

cloxacillin in the uncooked edible tissues of cattle and in milk.

[40 FR 28792, July 9, 1975]

§ 556.167 Colistimethate.

A tolerance for residues of colistimethate in the edible tissues of chickens is not required.

[63 FR 13123, Mar. 18, 1998]

§ 556.170 Decoquinatate.

Tolerances for residues of decoquinatate in food are established as follows in uncooked edible tissues of chickens, cattle, and goats at 2 parts per million in tissues other than skeletal muscle and 1 part per million in skeletal muscle.

[52 FR 43061, Nov. 9, 1987]

§ 556.180 Dichlorvos.

A tolerance of 0.1 part per million is established for negligible residues of dichlorvos (2,2-dichlorovinyl dimethyl phosphate) in the edible tissues of swine.

§ 556.200 Dihydrostreptomycin.

Tolerances are established for residues of dihydrostreptomycin in uncooked, edible tissues of cattle and swine of 2.0 parts per million (ppm) in kidney and 0.5 ppm in other tissues, and 0.125 ppm in milk.

[59 FR 41977, Aug. 16, 1994]

§ 556.220 3,5-Dinitrobenzamide.

No residues of 3,5-dinitrobenzamide may be found in the uncooked edible tissues of chickens as determined by the following method of analysis:

I. *Method of analysis—3,5-dinitrobenzamide.* A method for 3,5-dinitrobenzamide (3,5-DNBA) in chicken tissues is described with a cleanup step that removes most of the interfering materials, thus allowing uncompensated measurements to be read. The 3,5-DNBA is extracted from the sample with acetone and chloroform and prepared for chromatography by removing the aqueous phase in a separatory funnel and the solvents in a flash evaporator. The extract residue is chromatographed on alumina to remove several lipid components and residues of other drugs. The benzamide eluate is passed through a column of Dowex-50 resin, or equivalent, to remove arylamines; for example, 3-amino-5-nitrobenzamide. The 3,5-DNBA

fraction is reduced, after removal of alcohol, with $TiCl_3$ in basic solution to an arylamine, presumably 3,5-diaminobenzamide. The reduced fraction is placed on another Dowex-50 column, most of the interfering substances are removed with washings of alcohol and water, and the arylamine residue is eluted with 4*N* HCl. Colorimetric measurement is made in a 100-millimeter cell at 530 millimicrons after reacting the residue with Bratton-Marshall reagents.

II. *Reagents.* A. Acetone.

B. Acetyl-(*p*-nitrophenyl)-sulfanilamide (APNPS) standard—melting point range 264° C.–267° C. Weigh and transfer 10 milligrams of APNPS to a 100-milliliter flask, dissolve and dilute to volume with acetone.

C. Alumina—activated F-20, 80–200 mesh, Aluminum Co. of America, or equivalent substance.

D. Ammonium sulfamate.

E. Ammonium sulfamate solution 1.25 grams of ammonium sulfamate per 100 milliliters of water. Refrigerate when not in use. Prepare fresh weekly.

F. Cation-exchange resin—Dowex 50W-X8, 200–400 mesh, Baker Analyzed Reagent, or equivalent, prepared as follows:

1. Place 500 grams of resin into a 3-liter beaker.

2. Add 2,000 milligrams of 6*N*HCl.

3. Heat and stir while on a bath at 80° C. for 6 hours. Discontinue heating and continue stirring overnight.

4. Filter the resin on a Buchner funnel (24 cm.) fitted with Whatman No. 1 paper.

5. Wash the resin bed with four 500-milliliter portions of 6*N*HCl.

6. Wash the resin bed with 500-milliliter portions of deionized water until the effluent has a pH of 5 or higher.

7. Wash the resin bed with three 400-milliliter portions of specially denatured alcohol 3A. Drain thoroughly.

8. Make a slurry of resin in 1,250 milliliters of specially denatured alcohol 3A.

G. Chloroform.

H. Coupling reagent—0.25 gram of *N*-1-naphthyl-ethylenediamine dihydrochloride per 100 milliliters of water. Refrigerate when not in use. Prepare fresh weekly.

I. 3,5-Dinitrobenzamide (3,5-DNBA standard). Add to boiling specially denatured alcohol 3A until a saturated solution is obtained and treat with activated carbon, filtered and crystallize by cooling to room temperature. The 3,5-DNBA therefrom is treated a second time with activated carbon and then recrystallized three more times from specially denatured alcohol 3A. The third crystallization is washed with diethyl ether and dried in a vacuum desiccator, melting point range 185° C.–186° C.

J. Ethyl alcohol—absolute, A.C.S.

K. Eluting reagent A. The formula and volume required in procedure step V-D is dependent on the adsorptive strength of the Al_2