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A Preliminary Assessment of Herbicide Quality in Mali in the Presence of Proliferating Unregistered Brands of Glyphosate

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ABSTRACT

The preliminary analysis reported in this paper suggests that farmers have good reason to complain about the unreliable quality of glyphosate products currently available for sale in Mali. Of the 100 samples tested from four major agricultural markets in Mali, over one-third were either under-dosed or over-dosed.

Unregistered products are of lower quality, on average, than duly registered brands. All other factors equal, an unregistered brand is likely to have 16% less active ingredient than registered products. An unregistered salt formulation purchased in the Bamako Central market contains, on average, has half the active ingredient stated on the bottle label. Little wonder farmers complain about variable response rates, even when comparing outcomes from different bottles of the same brand.

The widespread availability of unregistered glyphosate products, combined with their highly variable quality, suggests that regulators and farmer advocate organizations will need to patrol markets and enforce regulations far more effectively than they have in the past. The wide margin of error found in these laboratory tests suggests that investments in improved laboratory equipment and processes will be important as part of overall efforts to improve regulatory oversight of herbicides and other pesticides.

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ACRONYMS

CILSS	Comité Permanent Inter-états de Lutte contre la Sécheresse au Sahel
CNGP	Comité National de Gestion des Pesticides
COAHP	Comité Ouest Africain d'Homologation des Pesticides
CSP	Comité Sahélien des Pesticides
DNA	Direction Nationale de l'Agriculture, Ministry of Agriculture
DPV	Direction de la Protection des Végétaux, Ministry of Agriculture
ECOWAS	Economic Community of West African States
LCAE	Laboratoire Central d'Agrochimie et l'Ecotoxicologie
LNPV	Laboratoire National de la Protection des végétaux
MC	Ministry of Commerce
ME	Ministry of Environment
MOA	Ministry of Agriculture
OMA	Observatoire du Marché Agricole Mali
UEMOA	Union Economique et Monétaire Ouest Africaine (WAEMU in English)

1. INTRODUCTION

Sales of glyphosate, the world's most widely purchased herbicide, have increased rapidly in Mali over the past decade and a half, driven by falling herbicide prices and rising wage rates for hand weeding labor. In Southern Mali, where about 60% of sorghum and maize farmers use herbicides, glyphosate accounts for two-thirds of the volume of herbicides used (Haggblade et al. 2017). Glyphosate prices in Mali have fallen by roughly 50% since 2000, triggered by expiring global patent protection and the emergence of large numbers of Chinese suppliers in global markets (Huang et al. 2017). In Mali, as in much of West Africa, a growing cohort of local traders supply a proliferating number of glyphosate brands. While regulators had approved only 1 brand of glyphosate for sale in 1995, by 2015 regulators had approved a total of 38 glyphosate products for sale.

Sales of unregistered and counterfeit herbicides have increased as well (Figure 1). Market visits and farm household surveys in Mali indicate that unregistered products may account up to 45% of glyphosate purchases (Haggblade et al. 2017). Not surprisingly, farmers complain of uneven product quality (Keita et al. 2017). In village focus groups, Malian farmers frequently voice frustration their inability to evaluate quality from closely mimicked packaging of unregistered brands (Figure 2).

Farmer perceptions of product quality clearly affect their demand for productivity-enhancing inputs (Ashour et al. 2016). The widespread presence of unregistered glyphosate products risks depressing farmer demand for an important productivity-enhancing input. Uncertain product quality poses unknown environmental and health risks as well.

The problem of variable pesticide quality appears significant across West Africa. Amid wide variation, a study across eight West African countries finds over 60% of pesticide products unregistered on average (Mir-Plus 2012, Table 9). In Gambia, laboratory analysis of 128 pesticide products on sale in local markets found only 10% labeled. Among the 90% unlabeled, 28% contained banned substances, primarily highly toxic insecticides (Murphy et al. 2012). In Uganda, laboratory analysis of commercial glyphosate samples found that one-third of products tested contained below 75% of the stated concentration levels of active ingredient. Similar quality assessments based on farmer recall estimates of inputs and outputs, not laboratory measurement, finds that registered herbicides reduce labor requirements for weeding by twice as much as unregistered products, suggesting significantly higher quality of registered products (Assima et al. 2016). Recent studies in Ethiopia and across half a dozen countries in West Africa suggest that herbicide markets have grown much faster than regulatory capacity over the past decade and a half (Tamru et al. 2017; Haggblade et al. 2017b; Traoré and Haggblade, 2017). In this environment, of rapid market growth and weak regulatory monitoring capacity, quality problems seem likely to increase. Because growth in glyphosate use has surged only over the past decade in most of Africa, and because glyphosate dominates among herbicides used, quality testing of this single active ingredient offers an unusual opportunity to verify the range of quality currently available to farmers in registered and unregistered glyphosate brands.

Figure 1. A Partial Array of Glyphosate Brands Sold in Mali

a. Roundup and imitators (above)

b. Glycel and imitators (above)

This paper aims to evaluate the quality of glyphosate products currently available to farmers in Mali. Beyond its obvious importance to Malian farmers and regulators, this quality assessment holds much wider significance. Quality problems plague many major agricultural inputs in West Africa, including fertilizer, insecticides, seeds and herbicides (Sheahan and Barrett 2015; Theriault, 2016). Uncertainties about stated dosages and concentration levels lead to potentially high variability in on-farm outcomes as well as inefficiencies stemming from under- and over-dosing plots. These uncertain outcomes, in turn, tend to depress demand for these productivity-enhancing inputs.

This paper begins with a description of the data and methods. It then reports laboratory testing results from 100 glyphosate samples purchased in four major markets across Mali. The discussion and conclusions explore key policy implications for regulators, herbicide suppliers and farmers. Given rapid growth in many African herbicide markets over the past decade, and reported uneven levels of product quality elsewhere, these results should provide interest to broader efforts to promote farm productivity growth via input intensification (Ashour et al. 2016, Haggblade et al. 2017b; Mir-Plus 2012, Murphy et al. 2012; Shaheen and Barrett 2016).

Figure 2. Packaging similarities between registered and unregistered herbicides sold in Mali

a. Roundup and imitation

b. Glycel and imitation "Red Beret"

2. DATA AND METHODS

2.1. Glyphosate samples

The study team aimed to procure glyphosate samples from a range of different agro-ecological zones and agro-dealers. Collection therefore focused on agricultural input suppliers in four major market centers: Bamako (the capital city and cite of most major import warehouses), Niono (a major agricultural market town in the irrigated rice zone of central Mali roughly 300 kilometers north of Bamako, along the Niger River), Koutiala (a horticultural hub 275 kilometers east of Bamako) and Sikasso (the market center for the high-rainfall southern zone of Mali, 300 kilometers southeast of Bamako). Among Bamako's many markets, the team selected the central market in the river quarter as well as smaller markets of Bozola and Kati. Niono, Koutiala and Sikasso purchases took place from the main market in each town. The team targeted procurement of 40 sample products from Bamako and 60 from the other market centers.

In each location, the team first consulted with the local agro-dealers' association to obtain a list of all registered input retailers. From this listing, we selected 20 distributors at random. One team member then visited each shop, posing as a farmer, and asked the shop owner to recommend two glyphosate products for him to try on his fields -- the best quality product he sold as well as the cheapest. This procurement protocol aimed to capture a range of glyphosate brands and qualities. In trial runs, we discovered that asking for one registered and one unregistered product quickly made suppliers very nervous, given the illegality of unregistered product sales. However, many farmers ask for cheap options when purchasing, and so this second approach proved to work well. Where stocks permitted, we purchased two one-liter bottles from each supplier. The purchases took place in December 2016.

The resulting distribution of samples included 40 from Bamako, 30 from Niono, 16 from Sikasso and 14 from Koutiala (Table 1). Nearly two-thirds came from China, while an additional 18% came from Europe. Prices ranged considerably, from 2,500 to 8,000 CFAF/liter ($4.30 to $13.80). Fabrication dates likewise suggest a wide range of time in inventory prior to sale. The oldest product listed a fabrication date of February 2009, compared to the most recent in December 2016 (Table 2). Given a mean production date of July 2015, the samples averaged one and a half years old.

In terms of regulatory approval, slightly over half of the products purchased (52) were registered by the Comité Sahélien des Pesticides (CSP). This regional regulatory body reviews all pesticide products (herbicides, insecticides, fungicides and nematicides) prior to their authorization for sale in nine Sahelian countries, including Mali, who regulate pesticides collectively through the regional regulator, CSP (Abiola et al. 2004). Another 6 products were registered elsewhere (mostly in Ghana or Guinea) and smuggled into Mali. Though not registered by the CSP, they did undergo regulatory review in these neighboring countries through their national review systems. The remaining 42 glyphosate products purchased for testing have not undergone regulatory review anywhere (Table 1).

Table 1. Sample characteristics

Sample characteristics categories	sample size	Sample characteristics categories	sample size
		Glyphosate level, as stated on bottle	
Purchase location		356	1
Bamako		360	43
central market	27	410	3
other markets	13	450	12
Niono	30	480	36
Sikasso	16	500	5
Koutiala	14	total	100
total	100		
		Formulation	
		salt	42
Country of fabrication		acid	41
China	63	not indicated	17
Belgium	12	total	100
France	6		
India	2	Registration status	
Ghana	1	CILSS	52
Mexico	1	other	6
not indicated	15	none	42
total	100	total	100

Table 2. Sample summary statistics

Sample characteristics	mean	s.d.	min	max
glyphosate level stated on bottle (g/l)	422	58	356	500
price (CFAF/liter)	3830	985	2500	8000
date of fabrication	12-Jul-15	377	4-Feb-09	24-Dec-16

2.2. Laboratory selection

Mali has no laboratory accredited for formulation verification testing under the international testing standard ISO 17025. Therefore, laboratory testing of the 100 glyphosate samples took place at outside laboratories outside of Mali. Our team selected two laboratories for this purpose, one in West Africa and one in the USA. From each purchased bottle, we pulled two 100 ml samples and sent one batch to each laboratory for independent analysis.

To identify a suitable laboratory in West Africa, we consulted with CSP regulators and major private sector importers to see if they could identify a lab accredited for formulation verification analysis in any of the surrounding countries. As best we could determine, none had yet obtained accreditation. Based on recommendations from our Malian colleagues, we selected a large government laboratory that has begun the international accreditation process. In late February 2017, we shipped one set of 100 mL samples overland to this regional lab in West Africa accompanied by one of our staff members.

To select a suitable, accredited laboratory in the USA, our team contacted three private testing laboratories suggested by Michigan State University's Department of Crop Sciences. Following a review of their experience, testing protocols and facilities, our team selected one of them to conduct the second round of testing. To export the samples to the USA by air, we required an export permit from the Government of Mali as well as an import permit from the US Environmental Protection Agency (EPA). Given delays in receiving approval for shipment, these results were not yet available at the time of this publication. The discussion below, therefore, focuses only on the results reported from Lab #1.

2.3. Sample preparation

In February 2017, a laboratory technician from Mali's national veterinary laboratory pulled two 100 ml samples from each of the 100 one-liter glyphosate bottles purchased. Materials he used for this purpose include a 20 ml pipette, face mask, 200 brand new high-density polyethylene (HDPE) containers with locking caps, laboratory gloves and a work table covered with clean plastic sheeting. He set up a temporary work station on a shaded, well aerated veranda at the MSU Bamako offices. All samples remained indoors at ambient temperature at the MSU office between the time of sample purchase in December 2016 and their shipment to the two laboratories in February and May 2017.

The technician shook all original bottles vigorously before opening them and drawing five 20 ml pipettes to transfer to the 100 ml HDPE sample containers. After filling each sample container, the technician used the locking caps to hermetically seal the HDPE sample containers.

Our research team labeled each 100 ml container with an alphanumeric code to identify the location of purchase as well as the specific brand name and bottle of glyphosate being evaluated. The laboratories received only the sample code numbers. They have no knowledge of the individual brand names, locations of purchase, price, registration status or fabrication location.

The laboratories both conducted their analyses blind, knowing only the sample code numbers (Figure 3). The wide range of colors found among the 100 products tested suggests clear differences in the various glyphosate formulations sold.

Figure 3. Glyphosate samples purchased from Malian pesticide dealers: diverse contents despite allegedly identical active ingredients

2.4. Laboratory testing methods

The West African lab (Lab #1) tested all 100 samples using high performance liquid chromatography with ultraviolet detection (HPLC-UV). The equipment used included an HPLC (PROMIN 20AT Shimadzu) equipped with two pumps (LC 20A); an automatic injector (SIL-20A); a hot column (CTO-20A); Interchrom C18 column (5 µm particle size, Length x I.D: 250 mm x 4.6 mm); Detector Sensitivity (1 AUFS) and a computer system to check solvent gradient.

The laboratory analysis began by preparing a dilution of 1:10,000 using distilled water. From this dilution, the lab technicians derived two solutions, first with fluorenylmethyl chloroformate (FMOC) to obtain a solution of 10 g/L. The second was a 5% solution of tampon borate. In 1 ml of herbicide diluted to 1/10,000, they added 1 ml of solution 2 and(tetraborate 5%) and 1 ml of solution 1 (FMOC). The resulting solution was agitated for one hour in darkness at room temperature, then centrifuged at 4,500 RPM. The resulting supernatant was transferred into a vial and then injected into the HPLC column with the following parameters:

Column:	Lichrospher 100)DS (250 L x 4.6 mm)
Eluent A	50 Mm ammonium acetate (40%)
Eluent B	HPLC grade acentoitrile (60%)
Debit:	0.5 Ml/min
Temperature	30 degrees centigrade
Volume of injection:	5 uL
UV wavelength:	254 nm

To calibrate the glyphosate concentration ratings, the lab prepared a control solution of pure water as well as a pure glyphosate control sample. The retention time of the glyphosate was determined by comparison with glyphosate and blank solutions. The team then prepared five calibrating solutions at known concentrations of 0.167, 1.67, 16.67, 33.33 and 66.67 mg/L glyphosate. Estimation of a linear regression line established interpolation line relating glyphosate concentration (Ga) to peak height (H) of the chromatograph:

Equation 1: $Ga = m H + b$.

The slope coefficient m enables the laboratory to estimate glyphosate concentration (Ga) in mg/L as a function of the chromotagraph's peak height (H), measured in nm.

To validate the calibration, the lab prepared a test solution of glyphosate of 50 mg/L concentration. The team then ran 10 successive tests to assess the range of measurement errors expected. These test runs resulted in concentrations ranging between 41.5 and 50.7 mg/L. The mean was 47.6 with a standard deviation of 3.3 leading to a coefficient of variation (sd/mean) of 6.9.

3. RESULTS

3.1. Duplicate samples

Of the 100 samples submitted to each laboratory, 10 were duplicates. For each pair, the laboratory received two separate 10 ml samples drawn from the same bottle of glyphosate and submitted in two separate specimen bottles (Figure 3) under two different sample numbers. The laboratory did not know of the duplicates or their id numbers. As a result, the labs treated each duplicate as a separate submission.

The duplicate results provide a second check on the reliability of the laboratory testing and calibration procedures. These comparisons suggest extremely close correspondence in three duplicate pairs and unexpectedly wide divergence in two cases (Table 3). Overall, 7 out of 10 pairs produced results within +/- 10% of each other. The three remaining duplicate pairs differed by 14%, 22% and 72%. All three of the large variations emerged from salt-based formulations. This suggests a potentially large margin of error in 30%, particularly among the salt formulations.

Table 3. Dispersion of concentration levels reported for 10 duplicate glyphosate samples

	Sample label information		Laboratory 1 test results		
Duplicate numbers	glyphosate concentration (g/L)	formulation	result 1	result 2	% difference
1	360	acide	480.6	450.8	6%
2	360	acide	360.5	380.7	-6%
3	480	sel	360.2	410.5	-14%
4	480	sel	390.7	360.1	8%
5	500	sel	450.6	550.4	-22%
6	480	sel	360.1	620.1	-72%
7	360	Non indique	380.4	380.5	0%
8	360	acide	450.2	450.1	0%
9	480	sel	360.6	360.9	0%
10	480	sel	360.3	380.3	-6%

The MSU team is currently in discussion with the laboratory technicians at Laboratory #1 to seek clarification and possible remedies, including reanalysis of certain of the submitted formulations. For this reason, we consider the results reported below to be preliminary.

3.2. Ranges of under and over-dosing

On average, the 100 commercial samples tested matched the glyphosate concentration rating reported on the label. The laboratory measured glyphosate concentrations of 408 g/L, on average, or 99% of the average concentration listed on the bottles of 422 g/L.

The minimum ratio of 19% suggests that some products contained less than 20% of the stated concentration level. This would explain why some farmers complain about ineffective results. At the other extreme, the maximum laboratory measurement of 720 g/L was nearly double the standard 360 g/L concentration found on most commercial products available on the market.

Table 4. Summary laboratory results

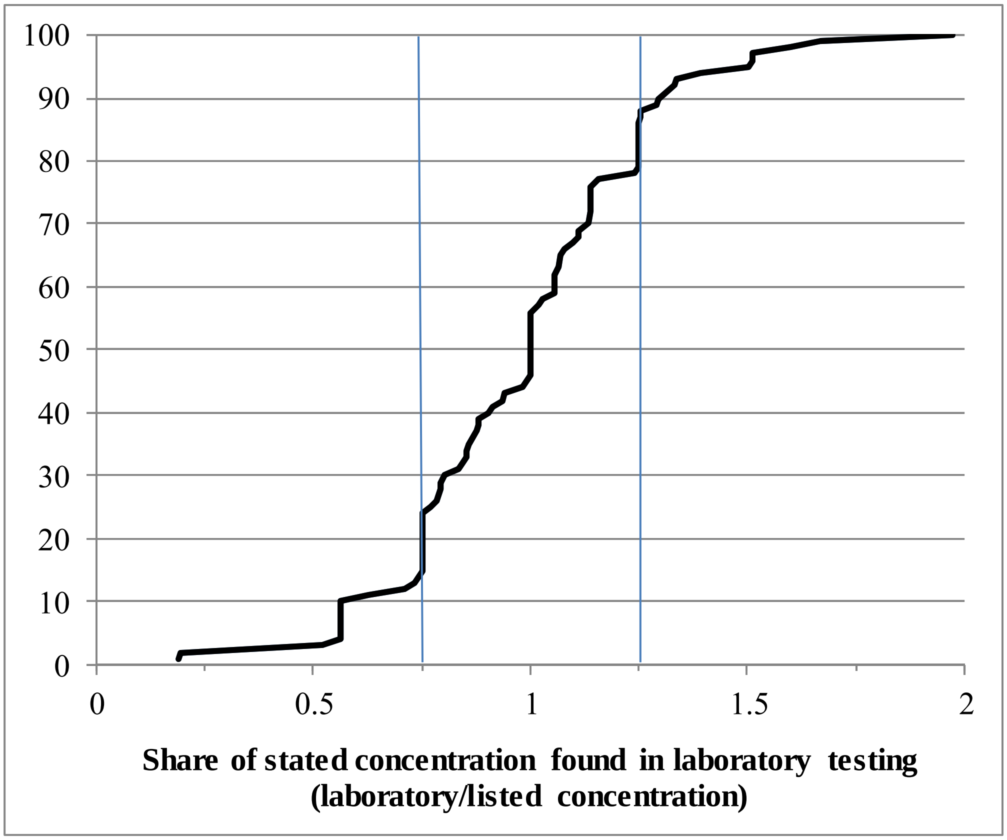
	mean	s.d.	min	max
Glyphosate level (grams/liter)				
stated on bottle	422	58	356	500
laboratory measurement	408	110	87	720
Share (lab/bottle)	0.99	0.30	0.19	1.97

Figure 4 shows the full distribution of dosing percentages. A value of 1.0 indicates that the laboratory test exactly equaled the stated concentration as listed on the label, while a share value of 0.5 indicates that the laboratory found only half as much glyphosate as the label promised. The two vertical lines in Figure 4 mark the range plus or minus 25% of full dosage.

The findings from this cumulative distribution indicate that 24% of samples fall below 75% of stated concentration. We consider these under-dosed. Perhaps surprisingly, a further 12% come in at over 125% of stated concentration.

This range suggests considerable variability in the actual dosage as compared to what the farmer expects. Overall, slightly over one-third of commercial glyphosate products are either under or over-dosed with active ingredient.

Figure 4. Cumulative frequency of measured glyphosate share
(laboratory measurement / stated concentration listed on the bottle)



3.3. Factors affecting herbicide quality

Using simple statistical correlations and regression analysis, Tables 5 and 6 explore various factors that farmers, traders and researchers suggest may affect glyphosate quality, as measured by the share of active ingredient found in commercial samples compared to the dosage promised on the product label.

The simple correlations in Table 5 suggest that several factors correlate most strongly with dosage. First, registration status matters. Unregistered glyphosate brands correlate strongly and negatively with actual dosage share. Second, the type of formulation matters. Acid formulations correlate positively with dosage levels while salt formulations are strongly negatively correlates. Third location of purchase seems to matter, with most underdosed products being sold in the Bamako Central market, a known hotbed of smuggled products.

Table 5. Correlation matrix

	share	unreg	other_reg	europe	china	other_fab	price	BamakoM	salt	acid	date
share	1.000										
unregistered	-0.560	1.000									
other_registration	-0.050	-0.218	1.000								
europe	0.328	-0.265	0.052	1.000							
china	-0.233	0.123	-0.029	-0.535	1.000						
other_fabrication location	0.001	0.075	-0.009	-0.203	-0.719	1.000					
price	0.081	-0.146	-0.043	0.219	-0.464	0.358	1.000				
BamakoMC	-0.355	0.331	0.297	-0.080	-0.003	0.070	-0.013	1.000			
salt	-0.539	0.540	-0.021	-0.344	-0.112	0.413	0.096	0.101	1.000		
acid	0.451	-0.580	0.114	0.422	-0.049	-0.290	0.022	-0.152	-0.696	1.000	
date	-0.063	0.041	0.037	-0.365	0.352	-0.108	-0.166	0.045	0.217	-0.164	1.000

Table 6. Regression results: LSH variable = share (laboratory measurement / content as stated on bottle)

Independent variables	Model 1 coeff.		Model 2 coeff.		Model 3 coeff.		Model 4 coeff.		Model 5 coeff.		Model 6 coeff.	
Registration status (0 = CILSS)												
unregistered	-0.36	***	-0.32	***	-0.33	***	-0.30	***	-0.16	***	-0.12	*
other (non-CILSS)	-0.23	**	-0.22	**	-0.23	**	-0.16		-0.08		-0.06	
Production location (0 = China)												
Europe			0.17	***	0.20	***	0.21	***	0.14	**	0.20	**
other			0.06		0.09		0.10		0.19	***	0.23	***
Price (CFAF/liter)					0.00		0.00		0.00		0.00	
Bamako Central Market purchase							-0.10		-0.13	**	-0.16	**
Salt (0=acid formulation or unspecified)									-0.25	***	-0.29	***
Date fabricated (weeks after earliest sample)											0.001	*
Constant	1.15	***	1.09	***	1.22	***	1.22	***	1.22	***	-4.45	
n	90		90		90		90		90		84	
adj R2	0.3316		0.3636		0.3671		0.3779		0.4632		0.4748	

Significance levels: *** = 99%; ** = 95%; * = 90%.

Many of these factors are inter-related. Most acid formulations come from Europe, while the Chinese manufactured products generally use a salt formulation. Unregistered products, likewise, appear almost exclusively in salt formulations. Table 6 evaluates the approximate impact each factor alone on concentration shares comparing laboratory evaluations with stated content as listed on the bottle labels.

Unregistered products generally have lower content than stated, likely between 12% to 16% less active ingredient than stated on the bottle. Salt-based formulations contain 25% to 29% lower than stated concentration levels; and this correlates closely with unregistered status (Table 6, Models 5 and 6). An unregistered salt-based product has, on average, 41% less active ingredient concentration than reported on the bottle.

Bamako central market purchases, on average, contain 13% to 16% less active ingredient than stated. Fraudulent products appear there frequently. On average, an unregistered glyphosate bottle purchased there would contain 30% less active ingredient than stated on the bottle. European and other non-Chinese suppliers have 14 to 20% more active ingredient than stated on the bottle.

Price, surprisingly, does not affect the reliability of the stated concentration. Nor does old inventory appear to make much difference. A new product compared to one produced one full year earlier (365 days) would have only 0.4% more active ingredient.

4. CONCLUSIONS

This preliminary analysis suggests that farmers have good reason to complain about the unreliable quality of glyphosate products currently available for sale in Mali. Over one-third of products tested were either under-dosed or over-dosed.

Unregistered products are of lower quality, on average, than duly registered brands. All other factors equal, an unregistered brand is likely to have 16% less active ingredient than registered products (Table 6, Model 5). An unregistered (minus 16%) salt formulation (minus 25%) purchased in the Bamako Central market (minus 13%) contains, on average, has half the active ingredient stated on the bottle label. Little wonder farmers complain about variable response rates, even when comparing outcomes from different bottles of the same brand.

The widespread availability of unregistered glyphosate products, combined with their highly variable quality, suggests that regulators and farmer advocate organizations will need to patrol markets and enforce regulations far more effectively than they have in the past. The wide margin of error found in these laboratory tests suggests that investments in improved laboratory equipment and processes will be important as part of overall efforts to improve regulatory oversight of herbicides and other pesticides.

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