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	NiCl <sub>2</sub> (0.1M)/ml	$Na_2H_2PO_2(0.2M)/ml$	H <sub>2</sub> O/ml
А	20	0	30
В	0	20	30
С	20	20	10

Table S1 Composition of different electrolytes used in the electrochemical test of CdS-NiP<sub>x</sub>.



Figure S1 TEM (a) and HRTEM (b) images of CdS.

Content	wt.%		at.%		wt.%
Samples	Ni	Р	Ni	Р	NiP <sub>x</sub>
CdS-5NiP <sub>x</sub>	0.30	0.70	0.40	2.00	1.00
CdS-10NiP <sub>x</sub>	0.36	0.76	0.50	2.30	1.12
CdS-20NiP <sub>x</sub>	0.50	0.80	0.70	2.50	1.30
CdS-30NiP <sub>x</sub>	0.70	1.20	1.00	3.30	1.90

Table S2 Approximate content of NiP<sub>x</sub> in CdS-NiP<sub>x</sub> photocatalysts.



Figure S2 Wide scan XPS spectra of CdS and CdS-20NiP<sub>x</sub>.



Figure S3 The Tauc plots of CdS and CdS-20NiP<sub>x</sub> obtained from Fig. 5(a).



Figure S4 Proposed preparation process of  $CdS-NiP_x$  by photochemical reduction method.



Figure S5 (a) Time courses of photocatalytic hydrogen production of different CdS-NiP<sub>x</sub> and bare CdS, and (b) long-term photocatalytic hydrogen production of CdS-20NiP<sub>x</sub>.



Figure S6 Time courses of photocatalytic hydrogen production of CdS with different sacrificial agents.