

Improved Determination of Chromic Oxide in Cow Feed and Feces

FUMIKO T. KIMURA and
V. L. MILLER

Western Washington Experiment
Station, State College of Washing-
ton, Puyallup, Wash.

Chromic oxide is widely used as a reference substance in nutrition investigations of domestic animals. An analytical method that is simple, specific, rapid, and requires only common laboratory equipment, may be used to determine chromic oxide in feeds and feces in the amounts commonly used in digestibility experiments with dairy cattle.

CHROMIC OXIDE (Cr_2O_3) is widely used as a reference substance for studies of digestibility of domestic animal feed and forage. Kane, Jacobson, and Moore (4) described a volumetric method modified from that of Edin, Kihlen, and Nordfeldt (3), using sodium peroxide fusion for the oxidation of the chromic oxide to dichromate. However, these methods are tedious. Bolin, King, and Klosterman (7) reported a colorimetric method using sulfuric-perchloric acid mixture with molybdate catalyst as oxidant. Low results may be obtained with this mixture with excessive heating, because the formation of hydrogen peroxide reduces the hexavalent chromium (5). However, Day (2) reported that this difficulty may be overcome by carefully controlled time, temperature, and acidity.

A colorimetric method is described, which uses a nitric-perchloric acid oxidation with molybdate catalyst. Time, temperature and acidity are not critical. This method requires only common laboratory equipment and eliminates the need for constant attention (2).

Experimental

All reagents used meet ACS specifications.

Analytical Procedure. Transfer a 1-gram sample containing 3 to 7 mg. of chromic oxide to a 100-ml. borosilicate glass volumetric flask. Add 1 to 5 mg. of sodium molybdate and 10 ml. of nitric acid. Boil gently on a hot plate until the acid is about half gone but not less than 10 minutes. Remove the flask, cool slightly, and add 5 ml. of 70% perchloric acid. Boil with stronger heat, mixing well to complete oxidation in 10 to 15 minutes, and continue heating for 2 to 3 minutes. Remove the flask from the hot plate. Allow to cool to room temperature and make up to volume with water. Centrifuge, or let stand to allow silica to settle.

Compare the sample solution with a standard curve prepared from known amounts of potassium dichromate solution of approximately the same acidity as the sample, to give a range of 10 to 80 γ of chromic oxide per ml. Determine the percentage transmittance in an Evelyn photoelectric colorimeter using the 440-m μ filter.

Discussion

One-gram samples of feces containing approximately 6, 13, and 19 mg. of chromic oxide were oxidized with 3, 5, and 7 ml. of perchloric acid. The use of 7 ml. of acid offered no advantages over

5 ml., but when 3 ml. was used the resultant solution was too viscous.

Different portions of the same sample were oxidized, using the described method, and the effect of time of digestion was observed. The data recorded in Table I show that heating for 15 or 20 minutes after the completion of oxidation had no detrimental effect. Rapid cooling and dilution as reported by Day (2) were not necessary.

Table II shows the recovery of known amounts of chromic oxide in the range of 3 to 38 mg. The standard deviation is ± 0.018 mg. in the range of 0.3 to 0.8% chromic oxide, most frequently encountered in digestion experiments.

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Literature Cited

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Table I. Effect of Extra Heating on Recovery of Chromic Oxide

Heating Time, Min.	Chromic Oxide Present, Av. Mg.		
2	6.27	13.0	18.9
5	6.27	13.1	19.0
10	6.22	13.0	19.0
15	6.22	13.0	18.9
20	...	13.0	19.0

Table II. Accuracy of Method

Chromic Oxide		
Weighed, mg.	Found, mg.	Recovered, %
3.30	3.29	99.7
5.95	5.90	99.2
5.25	5.21	99.2
6.40	6.40	100.0
7.95	7.92	99.6
10.1	10.1	100.0
10.7	10.6	99.1
14.4	14.3	99.3
15.1	14.8	98.0
15.1	15.1	100.0
26.9	27.0	100.4
38.4	38.4	100.0