The state committee of the Ukraine for in housing and utilities infrastructure

### The Ukrainian state research institute for problems of water supply, water disposal and protection of natural environmental resources "UkrWODGEO"

Outgoing # 175/01/05-09 from August 29<sup>th</sup>, 2005

To the general manager of the company "BITUM" Ltd., Kozlov P.V.

03062, Kiev, Prospekt Pobedi 67, of. 36.

### Dear Phillip Vladimirovich!

We send you the "conclusions concerning the efficiency of the FLEXIGUM bituminous-latex covering in protecting concrete from biogenic sulfuric acid attack", which were drawn on the basis of work performed by the Ukrainian state research institute "UkrWODGEO" in accordance with the agreement #10/2005 with the company "BITUM" Ltd. from 28.03.2005.

Attached: as specified – 1 copy.

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#### **CONCLUSIONS**

## concerning the efficiency of the FLEXIGUM bituminous-latex covering in protecting concrete from biogenic sulfuric acid attack

The work was performed according to agreement #10/2005 with the company "BITUM" Ltd. from 28.03.2005.

One type of coating was tested (one control sample, three test samples). The client applied the coatings.

#### 1. Testing principle.

Biogenic sulfuric acid attack results from the action of sulfuric acid produced by thiobacilli [1-3] upon a structure. The highest activity of this type of corrosion is observed in drainage networks [1]. The results of research conducted in the laboratory of microbiological problems of water distribution facilities of the Ukrainian state research institute "UkrWODGEO", as well as data from science and engineering literature [1-5] indicate that the concrete of the hood part of sewage collectors is attacked by nitric acid, volatile organic acids, and lipid-like compounds aside for sulfuric acid. However, the main aggressive factor remains sulfuric acid. The quantitative determination of the effectiveness of coatings of different compositions in protecting concrete from biogenic sulfuric acid attack is based on initiating diffusion of sulfuric acid through the coating and performing quantitative evaluation of the intensity of this diffusion [5]. If the coating allows the diffusion of sulfuric acid, then the products of interaction between the sulfuric acid and the components of the concrete (mostly cement hydrates) will inevitably destroy the coating.

Sulfuric acid diffusion was detected by comparing the values of the active reaction of the concrete environment (the pH level) under the coating in the control sample (in the laboratory) and in 3 parallel test samples placed in a sewage collector. The test samples (3 units) were hung in nets on the hood part of the collector operating in a section where the chemical composition of the atmosphere is monitored by the laboratory of the "Kharkovkommunochistvod" establishment. The main parameters of the air and gas atmosphere in which the samples were placed are: hydrogen sulfide concentration –  $40\pm20$ , sulfur dioxide  $30\pm10$  mg/m³; methane concentration – 0.1-0.5, carbon dioxide – 0.1-0.5 % by volume. The duration of exposure was 127 days (04.04-09.08.05). The aggressiveness of condensed moisture acting upon the samples was evaluated after the end of the exposure and analysis of the unprotected concrete samples was made.

The active reaction of the concrete was measured using a solid state electrode. When evaluating the pH of the concrete under coatings, the pH of every sample was taken as the least mean value of this parameter on one of the four faces. The confidence interval when measuring the pH of concrete by this method is  $\pm 0.5$ . The predictive evaluation – the time to failure of the coating and the atmospheric concentration of hydrogen sulfide at which the durability of the coating would be up to 20 years, were calculated from expressions and dependencies established by us in special studies. Allowing for the difference in the conditions which the samples hung in the collector may have experienced, the predictive evaluations and the conclusion concerning the overall reliability of the coating are made based on the properties of the tested sample, which has allowed the largest decrease in the pH of the concrete under the coating.

#### 2. Test results

2.1. Evaluation of the aggressiveness of the environment which acted upon the tested samples during their exposure.

As indicated by the data from inspecting the unprotected concrete samples (Appendix A, tables A 1, A 2), the concrete was attacked by sulfuric acid, which caused a radical drop of the pH of the concrete. As indicated by the data of studies made by the laboratory of microbiological problems of water distribution facilities of the Ukrainian state research institute "UkrWODGEO", after an exposure exceeding 3 months the pH of the surface layer of concrete is similar to the pH of the operating atmosphere of the structure. According to the ChuII 2.03.11-85 construction standard, atmospheres with a pH of 3.53 are very aggressive with respect to concrete.

2.2. Evaluation of the reaction activity on the surface of the coating.

After removing the concrete samples with the FLEXIGUM bituminous-latex coating from the exposure nets, they were washed and the damp surfaces were investigated using a solid state electrode. As indicated by the data in appendix B (tables B 1-B 4), the sulfuric acid acting upon the coating reduced the pH of the coating itself. The coating of the control sample has a weakly acidic active environment reaction. The reduction in pH of the test samples indicates that biogenic sulfuric acid did act upon the coating. In the control and test samples the coating is very soft, which is indicated by the deep marks (fig.1) from the net on which the samples were placed during their exposure. The bituminous-latex FLEXIGUM coating is very sticky both in the test and in the control versions.

2.3. Evaluation of the diffusion of sulfuric acid through the coating.

The samples exposed in the collector were washed and photographed with the control samples (fig.1). As figure 1 shows, no visible destruction of the coating were observed in any of the three tested samples. The bituminous-latex FLEXIGUM coating had a good-satisfactory adhesion to the concrete in the test and in the control samples. On opening the coating was removed in one layer.

Fig. 1. Concrete samples protected by a bituminous-latex FLEXIGUM coating (left to right: three test samples and control)

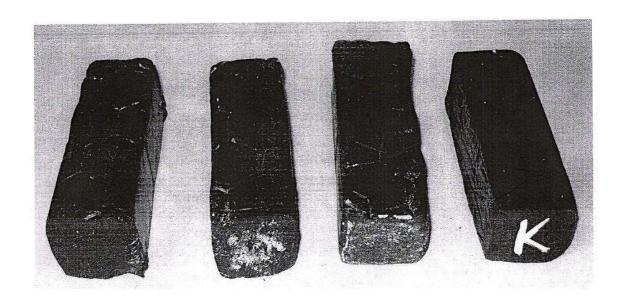


Table 1

Reliability evaluation of the bituminous-latex FLEXIGUM coating in protecting the concrete from biogenic sulfuric acid attack in sewer networks with a hydrogen sulfide concentration of 40±20 mg/dm³ in the air and gas atmosphere

			<u>, , , , , , , , , , , , , , , , , , , </u>						
Coating	Sample	Coating	pH of	pH of	Presence	Presence	Visual	Sulfuric acid	Evaluation
name	code	thickness,	concrete	concrete	of	of	adhesion	permeability	of protection of
		mm	under	under	external	bloating	assessment		concrete from
			coating	coating in	ruptures	in the			biogenic sulfuric
			in control	test sample		coating			acid attack
			sample			_			
FLEXIGUM	1.1	3.0-4.5	8.05	6.36	-	-	Satisfactory	+	Does not provide
bituminous-	1.2	4.1-5.3	8.05	7.33	-	-	Good-	+	Does not provide
latex based							satisfactory		_
	1.3	2.8-4.0	8.05	8.73	-	-	Good	-	Provides

Table 2

Predictive evaluation of the durability of the bituminous-latex FLEXIGUM coating in a drainage network atmosphere containing different hydrogen sulfide concentrations

	containing different nydrogen surface concentrations							
Coating	Sample	$pH_{initial}$ $-$	Exposure	Time to attain	Approximate	Mean hydrogen sulfide	Mean value of	
name	code	$\mathrm{pH}_{\mathrm{final}}$	time, days	similar	time to rupture	concentration in the	allowed	
				reduction in pH	of the coating,	atmosphere, for which	hydrogen sulfide	
				on the surface	days	the coating is reliable	concentration for	
				of unprotected		over 10 years (calculated	all samples,	
				concrete, days		for a period of 20 years),	$mg/m^3$	
				-		$mg/m^3$	_	
FLEXIGUM	1.1	8.05-6.36	127	5	1143	11.1		
bituminous-	1.2	8.05-7.33	127	3	1905	14.0	21	
latex based	1.3	8.05-8.73	127	0	exceeding 3650	40.0		

The determined values of the pH of the concrete under the coating in the test samples and their comparison to the pH of the concrete in the control sample (tables 1, 2, Appendix C – tables C 1-C 4) indicate that of the three parallel samples tested in conditions of an aggressive air and gas atmosphere and pellicular condensed moisture forming on the parts of the nets where exposure took place, two samples (1.1 and 1.2) did not provide reliable protection of the concrete from biogenic sulfuric acid attack, and one sample (1.3) – did. The measurements of the pH of the concrete of the test and control sample after removing a 1 mm concrete layer (Appendix D – table D 1-D 4) are given to exclude the possibility of an error in evaluation of the coating quality based on the concrete pH values under the coating as a result of an interaction of its components with cement hydrates during its application, their impregnation of the concrete, and diffusion into it, reducing its pH. This effect can be partially detected when measuring the pH of the concrete under the coating in the control sample. As indicated by the data from measuring the pH of the concrete after removing a 1 mm layer, in the control and test samples a significant increase in the pH was noted. In samples 1.2 and 1.3 the pH values of the concrete are the same as in the control (allowing for the confidence interval of this type of measurements). The diffusion of the sulfuric acid through the studied coating in sample 1.2 did not exceed 1 mm. in sample 1.1 the pH value of the concrete after removing a 1 mm layer is significantly lower than the pH of the concrete in the control sample, which attests to diffusion of sulfuric acid to a depth exceeding 1 mm.

### 2.4. Predictive evaluation of the reliability of the studied coatings.

The results of predictive evaluation of the durability of the studied bituminous-latex FLEXIGUM coating are shown in table 2. The predicted period until the destruction of the coating, calculated based on the sample which demonstrated the worst resistance to biogenic sulfuric acid attack (sample 1.1) is 1143 days; based on the sample which demonstrated medium resistance (sample 1.2) – 1905 days. The prediction did not take into account the elasticity of the coating, which should probably extend the predicted period. The calculated concentration of hydrogen sulfide in drainage network atmospheres, with which the studied coating will offer reliable protection of the concrete over more than 10 years (calculated for 20 years) is for the worst sample (sample 1.1) - 11±5.6 mg/m³, for the sample with medium resistance to biogenic sulfuric acid attack (sample 1.2) - 14±7.0 mg/m³.

The hydrogen sulfide concentrations at which the studied bituminous-latex FLEXIGUM coating shall protect the concrete over a period exceeding 10 years, averaged over all test samples, is 21 mg/m<sup>3</sup>. But, as noted above, the calculations and the conclusions are made based on the parameters of the test sample which had the lowest efficiency in protecting the concrete. Therefore the overall results of the tests of the coating lead to the conclusion that according to the predictive calculation the tested bituminous-latex FLEXIGUM coating with a thickness of at least 3.0 mm should afford reliable protection of concrete on drainage network facilities over at least 10 years (not exceeding 20 years) on sections where the average concentration of hydrogen sulfide is  $11\pm5.6$  mg/m<sup>3</sup>.

Manager of the laboratory of microbiological problems of water distribution facilities of the Ukrainian state research institute "UkrWODGEO", (-) Yurchenko V.A., Cand.Sci.

## Results of pH measurements of unprotected concrete samples Table A1

Table A2

Executor's control

Side	Ionometer readings	Concrete surface pH	Average pH	Min. pH value
1	-570	11.20		
	-620	12.57		
	-600	12.00		
	-570	11.20		
	-510	9.76		
	-505	9.65	11.06	
2	-540	10.46		
	-535	10.34		
	-550	10.70		
	-580	11.46		
	-550	10.70		
	-470	8.90	10.43	
3	-525	10.10		
	-555	10.82		
	-560	10.95		
	-534	10.31		
	-560	10.95		
	-510	9.76	10.48	
4	-500	9.54		
	-480	9.11		
	-465	8.80		
	-505	9.65		
	-485	9.21		
	-475	9.00	9.22	9.22

Execu	tor's contro	1 1.2 (after	r exposure	e)
Side	Ionometer	Concrete	Average	Min.
	readings	surface	pН	pН
	100000000000000000000000000000000000000	pН	r	value
1	-205	4.84		
Ĭ	-180	4.57		
	-170	4.46		
	-175	4.52		
	-180	4.57		
	-186	4.63		
	-165	4.41		
	-165	4.41		
	-173	4.50		
	-175	4.52	4.54	
2	-88	3.70		
	-175	4.52		
	-127	4.04		
	-140	4.17		
	-123	4.01		
	-126	4.03		
	-90	3.71		
	-5	3.05		
	-30	3.24		
	-15	3.13	3.76	
3	-77	3.60		
	-75	3.59		
	-105	3.84		
	-67	3.52		
	-88	3.70		
	-85	3.67		
	-45	3.35		
	-120	3.98		
	-105	3.84		
	-5	3.05	3.62	
4	-100	3.80		
	-120	3.98		
	-113	3.92		
	-95	3.76		
	-45	3.35		
	0	3.02		
	-15	3.13		
	-43	3.33		
	-75	3.59		
	-60	3.47	3.53	3.53

 $\begin{array}{c} & Appendix\ B\\ Results\ of\ pH\ measurements\ on\ the\ coating\ surface\ of\ samples\ after\ exposure\\ Table\ B1 & Table\ B2 \end{array}$ 

1.4	Bitum	control

Side	Ionometer	Surface	Average	Min.
	readings	pН	pН	pН
				value
1	-230	5.13		
	-232	5.15		
	-230	5.13		
	-235	5.18	5.15	
2	-215	4.95		
	-225	5.07		
	-213	4.93		
	-220	5.01	4.99	
3	-220	5.01		
	-215	4.95		
	-215	4.95		
	-205	4.84		
4	-235	5.18		
	-230	5.13		
	-237	5.21		
	-240	5.24	5.19	4.94

1	1	D:4 4	4
- 1	- 1	Bitum tes	ш

tum test			
Ionometer readings	Surface pH	Average pH	Min. pH value
-150	4.26		
-175	4.52		
-165	4.41		
-167	4.43	4.41	
-180	4.57		
-165	4.41		
-160	4.36		
-185	4.62	4.49	
-130	4.07		
-155	4.31		
-150	4.26		
-150	4.26	4.23	
-115	3.93		
-140	4.17		
-135	4.12		
-170	4.46	4.17	4.17
	Ionometer readings -150 -175 -165 -167 -180 -165 -160 -185 -130 -155 -150 -150 -115 -140 -135	Ionometer readings         Surface pH           -150         4.26           -175         4.52           -165         4.41           -167         4.43           -180         4.57           -165         4.41           -160         4.36           -185         4.62           -130         4.07           -155         4.31           -150         4.26           -115         3.93           -140         4.17           -135         4.12	Ionometer readings         Surface pH         Average pH           -150         4.26         4.26           -175         4.52         4.52           -165         4.41         4.41           -180         4.57         4.41           -165         4.41         4.41           -160         4.36         4.49           -130         4.07         4.26           -150         4.26         4.23           -115         3.93         4.17           -135         4.12         4.12

# Results of pH measurements of concrete under the coatings Table B1

Table B2

1.4 C	ontrol			
Side	Ionometer	Concrete	Average	Min.
	readings	surface	рН	pН
		рН		value
1	-445	8.40		
	-435	8.21		
	-423	7.99		
	-410	7.75		
	-445	8.40		
	-440	8.31		
	-455	8.60		
	-475	9.00		
	-477	9.05		
	-460	8.70	8.44	
2	-423	7.99		
	-425	8.03		
	-438	8.27		
	-424	8.01		
	-425	8.03		
	-425	8.03		
	-430	8.12		
	-445	8.40		
	-415	7.84		
	-410	7.75	8.05	
3	-437	8.25		
	-450	8.50		
	-478	9.07		
	-470	8.90		
	-450	8.50		
	-448	8.46		
	-440	8.31		
	-420	7.93		
	-440	8.31		
	-445	8.40	8.46	
4	-470	8.90		
	-445	8.40		
	-440	8.31		
	-423	7.99		
	-427	8.06		
	-445	8.40		
	-413	7.81		
	-410	7.75		
	-396	7.51		
	-385	7.32	8.05	8,05
	200	7.52	3.00	0,00

	1.1 Te	st			
Min.	Side	Ionometer	Concrete	Average	Min.
pН		readings	surface	pН	PH
value			рН		value
	1	-450	8.50		
		-440	8.31		
		-425	8.03		
		-445	8.40		
		-457	8.64		
		-433	8.17		
		-345	6.68		
		-365	6.99		
		-350	6.75		
		-372	7.10	7.76	
	2	-350	6.75		
		-370	7.07		
		-370	7.07		
		-376	7.17		
		-337	6.56		
		-340	6.60		
		-325	6.38		
		-378	7.20		
		-325	6.38	6.04	
		-376	7.17	6.84	
	3	-375	7.15		
		-340	6.60		
		-315	6.23		
		-342	6.63		
		-307	6.12		
		-303	6.06		
		-315	6.23		
		-332 -295	6.48 5.95		
		-305	6.09	6.36	
	4			0.30	
	4	-335	6.53 7.14		
		-374			
		-326 -345	6.39 6.68		
		-343	6.30		
		-325	6.38		
		-323	6.30		
		-320	6.45		
		-307	6.12		
8,05		-300	6.02	6.43	
0,03		-300	0.02	U.7J	

### 1.2 Test

### 1.3 Test

1.4 1		1	1		1.5 10		1		
Side	Ionometer	Concrete		Min.	Side	Ionometer	Concrete	Average	Min.
	readings	surface	pН	pН		readings	surface	pН	pН
		pН		value			pН		value
1	-380	7.24			1	-440	8.31		
	-392	7.44				-490	9.32		
	-320	6.30				-486	9.23		
	-410	7.75				-405	7.66		
	-297	5.98				-440	8.31		
	-285	5.82				-437	8.25		
	-425	8.03				-476	9.02	İ	
	-425	8.03				-470	8.90	İ	
	-440	8.31				-466	8.82		
	-443	8.37	7.33			-495	9.43	8.73	
2	-390	7.41			2	-480	9.11		
	-420	7.93				-527	10.15		
	-445	8.40				-512	9.80		
	-470	8.90				-535	10.34		
	-466	8.82				-530	10.22		
	-474	8.98				-525	10.10		
	-440	8.31				-515	9.87		
	-435	8.21				-492	9.36		
	-401	7.59				-481	9.13		
	-445	8.40	8.30			-480	9.11		
3	-400	7.58			3	-478	9.07		
3	-418	7.90				-495	9.43	1	
	-400	7.58				-470	8.90		
	-345	6.68				-463	8.76		
	-357	6.86				-480	9.11		
	-337	6.56				-470	8.90		
	-430	8.12				-460	8.70		
	-457	8.64				-475	9.00		
	-400	7.58				-461	8.72		
	-447	8.44	7.59			-450	8.50	8.91	
4	-445	8.40			4	-475	9.00	317	
	-477	9.05			'	-504	9.62		
	-435	8.21				-475	9.00		
	-450	8.50				-520	9.99		
	-434	8.19				-507	9.69		
	-480	9.11				-515	9.69		
	-442	8.35				-515	9.87		
	-410	7.75				-465	9.87		
	-450	8.50				-460	8.80		
	-461	8.72	8.48	7.33		-470	8.70	9.35	8.73
	-TU1	0.72	U.TU	1.33		- <b>T</b> /U	0.70	7.00	0.73

Appendix C Results of pH measurements of concrete under the coatings after removing a layer of 1 mm

Table C1 Table C2

1.4 C	Control				1.1 Te	est			
Side	Ionometer readings	Surface pH	Average pH	Min. pH value	Side	Ionometer readings	Surface pH	Average pH	Min. pH value
1	-510	9.76		7 442 47 4	1	-335	6.53		
	-525	10.10				-375	7.15		
	-465	8.80				-390	7.41		
	-465	8.80				-377	7.19		
	-500	9.54				-395	7.49		
	-490	9.32				-400	7.58		
	-491	9.34				-420	7.93		
	-483	9.17				-410	7.75		
	-495	9.43				-422	7.97		
	-490	9.32	9.36			-420	7.93	7.49	
2	-523	10.05			2	-450	8.50		
	-510	9.76				-430	8.12		
	-515	9.87				-430	8.12		
	-465	8.80				-460	8.70		
	-475	9.00				-453	8.56		
	-455	8.60				-425	8.03		
	-505	9.65				-430	8.12		
	-480	9.11				-430	8.12		
	-460	8.70				-440	8.31		
	-470	8.90	9.24			-428	8.08	8.26	
3	-490	9.32			3	-450	8.50		
	-470	8.90				-440	8.31		
	-470	8.90				-453	8.56		
	-486	9.23				-464	8.78		
	-500	9.54				-463	8.76		
	-500	9.54				-450	8.50		
	-505	9.65				-455	8.60		
	-480	9.11				-420	7.93		
	-480	9.11				-410	7.75		
	-492	9.36	9.27			-400	7.58	8.33	
4	-490	9.32			4	-432	8.16		
	-500	9.54				-437	8.25		
	-485	9.21				-450	8.50		
	-490	9.32				-430	8.12		
	-479	9.09				-420	7.93		
	-484	9.19				-405	7.66		
	-470	8.90				-410	7.75		
	-475	9.00				-420	7.93		
	-500	9.54				-432	8.16		
	-490	9.32	9.24	9.24		-430	8.12	8.06	7.49

### 1.2 Test

## 1.3 Test

Side	Ionometer	Concrete	Average	Min.	Side	Ionometer	Concrete	Average	Min.
	readings	surface	pН	pН		readings	surface	pН	pН
		pН		value			pН		value
1	-500	9.54			1	-557	10.87		
	-550	10.70				-560	10.95		
	-505	9.65				-545	10.58		
	-528	10.17				-525	10.10		
	-500	9.54				-513	9.83		
	-520	9.99				-528	10.17		
	-509	9.74				-534	10.31		
	-520	9.99				-525	10.10		
	-510	9.76				-520	9.99		
	-540	10.46	9.95			-504	9.62	10.25	
2	-477	9.05			2	-510	9.76		
	-493	9.38				-520	9.99		
	-496	9.45				-545	10.58		
	-423	7.99				-535	10.34		
	-515	9.87				-530	10.22		
	-510	9.76				-511	9.78		
	-503	9.60				-505	9.65		
	-525	10.10				-514	9.85		
	-495	9.43				-535	10.34		
	-510	9.76	9.44			-510	9.76	10.02	
3	-536	10.39			3	-500	9.54		
	-500	9.54				-505	9.65		
	-505	9.65				-528	10.17		
	-530	10.22				-499	9.51		
	-514	9.85				-495	9.43		
	-534	10.31				-500	9.54		
	-520	9.99				-493	9.38		
	-528	10.17				-410	7.75		
	-500	9.54				-417	7.88		
	-517	9.92	9.95			-507	9.69	9.25	
4	-545	10.58			4	-525	10.10		
	-545	10.58				-500	9.54		
	-530	10.22				-495	9.43		
	-540	10.46				-503	9.60		
	-543	10.53				-495	9.43		
	-535	10.34				-504	9.62		
	-538	10.41				-508	9.71		
	-544	10.55				-503	9.60		
	-528	10.17				-505	9.65		
	-517	9.92	10.37	9.44		-490	9.32	9.60	9.25