Table V. Dalapon Residue in Milk, Test 2

Days after Feeding Begun	Residue, P.P.M.										
	Cow T-1			Cow T-2			Cow T-3				
	Rep. 1	Rep. 2	Av.	Rep. 1	Rep. 2	Av.	Rep. 1	Rep. 2	Av.		
3	0.04			0.04			0.17				
9	0.05	0.05	0.05	0.13			0.34				
12	0.04	0.05	0.05	0.10	0.09	0.10	0.28	0.25	0.27		
16	0.06			0.21	0.22	0.22	0.28	0.24	0.26		
18	0.06	0.06	0.06	0.17	0.16	0.17	0.42	0.39	0.41		
23	0.05	0.05	0.05	0.14	0.13	0.14	0.36	0.34	0.35		
25	0.04	0,05	0.05	0,23	0.16	0.20	0.41	0.41	0.41		
27		••	••	0.22	0.28	0.25	0.35	0.35	0.35		
			Feeding	g Stopped	l on 27th	Day					
30				0.10			0.16				
32	• •			0.05	0.04	0.05	0.05	0.05	0.05		
34	• •	••		Nil	Nil	Nil	0.02	0.02	0.02		

Table VI. Dalapon Residue in Milk, Test 3

Days after	Residue, P.P.M.									
Feeding Begun	Cow T-4	Cow T-5	Cow T-6	Cow T-7	Cow T8	Cow T-9				
6	0.44	0.82	0.61	0.59	0.28	0.53				
8	0.47	0.45	0.51	0.41	0.61	0.72				
12	0.32	0.59	1.06	0.62	1.27	0.80				
14	0.28	0.45	0.60	0.89	1.25	0.80				
16	0.12	0.35	0.89	0.63	0:98	0.68				
20	0.19	0.28	0.70	0.51	1.11	0.63				
22	0.15	0.26	0,50	0.46	0.92	0.47				
26	0.33	0.32	0.77	0.70	1.06	0.68				
55	0.25	0.39	0.99	0.55	••	0.60				

Extract the ether solution with bicarbonate as in step 5 of the distribution procedure and determine dalapon in the extract as in step 6.

Blanks and Recovery Data. Samples of control milk, obtained from the various cows before feeding dalapon, were analyzed by the procedures just described to establish "blank" values (Table II). No variation in blanks among the various animals was found.

To determine the efficiency of the distribution procedure in recovering both combined and uncombined dalapon from milk, synthetic knowns were prepared by adding various amounts of dalapon and its glycerol tris ester, in various ratios, to control milk samples. These fortified samples were then subjected to the analytical scheme to determine per cent recovery. The results, summarized in Table III, show that recovery is complete.

Table III also summarizes the results obtained by applying the elution procedure to milk samples fortified with various amounts of dalapon.

#### Analytical Results

Test 1 (Cow S-1). Five milk samples were analyzed in duplicate for both combined and free dalapon. The data, presented in Table IV, show the absence of any significant quantity of combined dalapon. After this fact had been established, the remaining results were obtained by using the shorter elution procedure. Milk collected 17 days after feeding of dalapon had begun was analyzed by both methods and excellent agreement was obtained.

The data relating to uncombined dalapon are plotted in Figure 1. The average residue (10th through 55th day) of 0.69 p.p.m. is plotted in Figure 2.

Test 2 (Cows T-1 to T-3). Sufficient analyses, using the elution procedure, were run in duplicate to establish the residue levels in milk from cows fed 20, 50, and 100 p.p.m. of dalapon, respectively. These data are presented in Table V and plotted in Figure 1.

Test 3 (Cows T-4 to T-9). Analysis of milk samples from cows T-4 and T-5,

fed 100 p.p.m. of dalapon, cows T-6 and T-7, fed 200 p.p.m., and cows T-8 and T-9, fed 300 p.p.m., yielded the data shown in Table VI and plotted in Figure 1. Single determinations were made, since good agreement was obtained between duplicate samples in the two previous tests.

#### **Discussion and Conclusions**

The data presented show clearly that a residue to dalapon does appear in milk from cows fed the herbicide. Since no significant quantity of dalapon glycerides could be detected, the residue probably exists only as a simple salt. The magnitude of the residue appears to be directly proportional to the quantity ingested by the animal over the range studied. This is shown in Figure 2, where average residue levels are plotted against corresponding ingestion levels. The slope of the curve indicates that the level of residue to be expected in milk is approximately 0.3% of the level ingested.

The reason for the abnormally low residue found in milk from cow T-9 is not apparent. It is true that milk production by this animal was somewhat higher than for others in test 3 (see Table I). However, there appears to be no relation between residue level and milk production. The data from cow S-1 show good agreement of residues with those from other cows fed at the same level, despite the cow's low milk production rate.

### Literature Cited

(1) Schreiber, M. M., J. Agr. Food Chem. 7, 427-9 (1959).

(2) Smith, G. N., Getzendaner, M. E., Kutschinski, A. H., *Ibid.*, **5**, 675-8 (1957).

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## Correction

# Preparation of Labeled 2-Ethylthioethanol, a Demeton Intermediate

In this article by Kermit Groves and Roger Haugwitz [J. AGR. FOOD CHEM. 9, 262 (1961)], the name of the second author was misspelled. The correct spelling is Haugwitz.