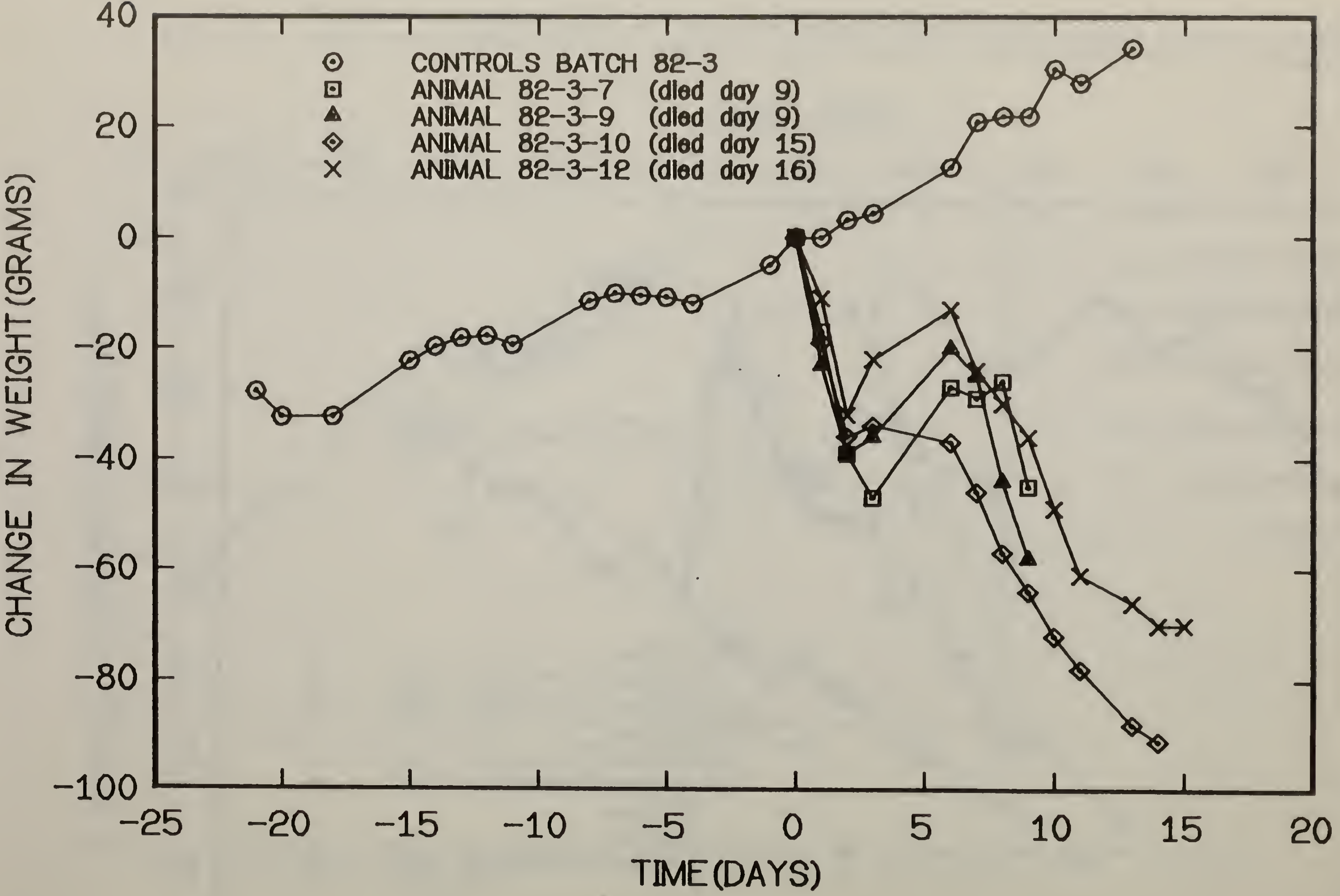

Figure 30. Weight loss in individual animals exposed to non-flaming decomposition products from sample #11 at a mass loading/chamber volume of 20 mg/ℓ.

APPENDIX

Table A-1

Material	AIT (°C)	Mode	Type of Exp.	Mass Loading Chamber Vol. (mg/ℓ)	Mass Consumed Chamber Vol. (mg/ℓ)	Average Gas Concentration[1] CO (ppm)	CO_2 (ppm)	O_2 (%)	HCN (ppm)	Average[2] Nose Temp. (°C)	Highest % COHb (30 min)	No. Died / No. Tested Within Exp.	Within + Post	Latest Day of Death	Weight Data on Graph #	LC_{50} 30 min. 14 days (mg/ℓ)
Poly-urethane # 11	400	NF	R	10.0	8.8	570	1770	20.4	6	30.0	ND	0/6	1/6	15	9	
			R	15.0	12.9	510	2030	20.4	4	29.3	ND	0/6	1/6	15	9	
			R	17.5	14.6	800	2610	20.4	5	ND	36.0	0/6	3/4	14	9	17.2
			A	20.1	17.3	910	820	20.5	2	ND	NA	NA	NA	NA	NA	(13.2-22.4)*
			R	20.1	17.5	800	1790	20.6	7	29.7	45.9	0/6	4/4	16	9,11,14	
			R	30.0	23.9	1290	2350	20.3	10	29.7	ND	0/5	3/3	1	9,11,13	
Poly-urethane # 11	400	NF	R-10	30.0	25.0	770	1060	20.6	1	31.7	ND	0/6	0/6	NA		Passed 10 min. test
			R-10	30.0	25.0	970	1200	20.5	3	30.6	ND	0/6	0/6	NA		
Poly-urethane # 11	400	F	A	19.8	18.9	700	16600	19.0	62	34.0	NA	NA	NA	NA		
			R	19.8	18.8	660	17300	18.7	41	33.4	ND	0/6	0/6	NA		
			R	29.9	28.1	930	22500	18.0	64	34.5	ND	0/6	1/6	8		>40
			R	40.0	38.1	1370	33000	16.8	67	37.0	ND	1/6	1/6	0		
			R	40.0	38.2	1370	31000	17.0	67	38.8	59.5	1/6	1/5	0		

Legend:

1. Average gas concentration = $\frac{\text{integrated area under instrument response curve for 30 minutes}}{\text{30 minutes}} = \frac{\text{ppm-min}}{\text{30 min}}$

2. Average temperature over 30 minute experiment at animal noses 1, 3, 6.
A. Analytical experiment
R. Rat experiment
R-10. 10 min. rat experiment
ND. Not determined
NA. Not applicable
NC. Not completed
F. Flaming
NF. Non-Flaming
AIT. Auto-ignition temperature
*. 95% confidence limits

Table A-2

Material	AIT (°C)	Mode	Type of Exp.	Mass Loading Chamber Vol. (mg/ℓ)	Mass Consumed Chamber Vol. (mg/ℓ)	Average Gas Concentration[1]				Average[2] Nose Temp. (°C)	Highest % COHb (30 min)	No. Died / No. Tested		Latest Day of Death	Weight Data on Graph #	LC_{50} 30 min. 14 days (mg/ℓ)
						CO (ppm)	CO_2 (ppm)	O_2 (%)	HCN (ppm)			Within Exp.	Within + Post			
Poly- urethane # 12	400	NF	A	10.0	8.8	390	440	20.4	ND	28.8	NA	NA	NA	NA		
			A	19.9	16.8	520	700	ND	3	29.5	NA	NA	NA	NA		
			R	20.1	16.6	580	2760	20.3	ND	29.6	ND	0/6	0/4	NA	11	37.8
			R	30.1	27.0	850	3160	20.2	1	27.8	ND	0/6	0/4	NA	11,13	(36.6-39.0)*
			R	37.5	30.5	1040	3110	ND	10	21.9	ND	0/6	2/5	11		
			R	38.2	31.3	990	3380	20.2	8	30.5	56.5	0/6	3/4	4		
			R	38.4	34.5	ND	ND	ND	14	ND	38.3	0/6	5/5	9		
			R	39.5	32.0	1170	3780	20.1	ND	27.8	47.2	0/6	3/4	14		
Poly- urethane # 12	400	F	A	10.2	9.3	190	9620	19.6	14	31.4	NA	NA	NA	NA		
			R	20.0	18.5	310	19360	18.2	20	33.1	13.2	0/6	0/4	NA		
			R	29.9	27.6	470	24950	17.9	38	34.4	ND[a]	0/6	0/4	NA		>40
			R	40.0	36.3	ND	ND	ND	42	ND	34.5	0/6	0/4	NA		

For legend see table A-1.

a. No final blood; value was 9.7% at 15 minutes.

Table A-3

Material	AIT (°C)	Mode	Type of Exp.	Mass Loading Chamber Vol. (mg/ℓ)	Mass Consumed Chamber Vol. (mg/ℓ)	Average Gas Concentration[1]				Average[2] Nose Temp. (°C)	Highest % COHb (30 min)	No. Died / No. Tested		Latest Day of Death	Weight Data on Graph #	LC_{50} 30 min. 14 days (mg/ℓ)
						CO (ppm)	CO_2 (ppm)	O_2 (%)	HCN (ppm)			Within Exp.	Within + Post			
Poly-urethane # 13	400	NF	A	10.0	9.3	420	470	20.8	8	29.9	NA	NA	NA	NA	NA	
			R	19.8	17.9	620	2370	20.4	ND	30.8	ND	0/6	1/6	14	10,12	
			R	30.0	26.6	700	2690	20.4	4	30.4	ND	0/6	1/6	11	10,12,13	37.0 (29.8-46.0)*
			R	32.0	27.4	600	2800	20.3	5	ND	26.3	0/6	1/5	7		
			R	35.0	31.1	ND	ND	ND	9	ND	47.0	0/6	4/4	11		
			R	40.0	34.3	740	2500	ND	1	32.4	ND	0/6	3/5	12	10,12	
Poly-urethane # 13	400	F	A	10.0	9.7	170	8700	19.9	19	32.7	NA	NA	NA	NA	NA	
			R	20.0	19.9	320	21400	18.2	17	ND	ND	0/6	0/6	NA	10	>40
			R	30.0	29.8	520	28400	17.4	24	34.7	ND	0/6	0/6	NA	10	
			R	40.0	39.1	840	33500	16.7	27	31.4	46.5	0/6	0/4	NA	10	

For legend see table A-1.

Table A-4

Material	AIT (°C)	Mode	Type of Exp.	Mass Loading Chamber Vol. (mg/ℓ)	Mass Consumed Chamber Vol. (mg/ℓ)	Average Gas Concentration[1]				Average[2] Nose Temp. (°C)	Highest % COHb (30 min)	No. Died / No. Tested		Latest Day of Death	Weight Data on Graph #	LC_{50} 30 min. 14 days (mg/ℓ)
						CO (ppm)	CO_2 (ppm)	O_2 (%)	HCN (ppm)			Within Exp.	Within + Post			
Polyurethane # 14	375	NF	A	10.0	8.1	160	390	20.7	3	29.5	NA	NA	NA	NA		
			R	19.8	16.0	350	3800	20.4	2	31.8	ND	0/6	0/6	NA	12	>40
			R	30.0	21.0	230	2800	20.5	2	32.2	ND	0/6	0/6	NA	12,13	
			R	39.9	ND	230	2960	20.5	2	ND	10.1	0/6	0/5	NA		
			R	40.1	28.5	400	3100	20.2	0.5	31.1	ND	0/6	0/6	NA	12	
Polyurethane #14	375	F	A	9.9	9.1	340	6020	ND	22	31.7	NA	NA	NA	NA		
			R	20.0	18.5	950	14500	18.9	45	32.2	34.1	0/6	0/5	NA		
			R	25.0	22.8	ND	ND	ND	49	ND	52.2	0/6	0/4	NA		27.8
			R	28.0	25.6	1600	22800	18.0	76	36.1	65.8	2/6	3/4	0		(23.3-33.1)*
			R	30.0	27.8	1440	23200	18.0	64	33.1	61.4	2/6	5/6	0		
			R	32.0	26.8	1590	24100	ND	89	33.6	ND[a]	1/6	2/5	0		
Polyurethane #14	375	F	R-10	30.0	26.9	1050	17100	18.8	41	30.1	ND	0/6	0/6	NA		Passed 10 min test
			R-10	30.0	27.4	1340	21900	18.1	72	30.2	ND	0/6	0/6	NA		

For legend see table A-1.

a. No final blood; value was 39.1% at 14 minutes.

Table A-5

Material	AIT (°C)	Mode	Type of Exp.	Mass Loading Chamber Vol. (mg/ℓ)	Mass Consumed Chamber Vol. (mg/ℓ)	Average Gas Concentration[1] CO (ppm)	CO_2 (ppm)	O_2 (%)	HCN (ppm)	Average[2] Nose Temp. (°C)	Highest % COHb (30 min)	No. Died / No. Tested Within Exp.	Within + Post	Latest Day of Death	Weight Data on Graph #	LC_{50} 30 min. 14 days (mg/ℓ)
Polyester #15	525	NF	A	10.3	9.2	782	990	20.7	ND	30.1	NA	NA	NA	NA		
			A	20.1	18.8	2060	2700	20.4	ND	29.6	NA	NA	NA	NA		
			R	20.0	19.9	2190	ND	20.4	ND	37.3	77.8	2/6	2/5	NA		
			R	22.0	20.3	1730	3890	20.2	ND	31.2	58.5	1/6	3/5	2		
			R	25.0	24.2	2620	4720	20.1	ND	ND	76.1	0/6	1/5	2		27.6
			R	27.1	26.2	3000	5240	20.3	ND	31.6	81.7	1/6	1/5	NA		(22.1-34.4)*
			R	30.0	29.0	3080	5630	20.2	ND	31.9	86.7	2/6	2/5	NA		
			R	32.1	29.9	2970	5140	20.0	ND	32.0	85.8	0/6	1/4	14		
			R	35.1	32.6	3220	ND	20.2	ND	30.4	ND	2/6	4/6	19		
			R	37.1	33.6	3260	ND	20.2	ND	29.5	85.6	4/6	5/6	12		
			A	40.1	35.8	3580	4590	20.7	ND	28.2	NA	NA	NA	NA		
Polyester #15	525	F	A	19.9	19.8	2020	16800	19.0	ND	ND	NA	NA	NA	NA		
			R	24.8	24.8	2570	18100	19.1	ND	35.0	80.3	0/6	0/4	NA		
			R	27.0	26.9	2690	ND	18.4	ND	32.9	82.0	1/6	2/5	0		
			R	28.5	28.4	2950	23100	18.4	ND	ND	ND	0/6	0/6	NA		
			R	29.5	29.3	3410	22400	18.5	ND	ND	ND[a]	2/6	2/5	NA		30.8
			R	30.0	29.6	3120	25300	ND	ND	33.1	79.0	2/6	2/5	NA		(28.2-33.6)*
			R	30.1	29.9	3190	ND	18.2	ND	31.4	ND[b]	6/6	6/6	NA		
			R	31.0	30.7	3100	25600	18.2	ND	32.6	ND	2/6	2/6	NA		
			R	32.0	31.9	3210	25200	18.2	ND	32.1	ND[c]	4/6	4/6	NA		
			R	33.0	32.7	3330	26900	17.7	ND	33.0	84.8	4/6	4/5	NA		
			A	35.0	34.7	3550	26900	18.0	ND	32.4	NA	NA	NA	NA		

a. No final blood; value at 14 min. was 70.1
b. No final blood; value at 14 min. was 68.4
c. No final blood; value at 17 min. was 81.6

For legend see table A1.

NBS-114A (REV. 2-80)

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An Acute Inhalation Toxicological Evaluation of Combustion Products from Fire Retarded and Non-Fire Retarded Flexible Polyurethane Foam and Polyester

5. AUTHOR(S) Barbara C. Levin, Maya Paabo, Mary Lou Fultz, Cheryl Bailey, Way Yin, and Steven E. Harris

6. PERFORMING ORGANIZATION *(If joint or other than NBS, see instructions)*

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

The acute inhalation toxicity of the combustion products from selected upholstered furniture filling materials with and without fire retardants was evaluated by the toxicity test method developed by the National Bureau of Standards. The five materials that were evaluated consisted of two different formulations of flexible polyurethane foam (each formulation was supplied in both a fire retarded and non-fire retarded form) and a polyester fiberfill (not fire retarded). Atmospheric concentrations of carbon monoxide, carbon dioxide, oxygen, and hydrogen cyanide in the exposure chamber were monitored throughout the thermal decomposition of the materials. In addition, time-to-ignition, rate of heat release, hydrogen cyanide generation via a two phase decomposition procedure and x-ray fluorescence measurements were performed. The LC_{50} (30 minute and 14 day) values did not distinguish any of the materials as being significantly more toxic than the others. In the worst case, one of the fire retarded polyurethane foams was only a factor of two more toxic than its untreated counterpart. Only the fire retarded foams and the polyester caused deaths of the Fischer 344 rats during the 30 minute exposures. Extensive weight loss and post-exposure deaths occurred after exposure to the combustion products from all the materials. Animal deaths during or following exposure to the polyurethane cannot be attributed to carbon monoxide or hydrogen cyanide alone. Deaths during exposure to the polyester combustion products appear to be more directly related to the concentrations of carbon monoxide.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Carbon monoxide; combustion products; fire retardants; heat release; hydrogen cyanide; inhalation; polyester; polyurethane foams; toxicology; weight loss

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