Two Unusual Phenolic Substances and One New Xanthone from *Hypericum sampsonii*

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Sampsone A (**1**), a novel prenylated aromatic lactone, and sampsone B (**2**), an unusual dihydrodibenzodioxinone, together with sampsone C (**3**), a new xanthone, were isolated from the aerial parts of *Hypericum sampsonii*. Their structures were determined by spectroscopic methods which were mainly 1D- and 2D-NMR techniques, and the structure of sampsone B (**2**) was also confirmed by X-ray crystallographic analysis. All of these compounds were evaluated for *in vitro* antibacterial activity against methicillin-resistant *Staphylococcus aureus* (MRSA). Only sampsone A showed moderate antibacterial activities at a minimum inhibitory concentration (*MIC*) of 32 μg/ml.

Introduction. – Plants of the genus *Hypericum* have been used as traditional medicinal plants in various parts of the world, and some of them have antidepressant, antiviral, wound-healing, and antimicrobial bioactivities [1]. In mainland China, *H. sampsonii* is used for the treatment of numerous disorders such as backache, burns, diarrhoea, snakebites, and swellings [2]. Because of its various bioactivities, *H. sampsonii* has been investigated, and some xanthones and polyprenylated benzophenone derivatives have been isolated [3–12]. As a part of a program to discover new antibacterial natural products from plant resources, a CH_2Cl_2 extract of the aerial parts of *H. sampsonii* was shown to exhibit significant biological activity in a preliminary *in vitro* screening against methicillin-resistant *Staphylococcus aureus* (MRSA) with an *MIC* of 64 μg/ml. Therefore, research was carried out on the bioactive constituents of the CH_2Cl_2 extract, and we isolated sampsone A (**1**), a novel prenylated aromatic lactone, and sampsone B (**2**), an unusual dihydrodibenzodioxinone, together with sampsone C (**3**), a new xanthone (=9*H*-xanthen-9-one). We now report the methods and results of isolating and characterizing these compounds from *H. sampsonii*, as well as the assessment of their bioactivity against MRSA.

Results and Discussion. – 1. *Structure Elucidation*. Compound **1**[1]) was obtained as a white amorphous powder, and had the molecular formula $C_{22}H_{24}O_6$ with eleven degrees of unsaturation as determined by HR-ESI-MS ($[M+H]^+$ at *m/z* 385.1643). The IR absorptions revealed the presence of OH (3423 cm^{-1}) and C=O groups (1719 and

[1]) Arbitrary atom numbering; for systematic names, see *Exper. Part.*

1655 cm^{-1}). The ^{13}C-NMR and DEPT spectra of **1** (*Table*) showed 22 signals, assigned as two C=O groups, six benzene C-atoms, six olefinic C-atoms, four Me, two sp^3 CH_2, and one O-bearing sp^3 CH group, and one sp^3 C-atom. Characteristic ^{13}C-NMR resonances including those for three O-bearing aromatic C-atoms (δ(C) 156.6, 164.7, and 161.1), for one aromatic C-atom (δ(C) 102.8), and for two aromatic CH groups (δ(C) 93.1 and 98.9), along with two aromatic H-atoms (δ(H) 6.29 (*d*, $J = 1.8$ Hz, 1 H) and 6.18 (*d*, $J = 1.8$ Hz, 1 H)) in the ^{1}H-NMR spectrum (*Table*) indicated the presence of a phloroglucinol (= benzene-1,3,5-triol) moiety. Besides, the ^{1}H-NMR spectrum showed a *s* at δ(H) 12.50 from an OH group H-bonded to a C=O, a 3-methylbut-2-enyl group (δ(H) 5.05 (*t*, $J = 6.3$ Hz, 1 H), 2.85 (*dd*, $J = 14.0$, 7.5 Hz, 1 H), 2.63 (*dd*, $J = 14.0$, 6.2 Hz, 1 H), 1.68 (*s*, 3 H), and 1.65 (*s*, 3 H)), and a 2-oxy-3-methylbut-3-enyl moiety (δ(H) 5.03 (*s*, 1 H), 4.93 (*s*, 1 H), 4.88 (*dd*, $J = 9.6$, 6.4 Hz, 1 H), 2.59 (*dd*, $J = 12.8$, 9.6 Hz, 1 H), 2.14 (*dd*, $J = 12.8$, 6.4 Hz, 1 H), and 1.61 (*s*, 3 H)). Two structural fragments, **1a** and **1b**, were further refined by analysis of the ^{1}H,^{1}H-COSY, HSQC, HMBC, and NOESY data of **1** (*Fig. 1*). The HMBC cross-peaks displayed the following correlations: CH_2(1)/C(11a), C(4a), C(2), C(11), C(1′), and C(1″), CH_2(1″)/C(11), C(11a), C(1), and C(4a), and Me(12)/C(4) and C(4a). The aforementioned data, together with the NOESY correlation Me(12)/H–C(2) suggested that C(2) and C(4) were linked through an ether bridge, which established the partial structure **1b** (*Fig. 1*). Considering the HMBCs of H–C(7)/C(6), C(8), C(5a), and C(9), and H–C(9)/C(8), C(9a), C(7), and C(5a), the other partial fragment **1a** was constructed. The connections of the two moieties **1a** and **1b** were elucidated from the following spectral data: *i*) The C-atom at δ(C) 156.63 in the ^{13}C-NMR spectrum was assigned to C(9a), which bonded to an O-atom, based on the HMBC spectrum; so the lactone moiety linked C(11a) to C(9a). *ii*) The ^{1}H-NMR spectrum revealed the presence of a

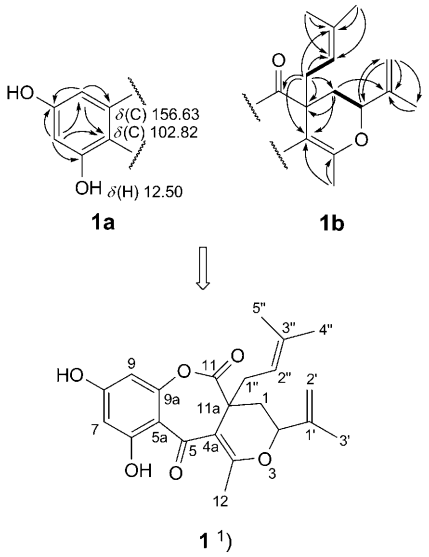


Fig. 1. *Partial structures* **1a** *and* **1b** *and the key* ^{1}*H,*^{1}*H-COSY* (—) *and HMBCs* (H → C) *of* **1**

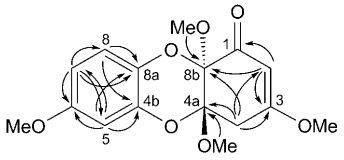
Table. *[1]H- and [13]C-NMR Data of* **1**–**3**[1]). δ in ppm, J in Hz.

	1 (in (D_6)DMSO)			**2** (in CD_3OD)			**3** (in CD_3OD)	
	δ(C)	δ(H)		δ(C)	δ(H)		δ(C)	δ(H)
CH_2(1)	38.5	2.14 (*dd*, *J* = 12.8, 9.6), 2.59 (*dd*, *J* = 12.8, 6.4)	C(1)	188.3		C(1)	165.0	
H–C(2)	79.1	4.88 (*dd*, *J* = 9.6, 6.4)	H–C(2)	99.7	5.43 (*d*, *J* = 1.5)	H–C(2)	99.4	6.16 (*d*, *J* = 1.8)
C(4)	164.3		C(3)	173.2		C(3)	167.2	
C(4a)	120.1		CH_2(4)	33.3	3.15 (*dd*, *J* = 17.6, 1.5), 3.09 (*d*, *J* = 17.6)	H–C(4)	95.4	6.42 (*d*, *J* = 1.8)
C(5)	180.3		C(4a)	96.2		C(4a)	159.5	
C(5a)	102.8		C(4b)	140.0		C(4b)	148.8	
C(6)	161.1		H–C(5)	102.9	6.56 (*d*, *J* = 2.9)	C(5)	130.7	
H–C(7)	98.9	6.18 (*d*, *J* = 1.8)	C(6)	156.1		C(6)	154.7	
C(8)	164.7		H–C(7)	108.4	6.54 (*dd*, *J* = 9.5, 2.9)	C(7)	129.6	
H–C(9)	93.1	6.29 (*d*, *J* = 1.8)	H–C(8)	118.4	7.10 (*d*, *J* = 9.5)	H–C(8)	113.7	7.72 (*s*)
C(9a)	156.6		C(8a)	134.0		C(8a)	116.9	
C(11)	178.4		C(8b)	92.8		C(9)	181.9	
C(11a)	48.0		*Me*O–C(3)	56.4	3.77 (*s*)	C(9a)	102.6	
Me(12)	20.6	2.47 (*s*)	*Me*O–C(4a)	49.5	3.25 (*s*)	C(2′)	72.2	
C(1′)	142.4		*Me*O–C(6)	55.7	3.76 (*s*)	H–C(3′)	100.4	4.42 (*d*, *J* = 4.1)
CH_2(2′)	112.1	5.03 (*s*), 4.93 (*s*)	*Me*O–C(8b)	51.3	3.27 (*s*)	H–C(4′)	73.6	5.40 (*d*, *J* = 4.1)
Me(3′)	17.2	1.61 (*s*)				Me(5′)	25.8	1.30 (*s*)
CH_2(1″)	34.4	2.85 (*dd*, *J* = 14.0, 7.5), 2.63 (*dd*, *J* = 14.0, 6.2)				Me(6′)	25.7	1.31 (*s*)
H–C(2″)	119.3	5.05 (*t*, *J* = 6.3)						
C(3″)	135.2							
Me(4″)	25.7	1.65 (*s*)						
Me(5″)	18.0	1.68 (*s*)						
OH–C(6)		12.50 (*s*)						
OH–C(8)		3.36[a])						

[a]) Overlapped with the residual H_2O in (D_6)DMSO protonated signal (δ(H) 3.36).

H-bonded OH group (δ(H) 12.50 (*s*, 1 H)) at C(6); so, the left over C=O group should be placed between C(5a) and C(4a) (*Fig. 1*). Thus, the planar structure of **1** was defined. Attempts to prepare crystals suitable for X-ray analysis of **1** were unsuccessful, and to the best of our knowledge, there are no compounds similar to **1** that have been reported up to now. Hence, herein we report the planar structure of sampsone A (**1**), the determination of its configuration requiring further research.

Compound **2**[1]) was obtained as colorless crystals (from MeOH) with the molecular formula $C_{16}H_{18}O_7$ as established by HR-ESI-MS ($[M+H]^+$ at *m*/*z* 323.1113), requiring eight degrees of unsaturation. The IR spectrum showed absorption bands at 1684 cm^{-1} (C=O group) and 1612 and 1604 cm^{-1} (C=C). The ^{13}C-NMR spectrum (*Table*) showed 16 signals which, combined with the analysis of the HSQC spectra, corresponded to seven quaternary C-atoms including one diagnostic ketone C-atom (δ(C) 188.3), and to four sp^2 CH, one sp^3 CH_2 (δ(C) 33.3), and four MeO groups (δ(C) 56.4, 55.7, 51.3, and 49.5). The ^{1}H-NMR spectrum (*Table*) showed the presence of four MeO groups (δ(H) 3.77, 3.76, 3.27, and 3.25) and three aromatic H-atoms (δ(H) 7.10 (*d*, *J* = 9.5 Hz, 1 H), 6.56 (*d*, *J* = 2.9 Hz, 1 H), and 6.54 (*dd*, *J* = 9.5, 2.9 Hz, 1 H)) indicating the presence of one 1,2,4-trisubstituted benzene ring. An olefinic H-atom at δ(H) 5.43 (*d*, *J* = 1.5 Hz, 1 H) correlated with C(3), C(4), and C(8b), and one MeO group at δ(H) 3.77 correlated with C(3) in the HMBC spectrum, indicating the substructure of an α,β-unsaturated ketone. Furthermore, the HMBCs of CH_2(4) with C(2), C(3), C(4a), and C(8b), and of the MeO groups at δ(H) 3.27 and 3.25 with the quaternary C-atoms C(8b) and C(4a), respectively, established that the partial structures of $-CH_2-$, α,β-unsaturated ketone, MeO–C(4a), and MeO–C(8b) formed a six-membered-ring moiety. Considering the molecular mass, the degrees of unsaturation, and the chemical shifts of C(4a), C(4b), C(8a), and C(8b) suggested that the six-membered-ring moiety was connected to the benzene moiety by two ether bonds from C(4a) and C(8b) to C(4b) and C(8a), respectively (*Fig. 2*). A single-crystal X-ray diffraction analysis was successfully conducted to confirm the planar structure of sampsone B (**2**) and allowed the determination of its relative configuration (*Fig. 3*).



2 [1])

Fig. 2. *Structure and key HMBCs* (H → C) *of* **2**

Compound **3** was obtained as a yellow amorphous powder. It was assigned the molecular formula $C_{18}H_{16}O_8$ as inferred from its HR-ESI-MS ($[M+H]^+$ at *m*/*z* 361.0921) and NMR data. Its UV (MeOH) spectrum (365, 282, 256, and 235 nm) suggested a xanthone-skeleton structure. The IR spectrum of **3** revealed the presence of OH (3440 cm^{-1}) and conjugated-ketone groups (1634 cm^{-1}). The ^{13}C-NMR spectrum of **3** (*Table*) showed 18 C-atom signals, which were analyzed by DEPT and HSQC techniques as two Me and five CH groups, and eleven quaternary C-atoms

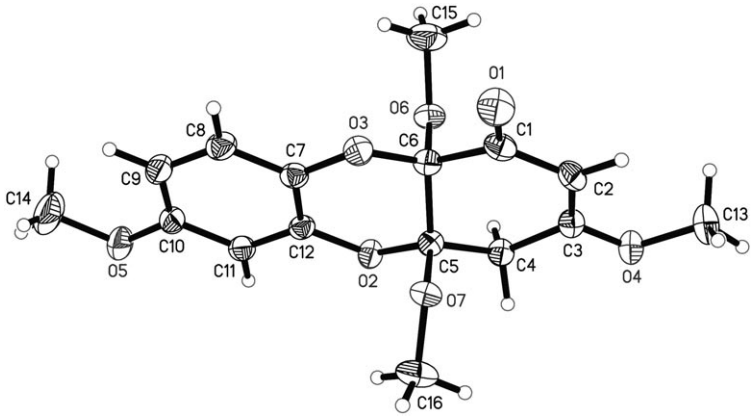
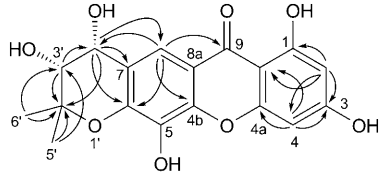


Fig. 3. *Single-crystal X-ray analysis of* **2**[1])

including a C=O group (δ(C) 182.0). The ^{1}H-NMR spectrum of **3** (*Table*) revealed the presence of three aromatic H-atoms (δ(H) 7.72 (*s*, 1 H), 6.42 (*d*, $J = 1.8$ Hz, 1 H), and 6.16 (*d*, $J = 1.8$ Hz, 1 H)). The high deshielding of the H-atom at δ(H) 7.72 suggested that it was located in the paramagnetic anisotropy cone of the C=O moiety, *i.e.*, at the C(8) position of the 9*H*-xanthen-9-one moiety. Furthermore, the ^{1}H- and ^{13}C-NMR spectra displayed evidence of the presence of one dimethylchromene (=dimethyl-2*H*-1-benzopyran) moiety (δ(H) 1.30 and 1.31 (2*s*, 3 H each), and 4.42 (*d*, $J = 4.2$ Hz, 1 H), and 5.40 (*d*, $J = 4.1$ Hz, 1 H); δ(C) 25.7, 25.8, 72.2, 100.4, and 73.6). The ^{1}H- and ^{13}C-NMR data of **3** were quite similar to those of a 3,4-dihydro-3,4,7,9,12-pentahydroxy-2,2-dimethyl-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one, except for the signals assigned to the chromene ring, suggesting that **3** was a stereoisomer of it [13] (*Table*). Furthermore, the HMBCs H–C(8)/C(4′) (δ(C) 73.6), H–C(4′) (δ(H) 5.40)/C(6) (δ(C) 154.7), C(7) (δ(C) 129.6), C(8) (δ(C) 113.7), C(2′) (δ(C) 72.2), and C(3′) (δ(C) 100.4), and H–C(3′) (δ(H) 4.42)/C(4′), C(2′), C(5′) (δ(C) 25.8) and C(6′) (δ(C) 25.7) also confirmed that the chromene moiety was fused in a linear form with the xanthene skeleton (*Fig. 4*). Moreover, the coupling constant J(3′,4′) of 4.3 Hz in **3** suggested that the two neighboring OH groups at C(3′) and C(4′) were *cis*-oriented [14]. Thus, the structure of **3** was established and named sampsone C (*Fig. 4*). This compound is a racemate since neither optical rotation nor CD effects could be measured for this compound.



3 [1])

Fig. 4. *Structure and key HMBCs* (H → C) *of* **3**

2. *Antibacterial Activity.* All compounds were assessed *in vitro* for their antibacterial activities against MRSA. Only sampsone A (**1**) exhibited moderate activity with an *MIC* of 32 μg/ml, while sampsone B (**2**) and sampsone C (**3**) showed less activity with *MIC* values $\geq$128 μg/ml.

This project was supported by the *Special Funds of Science and Technology* Commission of Shanghai (08DZ1971505).

Experimental Part

General. Anal. TLC: *HSGF254* SiO_2 plates (0.20–0.25 mm; *Yantai Chemical Industrial Institute*, P. R. China). Prep. TLC: *HSGF254* SiO_2 plates (0.40–0.50 mm; *Yantai Chemical Industrial Institute*, P. R. China). Column chromatography (CC): silica gel (SiO_2, 200–300 mesh; *Qingdao Marine Chemical Inc.*, Qingdao, P. R. China) or *Sephadex LH-20* (*Pharmacia Biotech AB*, Uppsala, Sweden) as packing materials. M.p.: *Büchi-B-540* melting point apparatus; uncorrected. Optical rotations: *Krüss-P800-T* polarimeter. IR Spectra: *Nicolet*™*-380* spectrometer from *Thermo Electron*; $\tilde{\nu}$ in cm^{-1}. 1D- and 2D-NMR Spectra: *Bruker-DRX-400* instrument; δ in ppm rel. to Me_4Si as internal standard, *J* in Hz. ESI-MS: *Finnigan-LCQ-DECAXP*^{plus} mass spectrometer; in *m*/*z* (rel. %). HR-ESI-MS: *ApexIII* FT-MS (7 Tesla) spectrometer (*Bruker Daltonics, Inc.*); in *m*/*z*.

Plant Material. The plant material (stems and leaves) of *H. sampsonii* was collected from Jinhua, Zhejiang Province, P. R. China, and was identified by *L.-P. Q.*, School of Pharmacy, Second Military Medical University, where the voucher specimen (No. 2008YBC1) was deposited.

Extraction and Isolation. The whole air-dried plant material (5 kg) was extracted with CH_2Cl_2 at r.t. After evaporation of the solvent, a part of the residue (100 g) was subjected to CC (SiO_2, petroleum ether/acetone 20:1, 10:1, 5:1, 3:1, and 1:1): *Frs. 1–5*. *Fr. 4* (8 g) was resubjected to CC (SiO_2, hexane/acetone 3:1) to give **3** (15 mg) as a yellow powder and three new fraction, *Fr. 4.1* (200 mg), *Fr. 4.2* (2.0 g), and *Fr. 4.3* (3.5 g). *Fr. 4.1* was resubjected to CC (*Sephadex LH-20*, MeOH): *Frs. 4.1a–4.1d*. *Fr. 4.1b* was purified by prep. TLC (hexane/$CHCl_3$/acetone 4:1:0.4): **1** (5 mg). *Fr. 4.1c* was purified by prep. TLC (hexane/acetone 5:1): **2** (8 mg).

Sampsone A (=*4,4a-Dihydro-8,10-dihydroxy-1-methyl-4a-(3-methylbut-2-en-1-yl)-3-(1-methylethenyl)-11*H*-pyrano[4.3-*c*][1]benzoxepin-5,11(3*H*)-dione*; **1**): White amorphous powder. M.p. 212–214°. $[\alpha]_D^{25} = +32.5$ ($c = 0.5$, MeOH). UV (MeOH): 207, 252, 259, 297. IR (KBr): 3423, 2924, 1719, 1655, 1618, 1508, 1383, 1350, 1167, 997. NMR: *Table*. HR-ESI-MS (pos.): 385.1643 ($[M+H]^+$, $C_{22}H_{25}O_6^+$; calc. 385.1646).

Sampsone B (=rel-*(4a*R*,10a*R*)-4a,10a-Dihydro-3,4a,7,10a-tetramethoxydibenzo[*b*,*e*][1,4]dioxin-1(4*H*)-one*; **2**): Colorless crystals (MeOH). M.p. 252–254°. $[\alpha]_D^{25} = -12.8$ ($c = 0.5$, MeOH). UV (MeOH): 203, 220, 255. IR (KBr): 2909, 1684, 1612, 1604, 1506, 1382, 1159, 1086. NMR: *Table.* HR-ESI-MS (pos.): 323.1113 ($[M+H]^+$, $C_{16}H_{19}O_7^+$; calc. 323.1125).

Sampsone C (=*(3*RS*,4*RS*)-3,4-Dihydro-3,4,7,9,12-pentahydroxy-2,2-dimethyl-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one*; **3**): Yellow amorphous powder. UV (MeOH): 365, 282, 256, 235. IR (KBr): 3440, 2924, 1634, 1568, 1483, 1320, 1167, 1052. NMR: *Table.* HR-ESI-MS (pos.): 361.0921 ($[M+H]^+$, $C_{18}H_{17}O_8^+$; calc. 361.0918).

X-Ray Crystallographic Analysis of **2**. Single crystals suitable for X-ray analysis were obtained by recrystallization from MeOH soln. A colorless prismatic crystal (*ca.* 0.327 × 0.189 × 0.186 mm) was used for analysis. All measurements were recorded by means of a *Bruker-Smart-CCD* area-detector diffractometer with graphite-monochromated MoK_α radiation (λ 0.71073 Å) at 293 K and in the $\varphi-\omega$ mode. Data collection and cell refinement: *Bruker Smart*. Program used to refine structure: SHELXL-97; refinement on F^2, full-matrix least-squares calculations. Crystal data and experimental details: $C_{16}H_{18}O_7$, M_r 322.30, orthorhombic, space group $P2_12_12_1$ ($Z = 4$); $a = 7.3901$ (10) Å, $b = 12.6190$ (17) Å, $c = 16.905$ (2) Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$; θ range 4.811–46.245°; R ($I > 2\sigma(I)$) = 0.0558, $wR_2 = 0.0958$. CCDC-779567 contains the supplementary crystallographic data for this article. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif.

Antibacterial Activity. In vitro antibacterial activity was screened against methicillin-resistant *Staphylococcus aureus* as reported previously [15]. Briefly, the *S. aureus* strain was cultured on nutrient agar (*Oxoid*) and incubated for 24 h at 37° prior to the *MIC* determination. Bacterial inoculums containing *ca.* 10^4 colony-forming units (CFU)/ml were used in these assays. *Muller–Hinton* broth (MHB; *Oxoid*) was adjusted to contain 20 and 10 mg/l of Ca^{2+} and Mg^{2+}, resp. MHB (125 µl) was dispensed into ten wells of a 96 well microtitre plate. Tested samples were dissolved in DMSO. Serial 2-fold dilutions of the tested samples were mixed with MHB in the ratio 1:100 in the microtitre plate. Final concentration of the test samples in agar ranged from 512 to 0.25 µg/ml. Compounds were serially diluted into each of the wells followed by the addition of the bacterial inoculum (125 µl), and the microtitre plate was incubated at 37° for 18 h. For the *MIC* determination, 20 ml of a 5 mg/ml MeOH soln. of 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl-2*H*-tetrazolium bromide (MTT; *Sigma*) was added to each of the wells and incubated for 20 min. The *MIC* was recorded as the lowest concentration at which no growth was observed. The test was performed in triplicate. All *MIC*s were determined on at least three independent occasions. Vancomycin was used as a pos. control drug from *Sigma Chemical*. To clarify any participating role of DMSO in the biological screening, separate studies were carried out with the solns. of DMSO alone, and they showed no activity against the bacterial strain.

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Received July 27, 2010