baked, the destructive phenomena now observed are considered to be highly relevant in a qualitative sense to artificial depletion of the nutritive value of food. The described studies should facilitate formulating baking conditions to minimize loss of nutrients and the formation of antinutrients.

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Registry No. HPMC, 9004-65-3; CMC, 9004-32-4; L-ascorbic acid, 50-81-7; amylose, 9005-82-7; starch, 9005-25-8; D-glucose, 50-99-7; D-fructose, 57-48-7; D-lactose, 63-42-3; D-maltose, 69-79-4; D-sucrose, 57-50-1; cellulose, 9004-34-6; hydrocellulose, 9034-34-8; sodium L-ascorbate, 134-03-2.

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Phosphine Residue and Its Desorption from Cereals

Jois R. Rangaswamy

The extent and the pattern of desorption of PH_3 residue in wheat, raw polished rice, raw unpolished rice, and parboiled rice were followed at laboratory level for 60 days. The PH_3 residue on each day of storage was computed by knowing the total amount of PH_3 desorbed over 60 days. The PH_3 desorption patterns from four types of cereals are discussed under three stages: up to 19 days, 20–49 days, and 50–60 days of storage. The relationship between the logarithm of such residues vs. storage in days is linear over the first 19 days, except in the case of residue from raw polished rice, which is only up to the first 9 days. During the period of linearity the desorption follows first-order kinetics. Between days 20 and 49 there is a waxing and waning in the amount of PH_3 desorbed. Between days 50 and 60 there is a regular fall in PH_3 residues due to uniform decreased desorption, except in raw unpolished rice in which desorption is a bit sluggish up to 56 days.

Although phosphine liberated from aluminium phosphide preparation has been in use as fumigant for cereals and their products since 1936, nothing is known about the manner and the rate of its residue elimination during subsequent storage after airing, except for the claim that PH_3 disappears completely on aeration (Degesch, 1962). As it has now been conclusively established that PH_3 is not a "nonresidue" fumigant, the manner of its elimination from fumigated cereals is important to assess the possibility of any physically bound toxic PH_3 residues persisting in cereals.

Cereals or their products absorb PH_3 gas depending on their gas-holding capacity; for example, wheat (Rangaswamy, 1984), raw polished rice, and paraboiled rice (Rangaswamy, 1985) absorb about 62%, 27%, and 84%,

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respectively, when an excess of PH_3 is available. So, the amount of PH_3 absorbed during the exposure period depends on a number of factors such as physical structure of the grain, varietal differences to some extent, fat content, temperature, and dose of fumigant.

Special care should be exercised to avoid subjecting the PH_3 to any condition that might produce fire or explosion. As PH_3 is highly toxic with a threshold limit value of 0.3 ppm, care should be taken to avoid exposures to high concentrations. The conditions and concentration levels that might produce fire or explosion have been dealt with in detail (Green et al., 1984; Bond, 1984). However, in my experience such explosions at such concentration levels are unlikely to occur spontaneously.

When cereal is fumigated with PH_3, two types of residues are likely to occur: (a) inorganic phosphorus compounds formed due to oxidation of PH_3; (b) physically bound free PH_3. The residues of (a) type are neither significant nor important from the point of toxicity of fumigated cereals. The (b) type residues are most important as the fumigant exerts its toxic effects as PH_3. Although chemisorption of PH_3 in cereal and its products has been discounted (Rauscher et al., 1972), there is no reason to preclude such possibilities due to the weak nucleophilic nature of PH_3. Some data have accumulated about the nature and extent of inorganic phosphorus residues in wheat (Robinson and Bond, 1970; Robinson, 1972; Tkachuk, 1972), and most of these studies have been done with $^{32}PH_3$. Although some studies have reported the extent of free PH_3 residues (Rangaswamy, 1984, 1985; Dumas, 1980; Dumas, 1978; Nowicki, 1978) persisting in cereals, no study has been reported about the manner and kinetics of elimination of PH_3 residues from cereals after airing during storage. The low levels of free PH_3 residues found by previous authors referred to is due to differences in extraction and assay procedures, in addition to lower dosages used for fumigation. Hence, the purpose of this study is to elucidate these parameters.

Experiments on the extent and the mode of desorption of unchanged PH_3 residues from wheat, raw polished rice, raw unpolished rice, and parboiled rice are reported in this paper.

EXPERIMENTAL METHODS

Fumigation and Aeration. Two kilograms each of wheat, raw polished rice, raw unpolished rice, and parboiled rice in three replicates were fumigated by placing a Phosfume (aluminium phosphide + binders + pelleting agents but no ammonium carbamate) pellet (0.60 g) in a paper pack underneath the cereal in a 2-L flask. Nearly 180 mg of PH_3 should have been produced by 0.6 g of Phosfume formulation. The flask was closed with a gas-tight silicon-greased glass stopper. The purpose of exposing to such a large dose (above the specified limit) was to allow cereals to absorb to their maximum capacity, so that the desorption could be studied after airing all the free PH_3 completely by spreading the commodities in the open in thin layers. At the end of a 2-week exposure at room temperature (28–30 °C), the initial PH_3 residue was determined and the cereals were aired by spreading as a thin layer in the open for 48 h.

Initial PH_3 Residue before Airing. The initial PH_3 residue in the fumigated cereals before airing was determined by extracting 10 g of grain with 21 mL of $AgNO_3$ solution (30 μg/mL; Rangaswamy, 1984, 1985). Ten grams of the corresponding crop control were similarly extracted, and the absorbance value was subtracted from that of the PH_3-treated samples. The same extracts were employed for scanning the absorption spectra of the chromophore in the range of 200–650 nm in a Spectronic-21 spectrophotometer against the corresponding crop control as blank.

Detector Strip Method (Linear Relationship between Micrograms of PH_3 vs. Area (cm^2) of the Red Band). A detector strip (1 cm wide × 6 cm long) was placed in a test tube (1.4-cm i.d. × 15-cm length) whose mouth was closed with a gas-tight butyl rubber septum. The strip was in the proximity of the septum. Detector strips were prepared by dipping Whatman No. 1 filter paper strips in a solution of dimethyl yellow (0.05%), cresol red (0.10%), and mercuric chloride (1.0%) in distilled dry methanol (Kashi and Muthu, 1975). Aliquots of PH_3 in the range of 0.02–0.4 μg (from a gas buret) were injected from a 1-μL Hamilton gas-tight syringe. The area of the red band corresponding to each aliquot of PH_3 was noted. Areas plotted against micrograms of PH_3 were found to be linear between 0.02 and 0.2 μg of PH_3. This linear relationship has been employed to estimate the amount of PH_3 desorbed from 200-g grains.

Comparison of the Amounts of PH_3 Desorbed by Two Methods. 1. Silver Nitrate Solution Method. The side spout of the flask in which 200 g of PH_3-fumigated and -aired grains was sealed gas tight was connected to the butt of an injection needle through a 6-cm-long polyethylene tubing. The butyl rubber septum closing the mouth of a 6-mL tube containing 3 mL of $AgNO_3$ solution was pierced so that the needle remained below the surface of the solution. PH_3 desorbed from the grain passing through the tubing reacts with $AgNO_3$ as it comes out of the needle, forming the characteristic chromophore. At the end of 24 h, the tube containing the chromophore was pulled out and another similar tube containing 3 mL of $AgNO_3$ solution was connected to the needle as before. The absorbance of the chromophore was read at 400 nm against blank. The values were used for calculating the amount of PH_3 desorbed (Rangaswamy, 1984).

2. Detector Strip Method. As in the above experiment, a glass test tube containing a detector strip (1 cm wide × 6 cm long) was attached to the side spout of the flask containing 200 g of grain. At the end of every 24 h, the tube containing the detector strip was pulled out, the red band portion of the detector strip was cut out and a fresh strip placed in the tube if needed, and the tube was refixed to the side spout of the flask quickly. The area (cm^2) of the red band was used to find the micrograms of PH_3 desorbed by the linear relationship between area vs. micrograms of PH_3. Comparison of the amounts of desorbed PH_3 from wheat and raw polished rice by these two methods is shown in Table I.

Desorption Experiments. A 200-g portion of cereal grain, aired for 48 h, was sealed (four replicates) in 250-mL Erlenmeyer flasks with side spouts. A glass test tube containing a detector strip (Kashi and Muthu, 1975) was fixed gas tight to the side spout of the flask as above. The flasks were stored in the laboratory at room temperature avoiding direct sun on the flasks. Fresh detector strips were placed in the tube as and when needed after cutting out the red band at the end of every 24 h. In order to gauge the amount of phosphine desorbed regularly, the storage was continued for 60 days. The phosphine residue on each day of storage from 0 to 60 days was computed by observing the amount of total PH_3 desorbed over the entire period. The amount of PH_3 (ppm) desorbed over 60 days was taken as the total PH_3 residue in the commodity before sealing with the detector strip. The amount of PH_3 desorbed over the next 24 h was subtracted from this total value successively from 0 to 59 days to get the

Table I. Comparison of the Amounts of Desorbed PH_3 (ppm) by the Detector Strip and Silver Nitrate Solution Methods[a]

days of storage	wheat		raw polished rice	
	$AgNO_3$ soln	strip	$AgNO_3$ soln	strip
1	0.057 ± 0.001	0.053 ± 0.001	0.061 ± 0.003	0.06 ± 0.01
2	0.071 ± 0.001	0.07 ± 0.00	0.069 ± 0.003	0.06 ± 0.001
3	0.078 ± 0.001	0.08 ± 0.001	0.041 ± 0.001	0.047 ± 0.002
4	0.076 ± 0.001	0.077 ± 0.001	0.067 ± 0.001	0.066 ± 0.001
5	0.078 ± 0.001	0.073 ± 0.002	0.051 ± 0.001	0.0533 ± 0.001
6	0.056 ± 0.001	0.061 ± 0.001	0.036 ± 0.00	0.035 ± 0.001
7	0.070 ± 0.00	0.075 ± 0.001	0.037 ± 0.001	0.037 ± 0.001

[a] Values are means ±SD of four replicates.

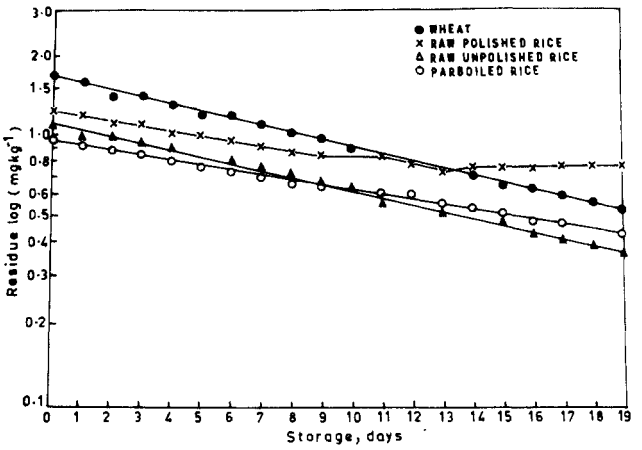

Figure 1. Fall of PH_3 residue as log (ppm) between days 0 and 19 in storage (cereals were fumigated at 0.3 g pellet/kg for 2 weeks and aired for 48 h before storage).

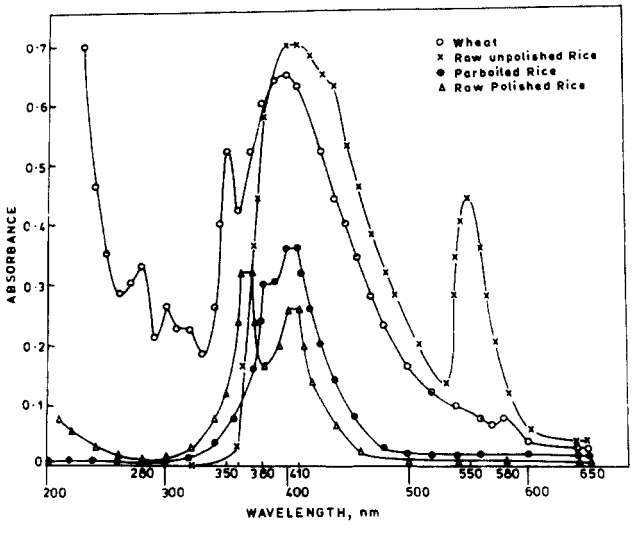

Figure 2. Absorption spectra of PH_3 residue–$AgNO_3$ chromophore from different types cereals.

residue for the subsequent day.

The fall of the PH_3 residue in four types of cereals during the first 19 days of storage, the absorption spectra of the $AgNO_3$–PH_3 residue chromophore, and the fall of the PH_3 residue in four types of cereals during the last 10 days of storage are shown in Figures 1–3, respectively.

Phosphorus Determinations. At the end of 60 days, the dusty patches formed on the inner walls of test tubes due to oxidation of PH_3 were used to determine the amount of phosphorus compounds formed. The dust was dissolved in hot distilled water containing a small amount of 2 N H_2SO_4. Complete dissolution was effected by scratching the inner walls of the test tube with a glass rod. The washings and scratchings were repeated 3–4 times, and then the combined washings were made up to a known volume (10 mL). This solution was reddish due to the dye

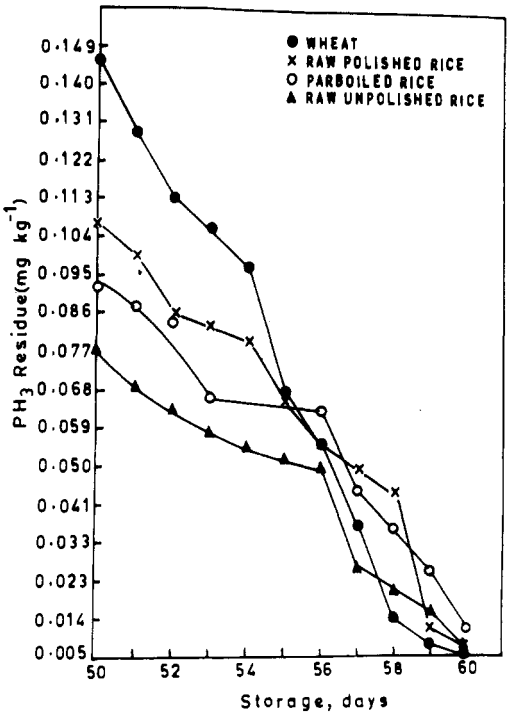

Figure 3. Fall of PH_3 residue (ppm) between days 50 and 60 in storage.

of the paper strip detector adhering to the walls of the tube. A 5-mL aliquot of this solution was employed for determination of phosphorus (Bruce et al., 1962). The amount of phosphorus was calculated from the calibration line drawn by employing KH_2PO_4 as the standard by the same method.

RESULTS AND DISCUSSION

The values of PH_3 desorbed (ppm) from 200 g of wheat and raw polished rice over a period of 8 days obtained by the detector strip method and $AgNO_3$ solution method shown in Table I indicate that there are no significant differences between the two methods. So, due to the ease of analysis afforded by the strip method, all further experiments on desorption of PH_3 from cereals were done by using this method. Cutting of the red band and refixing of the tube with a fresh detector strip are done in about 15–20 s. The amount of desorbed PH_3 lost to atmosphere during this time is extremely negligible when compared to the amount desorbed over 24 h; hence, this loss has not been taken into account.

Figure 1 shows that the fall of the PH_3 residue is not only regular but also linear for the first 19 days of storage for wheat, raw unpolished rice, and parboiled rice. The linearity stops at 9 days for raw polished rice; then after, the curve runs almost parallel to the day axis. This linear relationship of the fall of log (residue) in storage indicates that the desorption of PH_3 from these three types of cereals follows first-order kinetics for 19 days and that from

Table II. PH_3 Residues in Commodities and Inorganic Phosphorus Residues on the Walls of the Side Glass Tube (Fumigated with a 0.3 g Pellet/kg)[a]

commodity	init res before airing, mg kg^{-1}	res after airing for 48 h, mg kg^{-1}	phosph res,[b] μg	$-K$
wheat	2.69 ± 0.04	1.56 ± 0.004	30.4 ± 1.01 (33.36)	0.45
raw polished rice	2.04 ± 0.02	1.29 ± 0.004	33.25 ± 2.2 (36.50)	0.41
raw unpolished rice	2.03 ± 0.002	1.15 ± 0.002	36.46 ± 2.5 (40.01)	0.53
parboiled rice	2.79 ± 0.29	0.98 ± 0.002	37.06 ± 2.2 (40.67)	0.32

[a] Values are means ±SD of four replicates. Values in parentheses indicate the corresponding micrograms of PH_3. [b] On walls of side tube due to oxidation of phosphine desorbed from 200 g of grains of 60 days.

Table III. Phosphine (ppm) Desorbed between Days 20 and 49 during Storage[a]

days in storage	wheat	raw unpolished rice	raw unpolished rice	polished rice
20	0.01 ± 0.012	0.024 ± 0.004	0.13 ± 0.001	0.034 ± 0.003
25	0.08 ± 0.002	0.18 ± 0.01	0.06 ± 0.02	0.11 ± 0.01
30	0.12 ± 0.02	0.14 ± 0.02	0.07 ± 0.01	0.08 ± 0.01
35	0.06 ± 0.01	0.05 ± 0.01	0.064 ± 0.009	0.04 ± 0.00
40	0.04 ± 0.005	0.035 ± 0.003	0.04 ± 0.006	0.02 ± 0.001
45	0.075 ± 0.007	0.04 ± 0.01	0.03 ± 0.02	0.023 ± 0.002
49	0.039 ± 0.004	0.038 ± 0.01	0.016 ± 0.009	0.02 ± 0.01

[a] Values are means ±SD of four replicates.

raw polished rice only up to 9 days. This linear relationship can be represented as

$$\log (a - x) = (-K/2.303)t + \log a$$

where $a - x$ is the residue (mg kg^{-1}) at 24 h, t = 24 h (at the end of which the residue is computed), $-K/2.303$ is the slope of the line, a = initial PH_3 residue (mg kg^{-1}). Therefore, log a is a constant, and K is a constant of desorption; the negative sign for K appears in the equation because of desorption.

In Figure 1 a plot of PH_3 residue log $(a - x)$ (mg kg^{-1}) on the y axis and time of storage in days on the x axis has afforded straight lines over certain days of storage, indicating the desorption of PH_3 from these four types of cereals follows first-order kinetics.

On the basis of the values of slope of each line, values for K of the four types of cereals have been calculated and are shown in Table II.

It can be seen that raw unpolished rice has the higher coefficient of desorption (0.53); next in order are wheat (0.45), raw polished rice (0.41), and then parboiled rice (0.32). As can be seen from Figure 1, the larger angle is subtended by the line corresponding to parboiled rice, indicating a slow rate in fall of log (residue) due to slow desorption, hence having the lowest value for K. Similarly the line corresponding to raw unpolished rice subtends the smallest angle, indicating the fast fall of log (residue), suggestive of fast desorption hence having the highest value for K. On the basis of the amount of phosphorus formed (Table II) it can be inferred that 0.17–0.20 ppm of PH_3 desorbed from 200 g of cereals has undergone oxidation over 60 days in the side tube, which has not been included in the cumulative PH_3 residue after 48 h airing (Table II).

It is pertinent to mention here that in no commercial fumigation is the fumigated material aired by spreading as a thin layer in the open as was done in these model experiments. In spite of airing in the open for 48 h such high cumulative PH_3 residue (1.5556 ppm) was observed in wheat after 48 h of airing. This is in contrast to the observation of Dumas (1980) who states that most of the PH_3 is desorbed from wheat during 2–3 days. In commercial fumigation, be it of stacks under a gas-proof sheet or open grains in silo, one would expect higher residue as the fumigated cereals are not spread in the open to air.

Such prolonged desorption of PH_3 even from aired commodities cannot be only from physically bound PH_3 residues. Hence, these results are suggestive of some kind of loose, reversible interaction of PH_3 with low electron density sites of constituents of grain as PH_3 is weakly nucleophilic in nature. Further evidence for such interaction is provided by the absorption spectra of the $AgNO_3$–PH_3 chromophore from four types of fumigated cereals (Figure 2), while corresponding crop control has no absorption band at these wavelengths. It can be seen (Figure 2) that the spectrum of the $AgNO_3$–PH_3 chromophore from the fumigated cereals before airing, taking all four cereal types, has a band at 400 nm corresponding to free PH_3 residue (Rangaswamy, 1984). In addition to this band, additional bands suggestive of interaction are shown: wheat, 280, 300, 350, 580 nm; raw unpolished rice, 550 nm; parboiled rice, 380–390 nm; raw polished rice, 350–360 nm. Results of experiments under progress with PH_3-fumigated wheat types and their flour have indicated that when physically bound PH_3 residue is below the estimatable limits of the method (Rangaswamy, 1984), the band at 400 nm either disappears or is very much diminished in intensity, while the other bands continue to appear in varying intensities. This may partly explain that the slow desorption of PH_3 in smaller quantities detected by the strip is due to decomposition of these interaction complexes. Hence, in commercial fumigation with AlP, due to longer contact of PH_3 with commodities since they are not aired in the open, chances of formation of such loose interaction complexes are high. These perhaps ultimately may partly decompose to desorb as PH_3. It is interesting to note that the additional bands either are not found or diminish significantly in intensity when desorption of PH_3 is nil over 24–48 h. This may be one of the reasons for continuous evolution of PH_3 for more than 18 months from commercially fumigated wheat. Similar observations have also been made by Dumas (1980) who found small amounts of PH_3 desorption from wheat after 220 days of aeration. The situation becomes complex due to repetitive fumigation of the store with AlP at regular intervals of 3–6 months to control infestation. Although chemisorption of PH_3 with constituents of commodities has been discounted (Rauscher et al., 1972), there is no reason to preclude such possibilities as shown by additional bands (Figure 2).

The dose of fumigant AlP (0.6 g) for 2 kg of cereals employed in these model studies is very high, but such high residues are not uncommon in my experience, particularly in those grains that are nearer to the AlP tablet. In ad-

dition, experiments under progress with one-third of this dose also indicate the same trend of results except that the initial residue levels of PH_3 are small.

During 48 h of airing the loss of initial PH_3 residue is about 42% in wheat, 37% in raw polished rice, 43% in raw unpolished rice, and 65% in parboiled rice (Table II). Examination of the amount of PH_3 desorbed from the four types of cereals shown in Table III indicates that between days 25 and 30 there is a regular increase in desorption of PH_3 from wheat, while it is decreasing in parboiled rice. During this period, desorption of PH_3 from raw polished and raw unpolished rice types has almost remained constant. Between days 30 and 35 desorption has decreased by half in these cereals except in raw unpolished rice. Between days 35 and 49 there are waxing and waning in desorption of PH_3 from wheat, as for example on day 45 desorption is twice that on days 40 and 49, respectively, while desorption has almost remained constant in raw polished rice during this period. Similarly, desorption has remained constant over days 40 to 45 from raw unpolished rice and days 40 to 49 in parboiled rice. On day 49 wheat and raw polished rice were desorbing nearly twice the amount of PH_3 compared to that from raw unpolished and parboiled rice. In addition to differential sorptive capacity of these cereals to retain PH_3, differential rates in decomposition of loosely chemically bound residues in part may be the reason for such variations in desorption over long periods. Absorption spectra of $AgNO_3$–PH_3 chromophores from these four types of cereals (Figure 2) also substantiate such observations.

After 19 days of storage, the relationship between log (residue) vs. days in storage is no longer linear. So the desorption coefficient $-K$ on any day (between 20 and 60 days) of these four types of cereals can be calculated by the relation

$$-K = (2.303/t) \log [a/(a - x)]$$

The fall of the residue due to desorption of PH_3 between days 50 and 60 in storage is shown in Figure 3. Wheat and raw polished rice, although somewhat slow in desorption, show continuous a fall in their residue levels. The rate of desorption in raw unpolished rice and parboiled rice is much slower compared to that of wheat and raw polished rice. There is very little fall in PH_3 residue due to desorption in parboiled rice between days 53 and 56 while the fall is very slow in raw polished rice between days 50 and 56; then after in both, the fall is similar to that of wheat and raw polished rice.

Although regular study of desorption was discontinued at 60 days, flasks containing the cereals were stored up to 84 days. The amounts of PH_3 (mg kg^{-1}) desorbed over 24 days between days 60 and 84 are 0.0041 ± 0.0 (wheat), 0.0066 ± 0.0031 (raw polished rice), 0.0554 ± 0.0004 (raw unpolished rice), and 0.0554 ± 0.0007 (parboiled rice), the last two showing significant desorption. Commodities were still desorbing even after 100 days of storage.

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